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Cloud Point Depressants

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Title: Cloud Point Depressants	Subject (3-4 words): Cloud point depressants Pour point depressants Inhibited waxy crude oil Subsea pipeline restart
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<p>ABSTRACT</p> <p>Goal of work (key words): Wax formation and deposition in subsea crude oil pipeline is most important problem in cold environments faced by petroleum industry. Significant research is being going on at industrial as well as academic levels to develop additives which are able to break the wax crystal structure or at least weakens it. Addition of cloud point depressants has been found to be an effective way of dealing with waxes. The main focus of this project is chemical control of wax gel formation by using cloud point depressants (pour point depressants) and to find the action mechanisms of cloud point depressants on Model oil systems.</p> <p>Novel cloud point depressants were used and dosed in model wax oil system. Glass vessel experiments were performed on it and samples were analyzed using spectrfluorometer, NMR and GC apparatus to identify the mechanisms of these inhibitors.</p> <p>Conclusions and recommendations (key words): In this study, Protocols for Glass vessel and spectrofluorometer were developed to find out mechanisms of PPDs. Results indicate that these protocols works well for PPD2 and PPD3 in Model wax oil systems. PPDs help in reducing cloud point of Model wax oil systems but to a small extent, this gives an impression as they were not able to change the wax crystal structure. Probes taken from glass vessel experiemnt, when analyzed using spectrofluorometer, gives increased fluorescence signal illustrates that concentration of PPDs in probe is increasing. Low field NMR can be used to monitor the formation of solid/amorphous phase in a liquid containing wax. Results from GC analysis explains that high molecular alkanes are participating in forming wax crystals.</p> <p>A more detailed research on the mechanism of cloud point depressants should be carried out. For research in future, experiments can be performed by varying model oil composition in order to get closer to real crude oil composition, and hence the performance of PPDs could be seen brightly. Investigations of concentration dependence of PPDs content in Model wax oil systems on carbon number distribution of alkanes are suggested by GC analysis.</p>	
<p>I declare that this is an independent work according to the exam regulations of the Norwegian University of Science and Technology</p>	
<p>Date and signature: <i>Hassan Ali</i> 25/6/2012</p>	

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1 INTRODUCTION

Crude oil composition varies geographically. Petroleum production processes often encounter problems related to flow assurance. The discipline of flow assurance denotes the branch of knowledge associated with designing production systems for continuous, safe and uninterrupted transport of crude oil from reservoirs to processing facilities. Major challenges involved with ensuring flow of crude oil include solid deposition such as wax (paraffins), asphaltenes, scale, naphthenates and gas hydrates in flow lines [Kelland, 2009]. Waxes also named Paraffins are high molecular weight saturated aliphatic components of crude oil with carbon chain length varying from 16 to about 100. Asphaltenes are polynuclear aromatic hydrocarbon molecules in which non-aromatic aliphatic chains are also present along with heteroatoms. Gas hydrates are solids of crystalline nature consisting of water and light hydrocarbons.

Gelled pipeline shut-in and restart is a serious flow assurance problem currently faced in the petroleum transport industry [Jorda, 1966]. At reservoir temperature (Above 70⁰C) and pressure (Above 50 MPa), wax molecules are dissolved in the crude oil. However, as the crude oil flows through a subsea pipeline at a temperature of 4⁰C, due to heat losses to the surroundings the temperature of oil eventually decreases below its cloud point (wax appearance temperature). The solubility of wax decreases with decrease in temperature and wax molecules start to precipitate out of the crude oil.

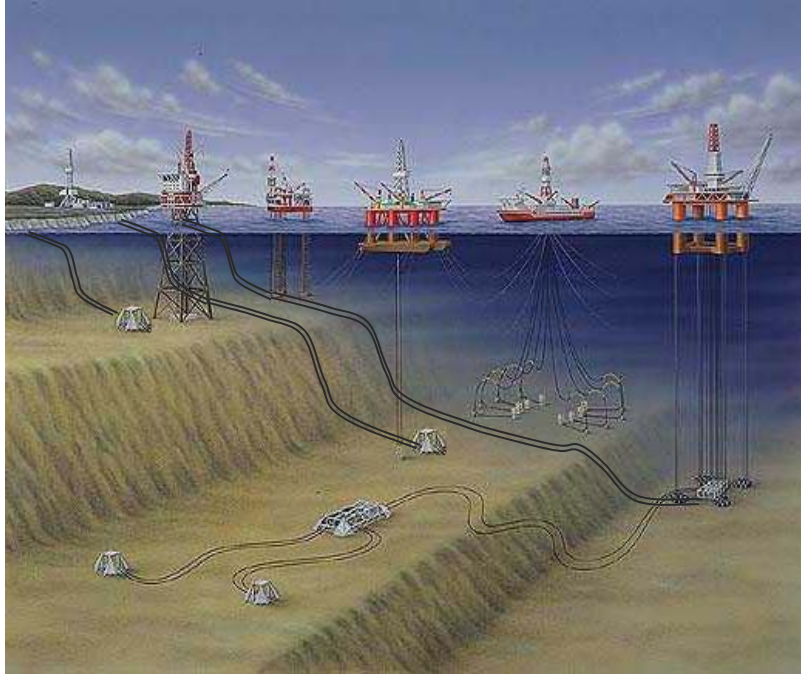


Figure 1.1: Deep sea crude oil production

Crude oil production in deep sea area has increased significantly in the past few years and there is a need of safer crude oil transportation lines as the oil wells are miles away from the shore as illustrated in figure 1.1. But wax problems have become more severe due to increased offshore crude oil transportation pipelines. Risk mitigation associated with solid deposition, gel formation and plugging comprises a focused effort of petroleum production companies. In addition, the term flow assurance encompasses econometric modeling of petroleum production value streams.

It is essential to gain a fundamental understanding of waxy crude oil and flow assurance issues involving the production and transportation operations in subsea environment. The focus of this research is the restart

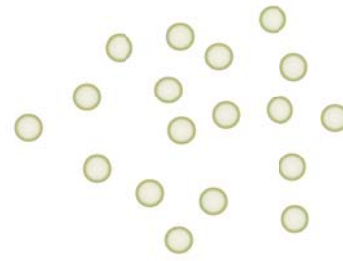
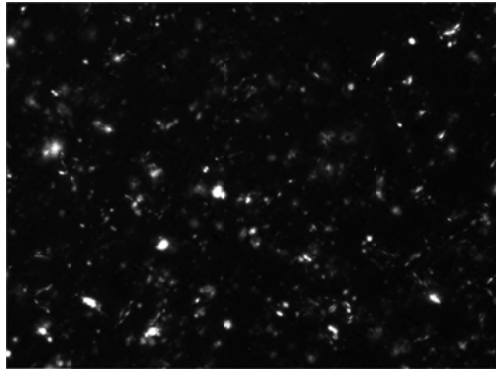
problem occurring following shut-in period as a result of the precipitation of waxy crystals and how to cope with this problem by the help of chemical inhibitors mainly cloud point depressants (also named as pour point depressants).

1.1 Paraffin wax

Pure paraffin waxes are solids consisting of normal, cyclic or branched alkanes with a number of carbon atoms greater than 16. Paraffin waxes are naturally present in crude oil and gas condensate. Cyclic alkanes and aromatic hydrocarbons may also be present. There are two primary classifications of paraffin waxes which are extracted from Crude oil:

1. Macro-crystalline waxes
2. Micro-crystalline waxes

Micro-crystalline wax contains a high fraction of naphthenic (cyclic) and iso-paraffins (branched) in addition to its high molecular weight straight chain hydrocarbons (C_{30} - C_{60}). This type of wax has small crystal size and form spherical morphology presented in figure 1.2a, which is difficult to treat. Sasol wax 3971 is a micro-crystalline wax. Crude oil containing micro-crystalline wax usually has high pour point values as compared to macro-crystalline wax. Macro-crystalline wax consists primarily of straight chain hydrocarbons (C_{20} - C_{50}). This type of wax has large crystal size and form plate like morphology as shown in figure 1.2b. [Hansen et al., 1991] Sasolwax 5405 is a macro-crystalline wax; its composition is given in table 1.1.



Spherical Morphology

Figure 1.2a: Micro-crystalline wax structure

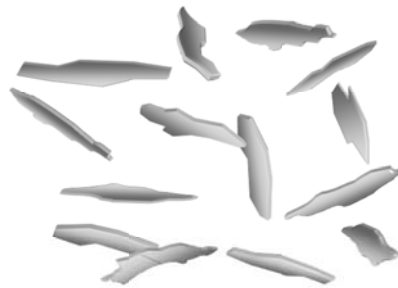
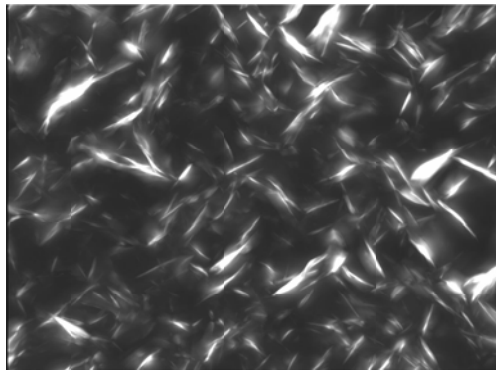


Plate-like Morphology

Figure 1.2b: Macro-crystalline wax structure

Table 1.1: Composition of Sasolwax 5405 [Paso et al., 2009]

	<i>Sasolwax 5405 composition</i>	
	<i>(w/w %)</i>	
	n-Paraffins	Non-n-paraffins
C ₂₀	0.2	0.1
C ₂₁	1.0	0.1
C ₂₂	2.6	0.1
C ₂₃	5.4	0.5
C ₂₄	8.6	1.0
C ₂₅	10.3	1.8
C ₂₆	10.7	2.3
C ₂₇	9.5	2.6
C ₂₈	7.8	2.9
C ₂₉	6.5	2.6
C ₃₀	4.5	2.1
C ₃₁	3.2	1.2
C ₃₂	2.2	1.1
C ₃₃	1.5	0.9
C ₃₄	1.0	0.8
C ₃₅	0.7	0.7
C ₃₆	0.5	0.5
C ₃₇	0.5	0.5
C ₃₈	0.3	0.4
C ₃₉	0.2	0.1
C ₄₀	0.2	0.1
C ₄₁	0.1	0.1
C ₄₂	0.1	0
C ₄₃	0.1	0
C ₄₄	0.1	0

Among several types, one synthetic type of wax is called Fischer-Tropsch wax, produced from a well-known Fischer-Tropsch process. Fischer-Tropsch is a method for the synthesis of hydrocarbons and other aliphatic compounds from synthesis gas; a mixture of hydrogen and carbon monoxide in the presence of a catalyst. Fischer-Tropsch wax consists mainly of saturated, straight chain hydrocarbons with almost no branches. This linear structure of Fischer-Tropsch wax is responsible for many desirable properties of Fischer-Tropsch waxes. [1] [2]

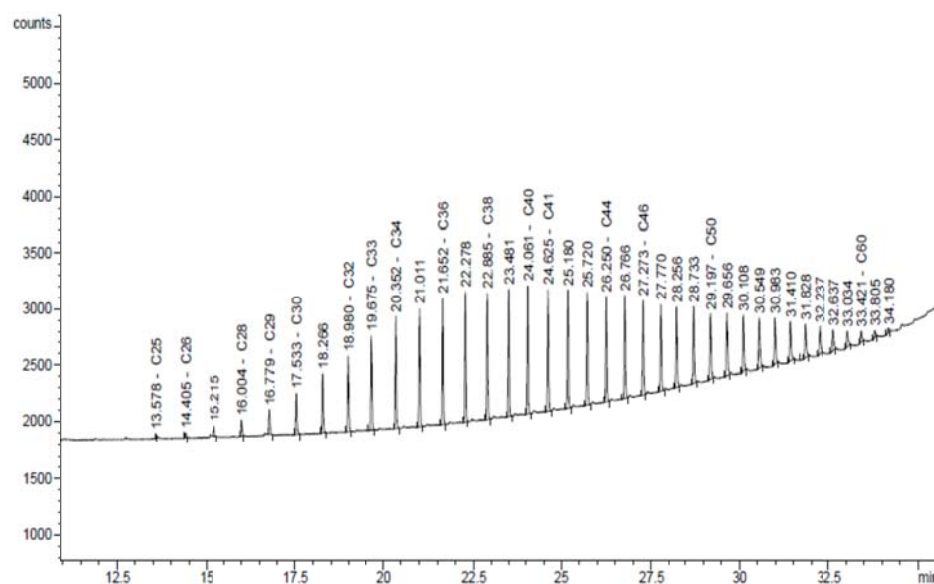


Figure 1.3: Carbon number distribution of FT wax by GC analysis [Oschmann, 1998]

The specific composition of paraffin wax depends on the nature of the crude oil. Wax composition may vary from area to area, well to well or from field to field. Some fields produce harder waxes than others due to the presence of heavier paraffins [Shock et al., 1955].

1.2 Waxy Crude Oil Problems

The precipitation of waxy crystals in subsea crude oil transportation lines due to the fact that temperature of crude oil goes below wax appearance temperature or cloud point, can be classified in two categories:

1. Wax deposition
2. Gel formation

1.2.1 Wax deposition

Wax deposition results from precipitation and surface adherence of naturally occurring saturated aliphatic hydrocarbons when the inner wall temperature of a pipe line falls below cloud point. A wax gel layer grows rapidly in thickness and obstructs the flow of oil as shown in figure 1.4a.

At reservoir conditions (above 70⁰C and 50 MPa), the waxes are typically soluble and completely dissolved in crude oil which have low viscosity and behaves as Newtonian fluid. [Singh et al., 2000]

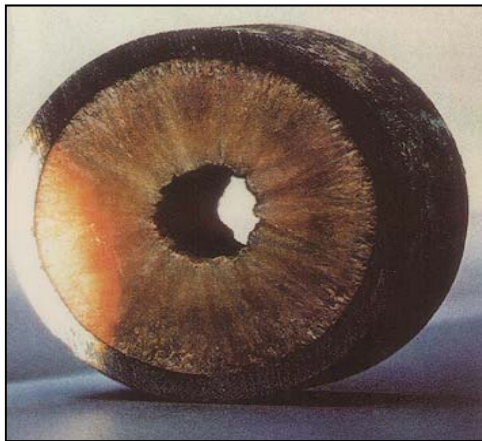


Figure 1.4a: Wax deposition in subsea pipelines
[Singh et al., 2000]

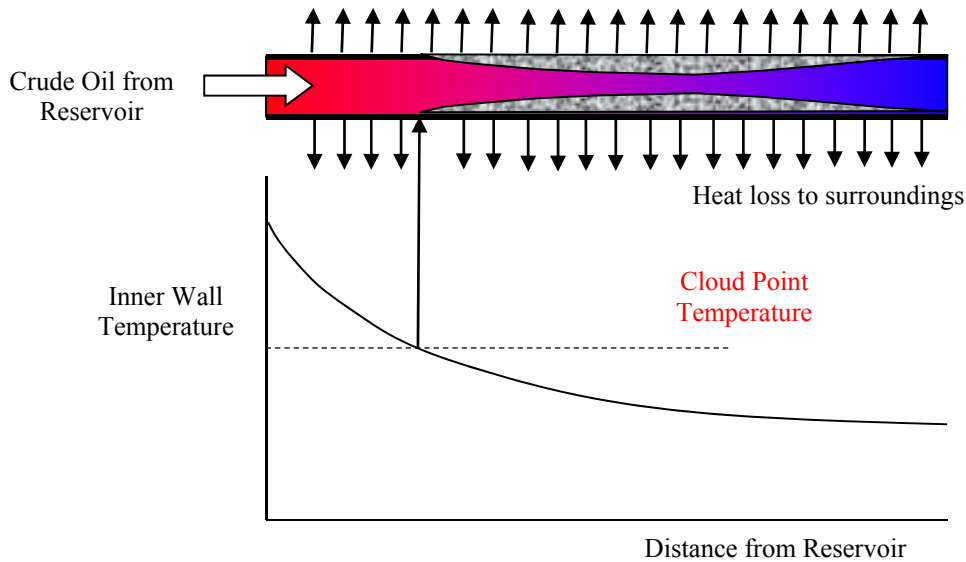


Figure 1.4b: Wax deposition in subsea pipelines [Hyun Su Lee, 2008]

The most severe wax deposition problems are faced in cold environment. When waxy crude oil exits a reservoir and is transported through a subsea pipeline, the fluid temperature is reduced along the length of the pipeline due to heat loss to the cold subsea environment as the temperature of the ocean water is approximately 4°C . The paraffins remain in the crude oil and crystallization does not start as long as temperature of crude oil stays above cloud point. When the temperature of pipe wall reaches cloud point the solubility of paraffins rapidly decreases resulting in the precipitation of wax molecule in the crude oil. These molecules precipitate in the form of solid crystals. As the temperature falls below the cloud point, the formed crystals start to deposit on the inner pipe wall surface. The highest temperature is at the center of subsea pipe line while the lowest temperature is at the inner pipe wall; hence there is a radial temperature gradient which causes the wax crystals to move towards pipe wall. With the passage of time, paraffin

film grows axially towards the center and becomes thicker and harder which reduces the diameter, hence blocking the pipe [Hyun Su Lee, 2008].

Wax crystals nucleate and grow at a temperature below cloud point for a specific pressure and oil composition as presented in figure 1.4b. As fluid pressure is reduced from the reservoir to surface facilities, lighter hydrocarbon components (which serve as strong wax solubilizers) flash off and the molar composition of heavy components in the remaining fluid increases, initiating wax precipitation and deposition [Bratland, 2010].

Wax deposition is a serious economic problem in oil industry causing deferred production losses and remediation costs in an order of billions of dollars. Successful management of wax deposition is the need of time as new offshore explorations are increasing.

1.2.2 Gel formation

When production pipelines are shut-in purposely or unexpectedly for longer time periods, the crude oil in a subsea pipe line is cooled at near-quiescent conditions, thus wax precipitation occurs and result in rapid paraffinic gel formation in the entire cross-section of the pipeline. At low temperatures, paraffin molecules precipitate to form stable orthorhombic crystallites. Flocculation of crystallized paraffins leads to formation of wax-gels with complex morphologies which impart a significant yield stress to fluid. This eventually causes a complete pipeline plugging. In order to restart the flow in gelled pipeline, this wax-oil gel in pipeline must be broken. When evaluating various options for restarting gelled pipeline, it is necessary to estimate the pressure required to break the plug of wax-oil gel. One option is to add chemical agents like

pour point depressants as it weakens the strength of the wax-oil gel [Hyun Su Lee, 2008]. Among other options, applying large pressure at the inlet of gelled pipeline is a good option which results in axial propagation of pressure wave in the flow line, degrading the paraffin wax-oil gel structure.

To correctly understand the phenomenon of pressure wave propagation, inner pipeline segments are considered from an axial perspective. When pressure is applied at the inlet of a flow line, the first segment undergoes compression mode. Consequently large shear stresses develop near the wall which causes the gel to rupture in the first segment. By means of this degradation, the pressure wave propagates to subsequent segments. The same phenomenon occurs in subsequent segments and the pressure wave continues to propagate. If the pressure wave is able to reach the outlet of the flow line, large scale flow may commence. Fluid motion in pipelines during pressure wave propagation occurs by compression flow which indicates that shearing rates are very low, on the order of 10^{-4} sec^{-1} [Paso et al., 2009b].



Figure 1.5: Gelled pipeline

Figure 1.5 shows a gelled pipeline and how wax has blocked the whole pipeline. Due to gel formation in pipelines, huge problems and losses in terms of production as well as investments has taken place and this number is increasing as oil exploration and production are being taken farther offshore.

1.3 Wax Prevention Methods

Significant research and development efforts have been directed to wax gel formation in subsea pipelines. About 80 years ago, paraffin deposition was reported by Reistle (1928, 1932). There are many methods available to manage wax deposition but the most effective method is to prevent continued wax build-up management. Techniques include:

- Maintaining a high temperature in the pipeline (above WAT)
- Insulation
- Timely mechanical removal (e.g., Pigging)
- Chemical inhibitors, pour point depressants and dispersants

The method of maintaining a high fluid temperature by heating insulated crude oil pipelines between the wellhead and platform to prevent nucleation of wax crystals is an excellent method to manage wax deposition. The heating method was proposed by Quenelle and Gunaltun in 1987. They proposed several types of heat insulation coating systems for the pipelines. Other methods include utilizing non-stick internal coatings in pipelines [Patton 1970; Bummer 1971]. The most common way to deal with wax deposition is mechanical pigging of a pipeline at regular intervals [Wang et al., 2001].

Implementation of preventive and remedial management methods has eased problems arising with the advent of extremely deep

subsea production, offshore drilling and ocean floor completions. The use of chemical additives or inhibitors is becoming more prevalent and economical.

1.4 Focus of the Research

The main focus of this project is a fundamental investigation for identification mechanisms of novel cloud point depressants in model oil systems. These model oil systems are prepared with and without polymer additives in the laboratory.

Several pour point depressants were used for this purpose containing high molecular weight polymers as active component which change wax crystallization process. These polymers are formulated in solvents and added to model waxy oil in different concentrations. The subsequent thrust of the research is to investigate the mechanisms of cloud point depressants whether they are thermodynamic, kinetic or steric in nature. Development of novel experimental setup which includes double walled glass vessel with control of thermal and mixing conditions was prepared for this purpose and is a major part of this research work. The model oils will be loaded, dissolved, cooled and sampled for analysis in spectrofluorometer, NMR and GC.

2 LITERATURE REVIEW

This chapter provides an overview and background of the fundamentals of wax deposition and restart flow phenomena and up-to-date advancements in the understanding of the main mechanisms of cloud point depressants/pour point depressants.

Crude oil consists of a number of hydrocarbon components, which can be distinguished in four classes known as SARA:

- Saturates
- Aromatics
- Resins
- Asphaltenes

Saturates consists of the normal, isomerized and cyclic alkane components of a petroleum fluid. Saturated hydrocarbon components with a number of carbon number atoms greater than 19 are commonly known as paraffin waxes, and form crystals at room temperature conditions [Paso, 2005]. The viscosity of the fluid increases and crude oil behaves as a non-Newtonian fluid. Some fundamental properties of waxy crude oils that indicate the temperature range over which a waxy crude oil exhibits non-Newtonian behavior are as follows.

2.1 Pour point

Pour point of crude oil is the temperature at which crude oil solidifies, in other words, the lowest temperature at which flow of crude oil ceases, as defined by ASTM standards [Hansen et al., 1991]. During

continuous production of crude oil, pour point temperature is usually not attained but in case of shut in, crude oil temperature may drop below the pour point and hence there is a need to reduce pour point of waxy crude oils to improve transportability.

ASTM-5853-95 and ASTM D-97 are the procedures used to measure pour point.

2.2 Cloud point/Wax appearance temperature

Cloud point also known as wax appearance temperature (WAT) is the temperature at which wax molecules do not remain in equilibrium and starts to crystallize. In other words, solubility limit of paraffins is reached at cloud point and first wax crystals precipitate out of solution.

Cloud point is a thermodynamic quantity and it does not depend on operating conditions. It can be determined by following techniques:

- Nuclear Magnetic Resonance (NMR)
- Cross Polarized Microscopy
- Differential Scanning Calorimetry (DSC)
- Rheometer

2.3 Gelation temperature

The gelation temperature is the temperature at which the waxy crude oil transforms into a gel structure. Usually pour point is considered as the temperature at which the gel structure is formed but if the gel structure is formed in absence of any stress, that temperature is also called gelation temperature [Venkatesan et al., 2002]. However, gelation typically occurs between wax appearance temperature and pour point if we apply stress. Gelation temperature strongly depends upon operating conditions, such as shear history and thermal history [Singh et al., 1999].

Rheometer is mostly used to measure gelation temperature, as it is capable of applying stress to waxy crude oil.

2.4 Chemicals/Inhibitors way of wax prevention

When crude oil is transported through a flow line, the primary flow assurance problem is continuous deposition of wax on the inner surface of pipeline due to radial temperature gradients. When subsea pipeline is shut-in, the primary flow assurance problem is wax gelling due to temperature reduction. Chemical inhibitors should precipitate at the same temperature range as the wax precipitates. These inhibitors should adsorb on crystal surfaces and provide steric hindrance [Kelland, 2009].

Wax Inhibitors are sometimes added to waxy oils to minimize transport problems of waxy crude oils in subsea pipelines where temperature can go below wax appearance temperature. Chemical inhibitors can be classified into three main groups: [Pederson and Ronningsen, 2003]

- Wax crystal modifiers
- Detergents
- Dispersants

Detergent and dispersants are basically surface active agents. Its main purpose is to keep the crystals dispersed as separate particles so that these crystals do not come in contact and form gel. These also help in modifying the surface of pipe wall hence reducing the tendency of wax crystals to adhere to solid surfaces.

The other major group used as inhibitors are wax crystal modifiers. It is believed that these hinder the formation and growth of wax crystal by modifying the structure of the crystals formed. They do so by

incorporating themselves at the edge of growing wax crystal and changing its growth and surface characteristics as illustrated in figure 2.1.

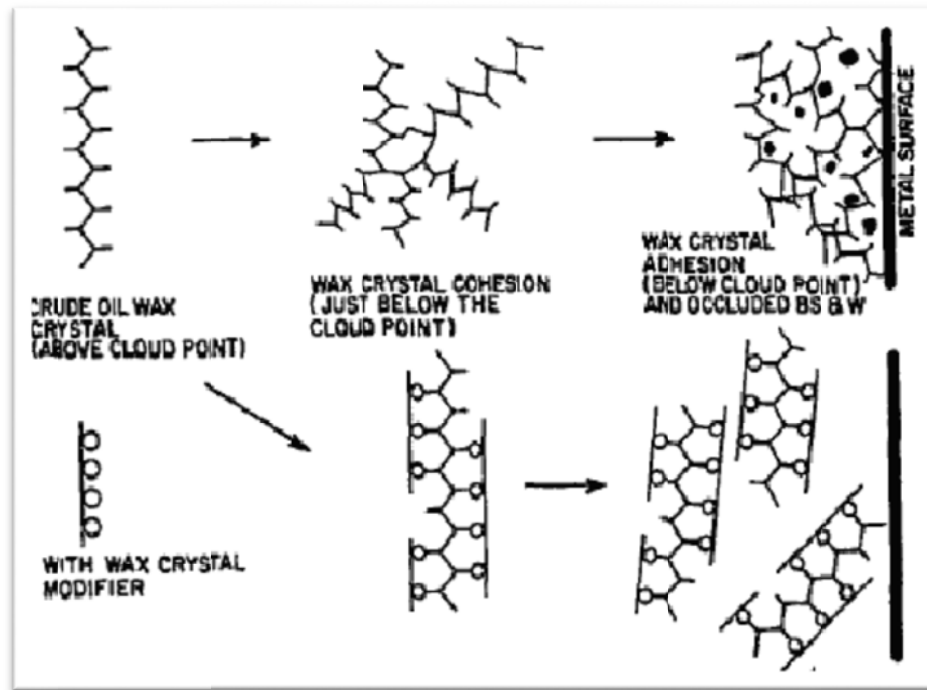


Figure 2.1: Effect of Wax crystal modifiers on wax deposition
[Bilderback and McDougall, 1969]

Therefore it lowers pour point as well as viscosity of crude oil. When the main purpose is to reduce the viscosity or prevent crude oil from gel formation, than those wax crystal modifiers are also termed as pour point depressants (PPD) [Jennings and Breitigam, 2009]. All pour point depressants are also known as cloud point depressants but not all cloud point depressants act as pour point depressants.

The active chemistries of PPDs are specialty polymers that alter wax crystallization process which in turn changes the characteristic of wax deposits and wax gel networks. Usually these polymers are dissolved in a solvent.

Performance of PPDs depends upon many factors such as:
[Jennings and Breitigam, 2009]

- Crude Oil composition
- Temperature and Pressure conditions
- Type of active polymers and its amount applied
- Thermal history

One additive mechanism is common to both inhibitors and PPDs that is crystal habit modification. Wax inhibitors may function as PPDs and vice versa. It has also been reported that several wax inhibitors are effective in reducing wax deposition but are not been able to eliminate deposition and weaker wax deposits may form, but weaker deposits may be easily removed by shear forces in the flow field [Jennings and Weispenning, 2006]. The formulations and application method can strongly influence the performance of PPD

2.5 Main types of Chemicals/Inhibitors

Chemicals which can be used to prevent subsea pipeline from wax deposition and gel formation, can be classified as follows:

2.5.1 Ethylene polymers and copolymers

Ethylene polymers used for the inhibition of wax are Poly (ethylene butane) and poly (ethylene-b-propylene). Tinsley et al. [2007] studied the effect of ethylene polymers on wax inhibition and found appreciable results.

Copolymers included in this category are:

- Ethylene/small alkenes copolymers
- Ethylene/acrylonitrile copolymers
- Ethylene vinyl acetate (EVA) copolymers

EVA copolymers are the most widely used ethylene copolymer. EVA's effectiveness is related to the presence of vinyl acetate. If the vinyl acetate content in the copolymer is increased, the polarity will also increase. Because of high polarity, the solubility is increased and hence crystallinity is reduced which is required for WAT reduction. On the other hand, pure polyethylene has a small effect on crystallization process as it co-crystallizes with structurally similar wax [Kelland, 2009].

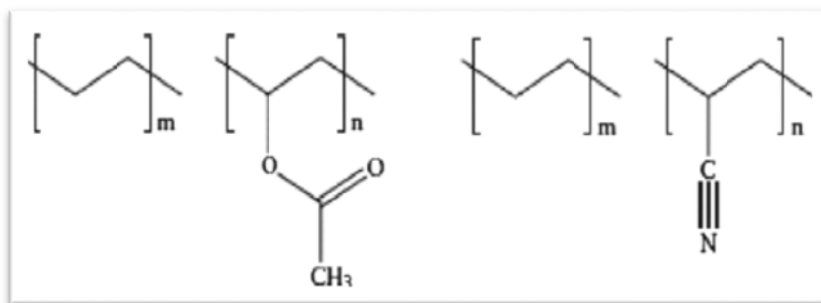


Figure 2.2: Ethylene vinyl acetate (left) and ethylene/acrylonitrile copolymers (right) [Kelland, 2009]

2.5.2 Comb polymers

Comb polymers have been investigated by several researchers and are considered as the most effective wax inhibitors. Wax inhibitor having comb-like structure with side arms of such a length as to interact favorably with the oil fraction will assist in reducing formation of harder wax [Jang et al., 2007]. These chemical inhibitors do not completely eliminate wax deposition. Therefore, mechanical techniques such as pigging are required along with inhibitors [Bello et al., 2006]. Some common monomers used in preparation of effective comb polymers are as: [Kelland, 2009]

- (Meth)acrylic acid
- Maleic anhydride
- Both

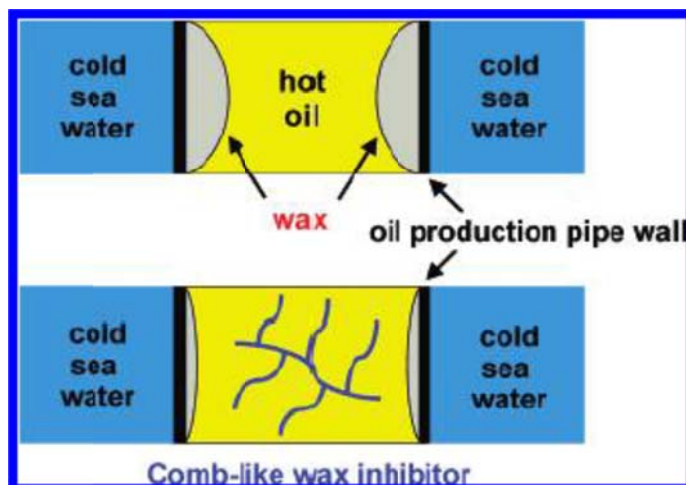


Figure 2.3: Wax formation (upper) and its inhibition by comb-like polymer (lower) [Jang et al., 2007]

Soni et al. [2008] investigated four new comb-shaped maleic anhydride co-polymeric diesters with aromatic and aliphatic pendent chains and concluded that all four are highly effective and can be used as PPDs. This important result shows that by adding suitable polymer additives, transportability of crude oil can be improved.

2.5.3 Miscellaneous polymers with long alkyl groups

High molecular weight material produced from reaction of phosphoric ester (having long chain hydrocarbons) with sodium aluminates are very efficient in lowering of WAT at an optimum concentration [Gentili et al., 2005]. Another polymer that can be used as flow improver is Alkyl phenol formaldehyde [Kelland, 2009]. Branched polymers also fall under this category e.g., the polymer obtained from the reaction of branched polyethyleneimine with 1,2-epoxyoctadecane or dendrimeric hyperbranched polyesteramides performs well as wax inhibitors [Kelland, 2009].

2.5.4 Paraffin Dispersants

Dispersants are surfactants. The mechanism of paraffin dispersant is to penetrate into wax oil gel, get adsorbed on individual particle, and reduce the tendency of wax particles to stick together. Alternatively, the dispersants may adsorb on the inner surface of a pipeline and reduce the ability of wax to adhere to pipe wall. There are several associated mechanisms:

- Creating a water-wet pipe surface
- Creating a weak layer on the pipe wall, allowing wax crystals to adhere onto the layer instead of directly to the pipe surface, allowing removal by turbulent flow

Dispersants are often blended with polymeric inhibitors to improve the additive performance. Low cost wax dispersants include alkyl sulfonates, fatty amine ethoxylates and other alkoxyated products. These products function well only when blended with polymeric inhibitors [Kelland, 2009]. Groffe et al. [2001] have developed a new dispersant P5 which interferes with wax crystal growth and reduces pour point as well as improves the flow characteristics of crude oil.

2.5.5 Polar crude fractions

In searching for low cost flow improvers, it has been observed that polar extracts from crude and distillate oil may be considered as a feasible option. Polar crude fractions consist of asphaltenes, resins and aromatics, and can be extracted by using superficial gases such as carbon dioxide [Kelland, 2009]. It has also been observed that asphaltenes depress the gelation temperature of waxy oils [Venkatesan et al., 2003] and inhibits the precipitation of wax, in a manner similar to comb polymers [Kriz and Andersen, 2005].

2.6 General Perception about PPD mechanisms

PPDs are most popular flow improvers which affect crystal growth, modify wax crystal structure and prevent the interlocking of wax crystals, thus enhances the flow properties of crude oil. It is believed that PPDs co-crystallize with wax, hence creating faults in wax crystals which change the shape and size of wax crystals and prevent the interlocking of crystals into different shapes. [Taraneh et al., 2008]

The exact way in which PPDs operate is not very clear but their basic effect is to modify crystal morphology. There is certainly a combination of different mechanisms involving:

- Nucleation
- Co-crystallization
- Adsorption

The effect of PPDs is nicely explained in figure 2.4.

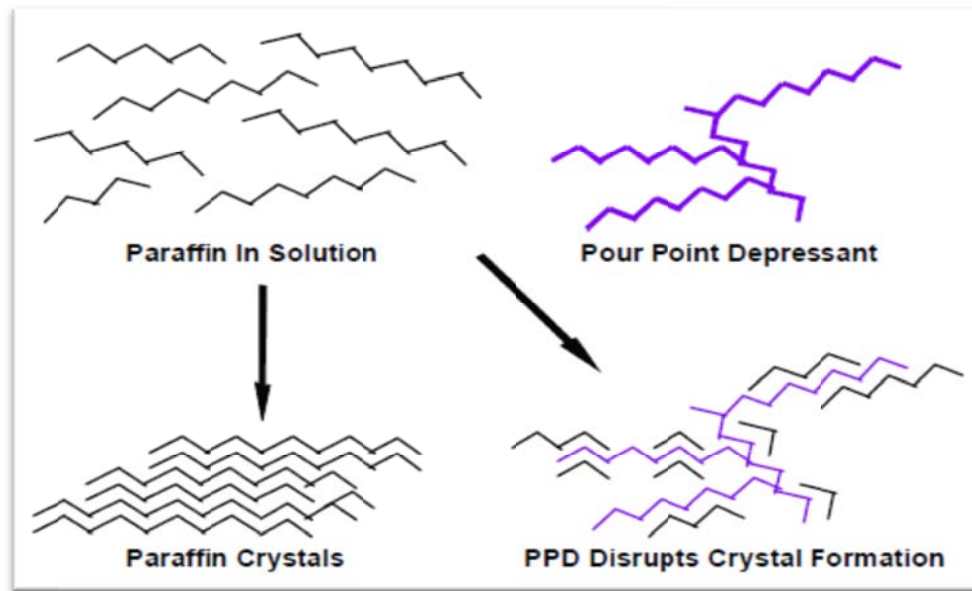


Figure 2.4: Idealized scheme of the mechanism of pour point depression. PPD interact with paraffin in the crude to disrupt crystal formation [Manka et al., 1999]

2.7 Background Research and Development

Many researchers have been working on wax crystals modifiers or specially PPDs to find out their action mechanisms.

Knox et al. [1962] was of the view that PPDs lower pour point of waxy crude oil by preventing needle star (spherulite) formation.

Lorenzen [1962] claimed that alkylated aromatics present in PPDs lower pour point by creating spherulites. He also noticed that not only size of one crystal reduces but it is more important that total surface area is reduced by formation of aggregates.

Birdwell [1964] followed the research of Lorenzen and found that most pour point reduction was accompanied by formation of relatively large crystal aggregates.

Holder and Winkler [1965] studied wax crystallization of n-paraffins from middle distillate fuel oils and reached to the conclusion that if a specific polymer is present in waxy oil in an optimum concentration, the crystal growth habit of wax is changed from lozenge shaped plates to dendrite and other forms. With change in crystal shape, the ability of waxy crystals to form gel is also decreased significantly resulting in lowering pour point of crude oil.

Bilderback and McDougall [1969] believed that there are three mechanisms by which wax crystal modifiers helps to modify wax crystal precipitation from crude oil:

1. The modifier comes out of solution at a temperature slightly higher than the cloud point temperature of the wax solution and causes nucleation.
2. The modifier comes out of solution at cloud point of waxy solution and co-crystallizes with the wax crystals.

3. The modifier comes out of solution at a temperature slightly lower than the original cloud point temperature and adsorbs to the wax crystals.

They mainly concluded from their laboratory testing that chemicals modify wax crystals precipitating from crude oil solution resulting in lowering of the cohesive forces between wax crystals themselves and in many cases, lowering of adhesive forces between wax crystal and pipe wall surfaces occurred. These in turn reduces pour point.

Mendell and Jessen [1970] studied the effect of pour point depressants, crystal modifiers and paraffin inhibitors on flow characteristics of crude oil at temperatures below the cloud point and correlated change in flow characteristics with deposition tendency. They determined that chemical additives exhibited varying degree of effectiveness, depending on the specific crude oil.

Fullford [1975] investigated the effect of polyethylene, ethylene copolymers, aromatics (such as xylene and naphthylene) as well as various surfactants (such as oil soluble alcohols, organic acids, esters, ethers, amines or a mixture of these chemicals) on preventing wax deposition. He concluded that low molecular weight ethylene copolymers prevent paraffin deposition while amorphous polyethylene and low molecular weight aromatics do not prevent paraffin formation and surfactants provide 0-90% inhibition.

Gillby [1983] proposed that the basic mechanism of action of PPDs is a combination of different mechanisms involving nucleation, co-crystallization and adsorption.

Other researchers including Addison [1984], Newberry and Barker [1985], Fielder and Johnson [1986] and Singhal et al. [1991]

found that chemical inhibitors may be used for cost effective paraffin control.

Suryanarayana et al. [1990] proposed that flow improvers modify the orientation of aliphatic portion of resin, wax and asphaltene and don't interact with polar groups.

Ronningsen et al. [1991, 2001] has observed a correlation between tendency to form spherulitic aggregates and ability to lower pour point.

According to Chanda et al. [1996, 1998], a number of polymeric compounds are used as flow improvers for waxy crude oil but the most widely used cold flow improver is ethylene vinyl acetate (EVA) copolymer. Wax is not the only component in crude oil, others are asphaltenes, resins, lighter distillates, polar aromatics etc. should also be considered as important factors while ascertaining the flow behavior of a crude oil.

Qian et al. [1996] observed that the apparent viscosity reduction and pour point depression of doped crude oil increases with decreasing coil dimension of EVA in parent solution.

Manka and Ziegler [2001] observed that additives function optimally when they are tailored to the paraffin distribution in the crude. Solvent type and dilution have no appreciable effect on the performance of wax control products. Carmen Garcia [2001] presented an experimental methodology for deposit characterization and paraffin deposition control treatments, oriented towards identification of "tailor made" preventive or remedial actions, appropriate for specific crude oil compositions and physiochemical properties. They conclude that the efficiency of specific paraffin inhibitor products is strongly correlated to crude oil composition.

Machado et al. [2001] has studied the performance of EVA copolymers and proposed that its activity strongly depends on copolymer composition and there is an optimum concentration to obtain the best efficiency.

Wang et al. [2003] presented a new perspective of the wax inhibition problem. Research results showed that most commercial wax inhibitors used to reduce deposition of low molecular weight paraffins (less than C_{35}) have very small or no effect on high molecular weight paraffins. Results of Wang et al. [2003] effectively reveal a larger problem because high molecular weight paraffins form harder wax deposits. The experimental results indicate that inhibitors which efficiently reduce the overall wax deposition also exhibit a maximum tendency to reduce WAT. Wax deposition can be further reduced by the addition of corrosion inhibitor such as oleic imidazoline to treated paraffin wax solutions.

Pederson and Rønningsen [2003] treated waxy North Sea crude oil with twelve different commercial wax crystal modifiers and found that these inhibitors has prominent effect on pour points and apparent viscosity but only minimal effects on wax appearance temperature. However, he concluded that wax inhibition is a strong tool by which waxy crude oil undergo some changes for easier transportation.

Zhang et al. [2004] studied EVA copolymers and tries to find out the requirement of polarity and affinity to alkane molecules of EVA molecules, the carbon atom number of the segment of EVA molecules should be lower than the new carbon atom number of alkane molecules composing wax crystals.

Bello et al. [2006] studied the performance of selected wax inhibitors on Nigerian crude oils and found that commercially available

anti-paraffin chemicals show positive effects on samples of Nigerian crude oils. However trichloroethylene-xylene TEX additive demonstrated improved performance and is also economically feasible.

Jang et al. [2007] studied wax inhibition by comb-like polymers and suggested that effective wax inhibitors should employ comb-like structures in which side chains should be of such length as to interact favorably with the fraction of the oil most likely to crystallize into the solid wax. Similarly, Tinsley et al. [2007] studied the effects of three different polymer additives in paraffinic oil. All polymers reduced the yield stress of gelled wax solution but did not necessarily decrease deposition.

Kuzmić et al. [2008] investigated the efficiency of long chain acrylic ester polymers to improve the flow properties of waxy crude oil and concluded that properties of these additives like pour point depression depends on its polarity, composition and properties of waxy crude oil.

Taraneh et al. [2008] studied effect of four types of ethylene-vinyl acetate copolymer with different molecular weight in five blendings from Iranian waxy crude oil and concluded that asphaltene soluble solvents can improve viscosity of asphaltene rich crude oil.

Chen et al. [2010] studied effect of two PPDs on waxy solution, one is derivative of poly long alkyl methacrylate and other is alkyl naphthalene copolymer. He found out that these PPDs do not prevent paraffin from precipitation but just shift precipitation to a lower temperature as they can partially transform the structure of paraffins from orthorhombic into hexagonal. They also concluded that PPDs lower the amount of precipitated wax but the effect of PPDs reduces or almost vanishes at lower temperatures.

The quest to find PPDs with improved performance is still going on but it has been clear that every PPD is not effective for every crude oil; in short PPDs are very selective.

2.8 Spectrofluorometer

The spectrofluorometer is an instrument which records both excitation and emission spectra. A certain excitation wavelength is selected, and the emission is observed either at a single wavelength or a scan is performed to record the intensity versus wavelength also called an emission spectra. An emission spectrum is the wavelength distribution of an emission measured at a single constant excitation wavelength. Conversely, an excitation spectrum is the dependence of emission intensity, measured at a single emission wavelength, upon scanning the excitation wavelength.

A schematic diagram of a general purpose spectrofluorometer is presented in figure 2.5. This instrument has a xenon lamp as a source of exciting light. Such lamps are generally useful because of their high intensity at all wavelengths ranging upward from 250 nm. The instrument is fortified with monochromators to select both the excitation and emission wavelengths. The excitation monochromator in this schematic contains two gratings, which decreases stray light, that is, light with wavelengths different from the chosen one. In addition, these monochromators use concave gratings, produced by holographic means to further decrease stray light. Both monochromators are motorized to allow automatic scanning of wavelength. The fluorescence is detected with photomultiplier tubes and measured with the appropriate electronic devices. The output is obtained in graphical form usually. [Lacowicz, 2006]

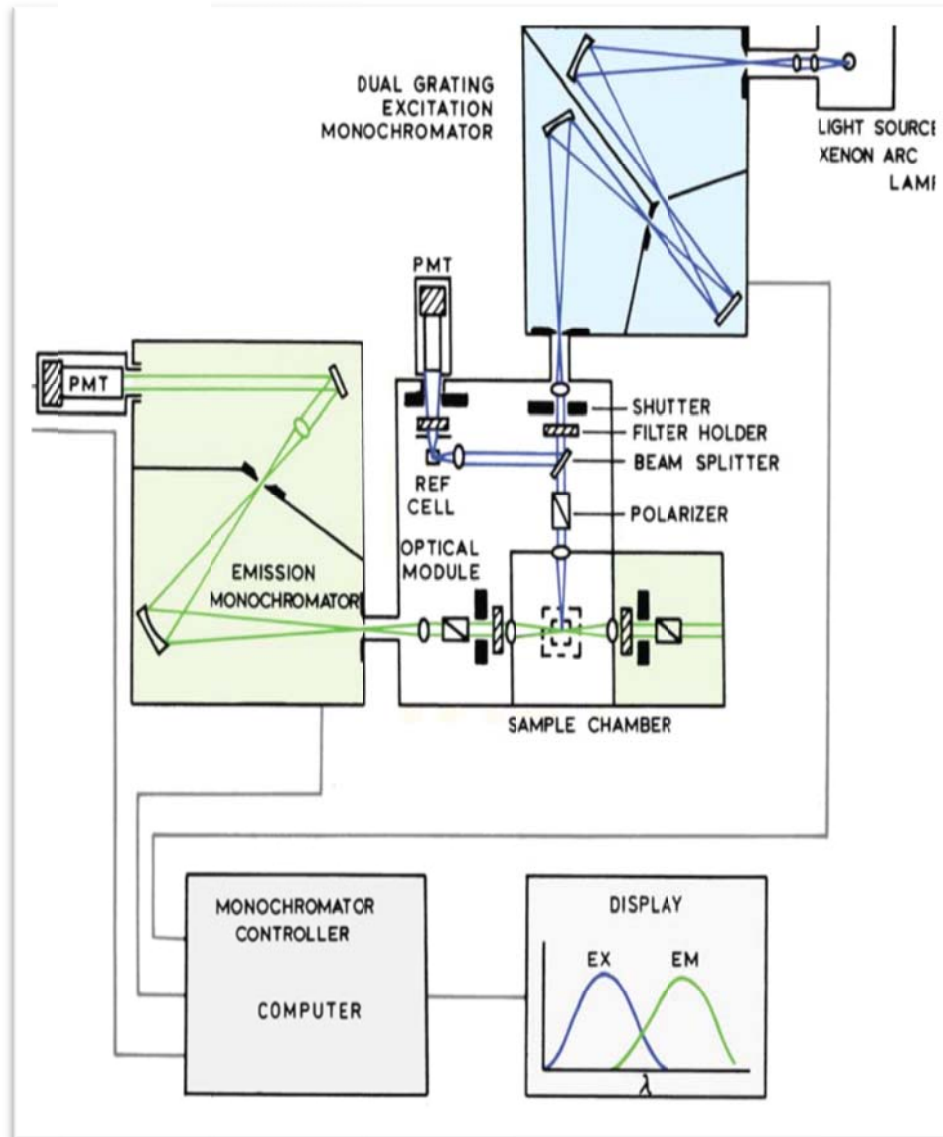


Figure 2.5: Schematic diagram of a spectrofluorometer
[Lacowicz, 2006]

2.9 Nuclear Magnetic Resonance (NMR)

It is a physical phenomenon in which magnetic nuclei in a magnetic field absorb and re-emit electromagnetic radiation.

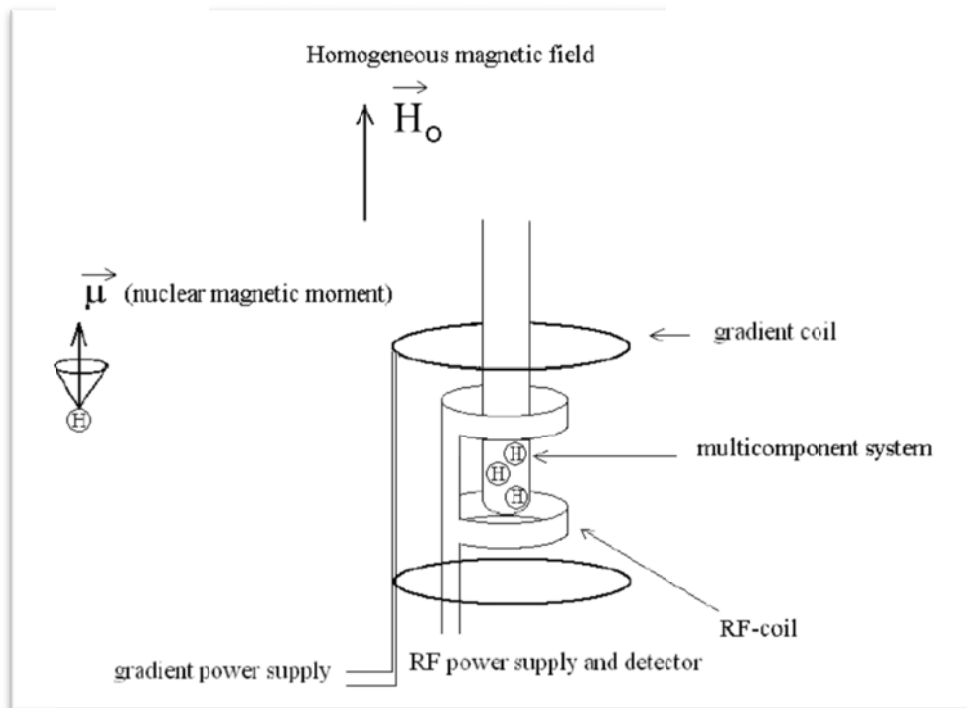


Figure 2.6: NMR setup

When placing hydrogen in an external magnetic field, the nuclear magnetic moment will align towards the direction of this field. The Hamiltonian for non-interacting nuclear magnetic spins in an external magnetic field can be written;

$$H = -\gamma h I H(t) \quad (1)$$

The time dependency of $H(t)$ is included in order to make (L1) valid when the system is influenced by an oscillating magnetic field (RF-field) and magnetic field gradients (g). When the Hamiltonian, $H(t)$, is constant and homogeneous (H_0), the eigenvalues, the energy level of the hydrogen's nuclear spin may be written;

$$E = \pm \frac{1}{2} \gamma h H_0 = \pm \frac{1}{2} h \omega_0 \quad (2)$$

The difference between two energy levels is thus;

$$\Delta E = h \omega_0 \quad (3)$$

In thermal equilibrium a difference population between upper and lower level is given by the Boltzmann factor;

$$\frac{n_{upper}}{n_{lower}} = e^{-\frac{h\omega}{kT}} \quad (4)$$

The difference in population will generate a net nuclear magnetic moment which will depend on the content of hydrogen/proton. In thermal equilibrium the moment will be aligned with the external magnetic field. By imposing an oscillating magnetic field, RF-field, transverse to the external magnetic field H_0 , transitions between the energy levels will occur. The direction of the net nuclear magnetic moment will then move away from thermal equilibrium with the external field. When the RF-field is switched off, the system will again tend to thermal equilibrium with the external field, the direction of H_0 . The speed back to thermal equilibrium is given by the characteristic relaxation times T_1 (longitudinal relaxation) and T_2 (transverse relaxation). The path back to thermal equilibrium in combination with an oscillating net nuclear magnetic moment transverse to H_0 , will cause changes in the magnetic flux which can be recorded with the same RF-coil which was used to excite the system. Assuming that the spins are interacting and diffusing and flowing in an external field the classical equation of motion of the spin system known as the Bloch-Torrey equations.

$$\frac{d}{dt} \overline{M} = \gamma \overline{M} \times H + D \nabla^2 \overline{M} - (v \cdot \nabla) \overline{M} \quad (5)$$

Which may be written as;

$$\begin{aligned}\frac{d}{dt}M_z &= -\gamma M_y H_1 + \frac{M_0 - M_z}{T_1} + D\nabla^2 M_z - (v \cdot \nabla) M_z \\ \frac{d}{dt}M_x &= \gamma M_x h_0 - \frac{M_x}{T_2} + D\nabla^2 M_x - (v \cdot \nabla) M_x \\ \frac{d}{dt}M_y &= \gamma (M_z H_1 - M_x h_0) - \frac{M_y}{T_2} + D\nabla^2 M_y - (v \cdot \nabla) M_y\end{aligned}\quad (6)$$

Where H_1 is the rotating magnetic field with a frequency ω , $h_0 = H_0 + \omega/\gamma$, T_1 is the longitudinal or spin lattice relaxation time. The acquisition of the NMR signal is done by sampling the induced magnetic moment in the transverse plane to the external field, and the phases for the transverse components are affected by diffusion in the presence of a magnetic field gradient. During any z-storage of the net magnetization, there is no effect from diffusion in the presence of magnetic field gradients. When assuming a Gaussian distribution of diffusivities and a mono-exponential attenuation of the NMR signal due to relaxation processes, the solution to the transverse magnetization ($M^+ = M_x + i M_y$) can be written;

$$M^+ = M_0^+ e^{-\frac{t_1}{T_2}} e^{-\frac{t_2}{T_1}} e^{-\gamma^2 D \int_0^t \left(\int_0^{t'} g(t'') dt'' \right)^2 dt'} e^{i\gamma v \cdot \int_0^t \int_0^{t'} g(t'') dt'' dt'} \quad (7)$$

In the following, we will denote M^+ to I and m_0^+ to I_0 . The induced current in the RF coil, the NMR signal, will be attenuated because of dephasing. This will be apparent in the natural logarithm of the pulsed field gradient stimulated echo attenuation $\ln(I/I_0)$ expressed as a function of the diffusion time and the gradient strength.

$$I = I_0 e^{-\frac{t_1}{T_2}} e^{-\frac{t_2}{T_1}} e^{-\gamma^2 g^2 D \int_0^t \left(\int_0^{t'} g(t'') dt'' \right)^2 dt'} e^{i\gamma v \cdot \int_0^t \int_0^{t'} g(t'') dt'' dt'} \quad (8)$$

2.10 Gas Chromatography (GC)

It is a type of chromatography that makes it possible to separate the volatile components of a very small sample and to determine the amount of each component present. Sample has been analyzed into the injection port of the GC device. The GC instrument vaporizes the sample and then separates and analyzes the various components. Each component ideally produces a specific spectral peak that may be recorded on a paper chart or electronically. The time elapsed between injection and elution is called the retention time. The retention time can help to differentiate between some compounds. The size of the peaks is proportional to the quantity of the corresponding substances in the specimen analyzed. [3]

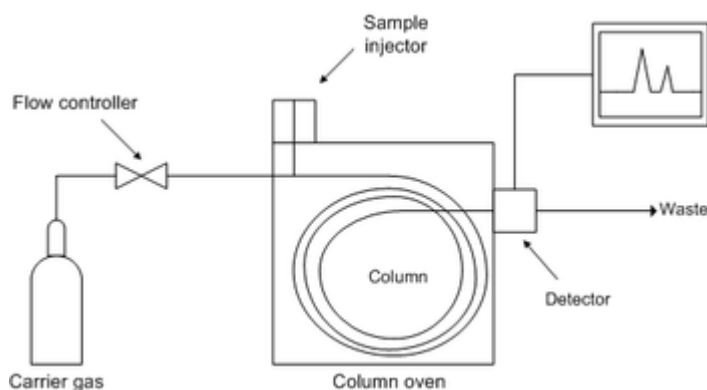


Figure 2.7: Typical GC system [4]

In gas chromatography, a tiny sample is injected onto the end of a column, now a very long, narrow fused silica tube coated with one of many stationary phases, depending on the substance to be determined.

Typically, the material is vaporized in an injector port, and then swept onto the column by helium gas (the flash vaporization method). In

some cases the material is injected directly onto the end of the column itself (the cool, on-column method). In any event, a carrier gas acts as the mobile phase. Most often helium is used, but other gases can be used as well, depending on the technique. The column is suspended in an oven that is temperature controlled. The temperature is typically above the boiling point of the least volatile expected component, but there are exceptions.

At the end of the column, the separated components enter a detector, where they are registered and quantitated. Unlike paper chromatography, most detectors for gas chromatography can also measure the amount of material passing through. The amount of time required for a particular component to pass from the injector port to the detector is called the retention time, and is characteristic for a given component, but there exists the possibility of overlap with other components. Standard solutions of known materials at known concentrations and solvents are run through the instrument before unknowns are run for calibration of retention times and signal intensities.

The detector is connected to some sort of a recorder that takes the signal and plots the shape of it, and calculates the areas but sometimes height of each signal, called peaks. This is compared to the calibration runs, and a quantitative result is calculated and displayed. Modern practice is almost always to use a computer to capture the data. [4]

3 MATERIALS AND METHODS

3.1 Materials

Waxes used in this research are obtained from Sasol wax Co. Ltd those are:

- Fischer Tropsch Sasolwax 5000
- Macro-crystalline Sasolwax 5405

Solvents used in this project are listed below:

- Toluene C_7H_8 supplied by VWR International, LLC.
- Heptane C_7H_{16} supplied by Merck KGaA.
- Methyl cyclohexane C_7H_{14} supplied by Sigma Aldrich Co. LLC.

Safety data sheets for waxes and solvents are presented in Appendix A6. Characteristics of solvents are presented in table 3.1.

<i>Name of Solvent</i>	<i>Melting Point</i>	<i>Boiling Point</i>	<i>Density @ 20 °C</i>
Heptane	-90 °C	98 °C	0.679 g/ml
Toluene	-95 °C	110 °C	0.866 g/ml
Methyl cyclohexane	-126 °C	101 °C	0.77 g/ml

3.2 Samples Preparation

Model waxy oils were prepared with varying concentration of wax from 1w/w% to 10w/w% in different solvents. All samples were weighed by an electronic analytical balance (PB403-S/FACT, Mettler-Toledo Inc., US) with precision of ± 0.001 g.

Subsequently inhibitors were added in each of the model oils. Four different types of PPDs were used. Dosage rates varied from 20 ppm to 1000 ppm. PPDs are provided by Champion Technologies and named as:

- PPD2
- PPD3
- PPD4
- PPD5

3.3 Experimental Setup

Experiments were performed in a double walled glass vessel which is attached to a heating/cooling pump and digital thermometer as shown in figure 3.1. Pump helps to circulate cold or hot water inside the jacket of double walled glass vessel and maintain steady temperature while a digital thermometer gives us accurate temperature reading of the treated model oils present inside glass vessel during the experiment.



Figure 3.1: Double Walled Glass Vessel attached with Heating/cooling Pump and Digital Thermometer. a: Double Walled Glass Vessel, b: Digital Thermometer, c: Filters, d: Heating/cooling pump, e: Inhibited wax oil solutions

3.4 Experimental Protocol

3.4.1 Thermal Protocol

The following thermal protocol was utilized:

1. The model oil was heated for over half hour at about 80°C to assure complete dissolution of wax.
2. Both model oils were dosed with inhibitor (if needed).
3. Treated model oil was again heated to 80°C for about half hour before taking it to double walled glass vessel.

3.4.2 Double Walled Glass Vessel Protocol

1. Heated model oil is transferred in the cylinder of double walled glass vessel and fill up to an optimum level. This glass vessel is already connected with heating/cooling pump which controls the temperature inside glass vessel by pumping water into the jacket of the double walled glass vessel.
2. The temperature of the model oil inside the glass vessel is found by digital thermometer by putting the temperature sensor wire in the model oil.
3. Heat this model oil 20°C above the cloud point. (e.g., if cloud point is 15°C , then model oil in glass vessel should be at 35°C).
4. Cool this model oil quickly from 35°C to 20°C .
5. Then with controlled cooling rate of approximately $1^{\circ}\text{C}/\text{min}$, cool the model oil from 20°C to 4°C or to any desired temperature. This cooling rate is controlled manually.
6. Subsequently, model oil is maintained at 4°C for ten minutes.
7. Collect the sample of about 5 mL by syringe using a filter (either by $0.45\ \mu\text{m}$ or by $0.2\ \mu\text{m}$) from the side of glass vessel (not from the top).
8. Take this sample for analysis to spectrofluorometer.
9. Risk analysis of this experimental setup is given in Appendix A5.

3.4.3 Spectrofluorometer Protocol

1. 10mm light path quartz cuvette was used for measuring fluorescence spectra of the samples. It is washed with toluene two times and with acetone, then dries it properly before loading the sample.

2. Sample (pure solvents; initial model oils and those model oils, which were taken from the glass vessel at desired temperature say 4⁰C by syringe using a filter, etc.) is loaded into quartz cuvette.
3. Steady-state spectra were taken using Jobin Yvon spectrofluorometer with Right Angle (RA) scheme. Emission range 395-750nm is chosen. For each inhibitor optimal excitation wavelength was chosen and applied.

For the analysis of some samples in spectrofluorometer which have low cloud point, in addition of the above written operating procedure, following step was added:

4. Spectra are measured at ambient temperature. For measuring at 35⁰C automatic temperature controller is being used, which is a part of Jobin Yvon spectrofluorometer. Samples are maintained for 10 minutes at given temperature before taking spectra for reaching thermal equilibrium.

3.4.4 NMR Protocol

Following protocol was used with waxy oil samples taken to NMR.

1. First of all, place the sample in NMR and then stabilize sample at a defined temperature (above cloud point).
2. Run a FID NMR experiment (= 90⁰ excitation pulse followed by recording of signal decay) If solid component is present, its signal will decay to an insignificant value within 100 us. The liquid component will decay to an insignificant value at time >> 100 us. Thus the two components can easily be separated.
3. Run a CPMG experiment (= 90⁰ excitation pulse followed by a train of (tau-180-tau-recording). This gives you the T2 attenuation

of the NMR signal, and it consists of liquid component only, as the shortest tau-value is 100 us, and the first signal is recorded at ~200 us). The initial signal thus corresponds to the signal from the liquid component. If a solidification process is taking place, the amount liquid signal will decrease with increasing solidification.

4. This data from FID and CPMG for that temperature has been saved in the computer attached to NMR.
5. Now manually change the temperature to a new one and redo above four steps again. (If a sample has cloud point of 15⁰C, then perform NMR experiments from 20⁰C to 4⁰C by taking reading after each 1⁰C).
6. Then, analyze the results.

3.4.5 GC analysis Protocol

The carbon number distribution (CND) of the paraffins in the crude oil was determined using a gas chromatograph (GC) from Agilent Technologies (6890N) with an SGE (AQ)-HT-5 column (12 m, 0.32 mm, 0.1 μm) and a flame ionization detector (FID). Hydrogen was used as carrier gas (2.3 mL/min). The temperature program started with 2 minutes at 50°C. The oven was then heated to 400°C at 10°C/min and held at 400°C for 15 minutes.

The wax content of the sample was calculated by integrating each peak corresponding to paraffins with carbon number 16 and higher. The peak areas were correlated to wax content by using a calibration based on ASTM standards.

The analysis of the carbon number distribution and the determination of the wax content are based on method ASTM D5442.

4 RESULTS AND DISCUSSIONS

The research was conducted in a series of steps in which the concentration of wax and PPD were varied, different types of PPDs and different solvents were used. Glass vessel experiments were performed with inhibited waxy model oils and samples/probes were collected. These probes were analyzed by using spectrofluorometer, whereas NMR and GC analysis were performed on model wax oil systems.

4.1 Development of Glass Vessel Protocol

The main points for the development of glass vessel protocol were:

- Thermal history
- Temperature profile during the experiment

Before using any model oil, inhibited or uninhibited, it is necessary to remove its thermal history. For that purpose, heat the sample far above its cloud point for a minimum of half hour and then fill the cylinder of double walled glass vessel.

The second major point was to have continuous or step-wise temperature profile of the model oils inside the cylinder of double walled glass vessel. Initial experiments with uncontrolled cooling rate did not give repeatable results. So, step wise cooling rate was suggested. In step wise cooling, we have to set a temperature and then we have to wait until that temperature is reached. After that we set a new temperature with a step 1⁰C difference. This procedure is time-consuming as it takes almost 5-10 minutes on average for every 1⁰C decrease above cloud point. When

temperature passes cloud point, 15-20 minutes is required for each 1⁰C decrease in temperature.

Then continuous cooling rate was suggested. Cooling rate was controlled manually as it is not possible with the type of heating/cooling pump available. A lot of experiments needed to acquire continuous cooling rate of 1⁰C per minute and it gives repeatable results. Hence we have devised a protocol for Glass vessel; see section 3.4.2

4.2 Development of Spectrofluorometer Protocol

Following solutions were prepared at the initial stage of the research work.

- Pure Heptane
- 1000 ppm PPD2 in Heptane
- 1000 ppm PPD3 in Heptane
- 1000 ppm PPD4 in Heptane
- 1000 ppm PPD5 in Heptane

The purpose of preparing these solutions was to develop spectrofluorometer protocol for analyzing inhibited model oil systems, so that we can control concentration of PPD in it. For this purpose, above mentioned solutions were analyzed using spectrofluorometer. In these experiments, slits of 3 – 14 nm were used while excitation wavelength varied from 330 to 500 nm in order to find out the optimum slit and excitation wavelength. Also, the initial experiments were performed at room temperature i.e., at 20⁰C. Obtained spectra were scattered which is due to the fact that room temperature is quite close to the cloud point of the samples. In order to avoid this problem, we raise the temperature to 35⁰C which is about 15-20⁰C above cloud point using temperature

controller, which is installed in Jobin Yvon spectrofluorometer. The data obtained from spectrofluorometer were gathered and then plotted steady-state spectra taking fluorescence signal intensity on y-axis and wavelength (nm) on x-axis as shown in fig 4.1.

About hundred steady-state spectra were taken with different slits and different excitation wavelengths. Spectra for PPD2, PPD3 and PPD4 were obtained using excitation wavelength of 380nm and slits 5 nm. While for PPD5 we suggest to use excitation wavelength 350 nm and slits 5nm. Here the spectra are presented in figure 4.1 with chosen parameters; excitation wavelength 380 nm.

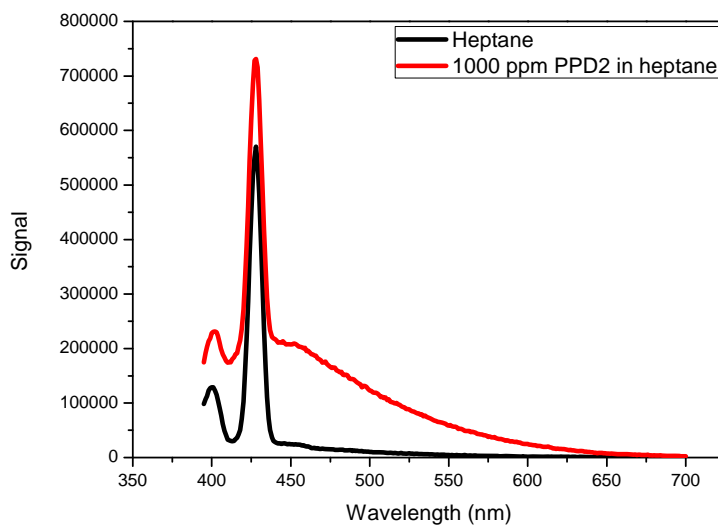


Figure 4.1: Steady-state spectra of 1000 ppm PPD2 in Heptane

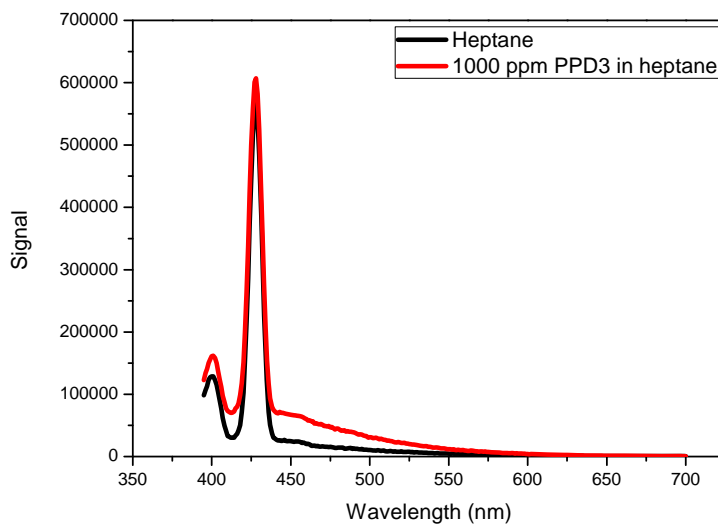


Figure 4.2: Steady-state spectra of 1000 ppm PPD3 in Heptane

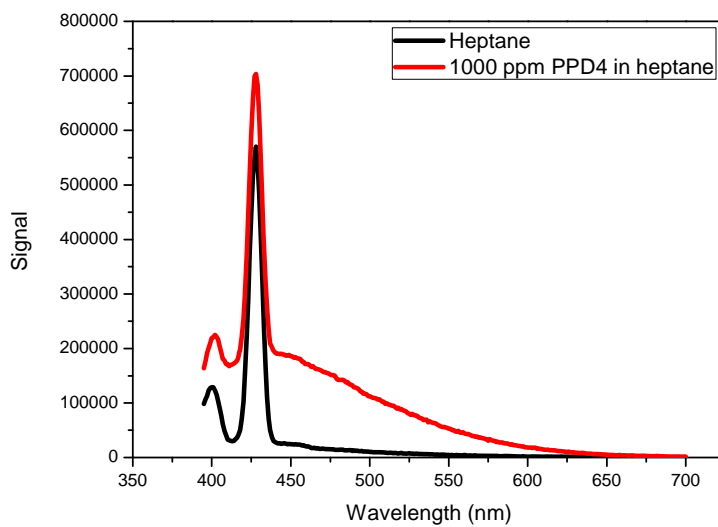


Figure 4.3: Steady-state spectra of 1000 ppm PPD4 in Heptane

Figure 4.2, 4.3 shows that fluorescence signals is increasing for the solutions containing PPDs.

With PPD5, along with 1000 ppm PPD5 in heptane, one more solution of 1000 ppm concentration is being prepared in toluene and analyzed by having excitation wavelength of 350 nm and slits 5 nm along with 1000 ppm PPD5 in Heptane. The steady-state spectra are shown in figure 4.4a, 4.4b and figure 4.5.

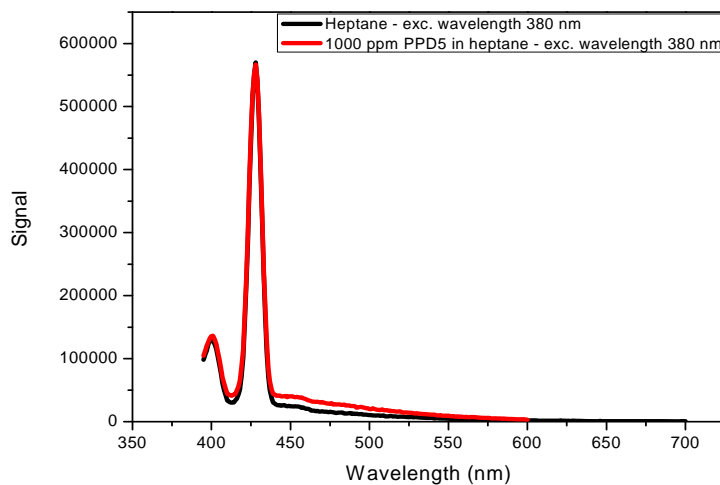


Figure 4.4a: Steady-state spectra of 1000 ppm PPD5 in heptane at excitation wavelength of 380 nm

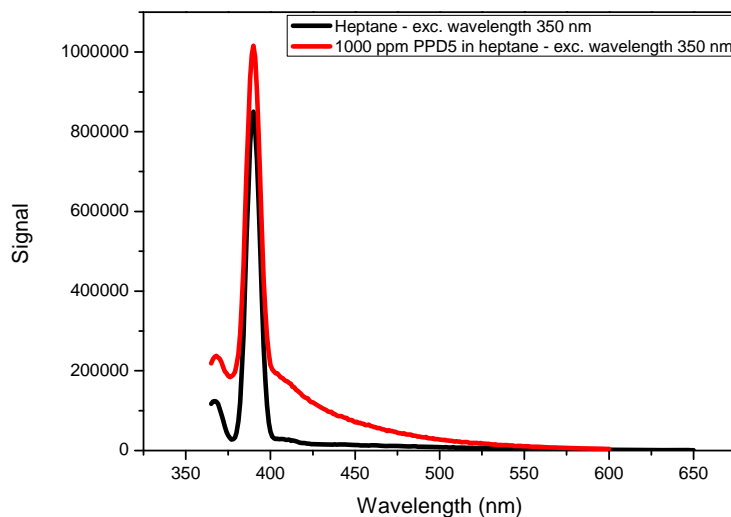


Figure 4.4b: Steady-state spectra of 1000 ppm PPD5 in heptane at excitation wavelength of 350 nm

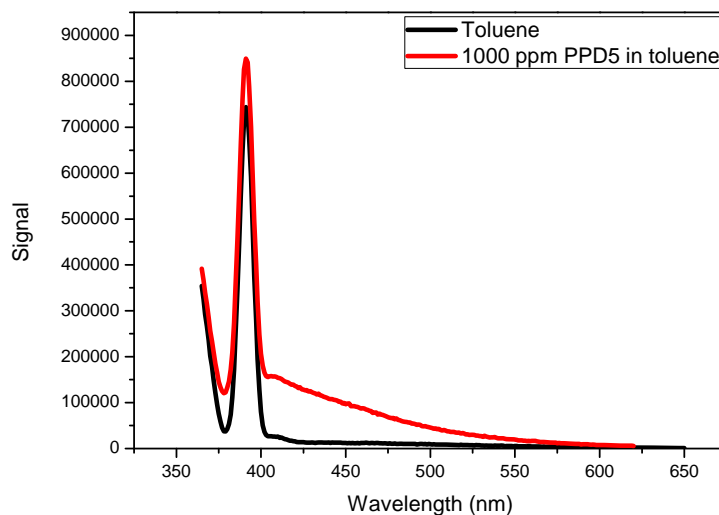


Figure 4.5: Steady-state spectra of 1000 ppm PPD5 in toluene at excitation wavelength of 350 nm

The signal is increasing from pure heptane to 1000 ppm PPD5 in heptane when excitation wavelength is 350 nm and slit 5 nm was used. The same is the case with toluene.

At this point, spectrofluorometer protocol for systems without waxes has been devised; see section 3.4.3 for the final spectrofluorometer protocol. The next step is to see effect of wax composition on spectra when we are working with oil systems containing waxes and PPDs.

4.3 Effects of wax type on steady-state spectra of Model oils

To see the effects of different waxes on solvents, various waxy solutions were prepared with FT wax and macro-crystalline wax 5405 and analyzed.

4.3.1 Fischer-Tropsch (FT) wax

(1) 500 ppm PPD2 in “5w/w% FT wax in Toluene”:

- A solution of 500 ppm PPD2 in “5w/w% FT wax in Toluene” was prepared.
- Its cloud point was measured using visual observation procedure and it was 14⁰C.

Cloud point of “5w/w% FT wax in Toluene” (no inhibitor) was 16⁰C.

- Glass vessel experiment was performed on it and probes were taken at 14, 10, 8 and 4⁰C by using filter 0.45 μ m. (When one probe was taken at 14⁰C, the model oil was heated once again and it follows same complete procedure to take another probe at 10⁰C).
- Then these probes were analyzed by spectrofluorometer and the results are presented in figure 4.6.

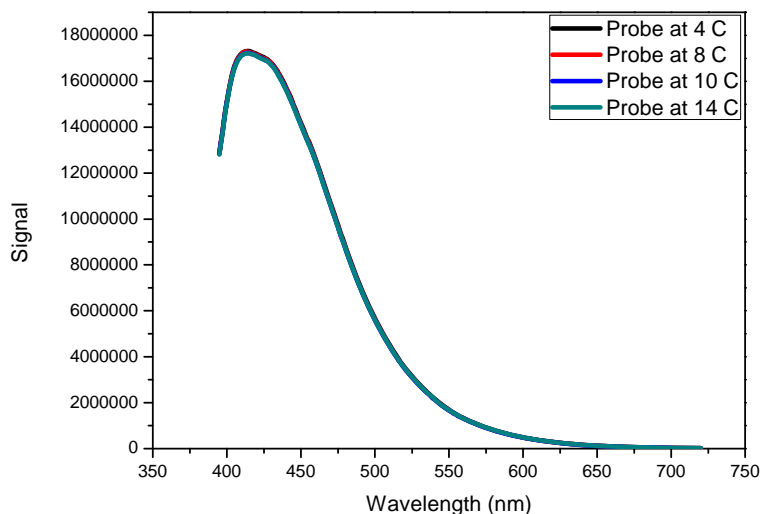


Figure 4.6: Steady-state spectra of Probes taken at 4, 8, 10 and 14 ⁰C from 500 ppm PPD2 in 5w/w% FT wax in toluene

The steady-state spectra obtained at ambient temperature show that there is not much difference in the spectra even with the probes taken at different temperatures. Also, the signal intensity values are much higher and are out of confidence range of the equipment. Increasing temperature to 35⁰C (20⁰C above cloud point) doesn't give any changes to spectra.

To see the effect of presence of FT wax, following solutions were prepared:

- 500 ppm PPD2 in Toluene
- 500 ppm PPD2 in "5 w/w% FT wax in Toluene"

Comparison of steady-state spectra of above mentioned solutions is presented in figure 4.7. The steady-state spectra without FT wax (black) lies within confident interval and out-of-range spectra for solution containing FT wax (red) that lies higher.

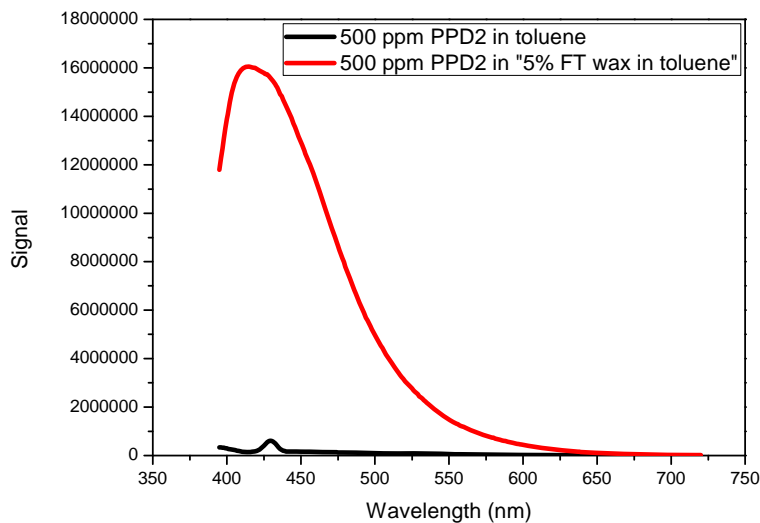


Figure 4.7: Steady-state spectra of "500ppm PPD2 in 5w/w% FT wax in Toluene" and "500ppm PPD2 in Toluene"

Out of range spectra for FT wax might be due to the fact that FT wax is Fischer-Tropsch paraffin wax, which is usually a mixture of n-alkanes, but there is a version that there are double bonds present as well along with n-alkanes. In molecules with alternating double bonds and single bonds, p-orbital overlap can exist over multiple atoms in a chain, giving rise to a conjugated system, which might give fluorescence. It leads to amplification of detected fluorescence signal. This explanation could be verified by gas chromatography analysis.

Another explanation might be the aromatic nature of the solvent – toluene, which may not be a proper solvent for FT wax. It could influence solubility of FT wax and quality of obtained spectra.

In order to understand better the behavior of FT wax we reduce the concentration of PPD2 from 500 ppm to 100 ppm in model oil of 5w/w% FT wax in Toluene and performed glass vessel experiment on it and then analyzed it using spectrofluorometer.

(2) 100 ppm PPD2 in “5w/w% FT wax in Toluene”:

- 100 ppm PPD2 in “5w/w% FT wax in Toluene” was prepared.
- Its cloud point was 14⁰C by visual observation procedure.
- Step wise cooling rate was decided. Temperature cooled quickly from 35⁰C to 20⁰C and then step wise cooling to 4⁰C.
- Glass vessel experiment was performed and probes were taken at 10⁰C and at 4⁰C with two different filters of 0.45 μm and 0.2 μm.
- These probes were analyzed under spectrofluorometer.
- The result was the same that fluorescence signal for FT wax was still far out of range.

Reducing the concentration of PPD doesn't help our cause so it was decided to reduce the concentration of FT wax from 5w/w% to

1w/w% and performed glass vessel experiment on it and then analyzed it using spectrofluorometer.

(3) 1w/w% FT wax in Toluene:

- This solution was prepared.
- Stepwise cooling rate was not feasible for our experiments so we switched to continuous cooling rate.
- I haven't measured its cloud point because we were trying to figure out, how to get 1⁰C continuous cooling rate and it was just a test run to see the effect of FT wax.
- The temperature goes quickly from 35⁰C to 20⁰C and @1⁰C/ min to 4⁰C.
- The probe was taken at 4⁰C.

The result indicates that decreasing of the content of FT wax doesn't influence steady-state spectra and fluorescence signal for FT wax is still out of range.

4.3.2 Macro-crystalline Sasolwax 5405

The results from spectrofluorometer were clearly indicating that the graphs of model oil containing FT wax are out of range. Instead of FT wax, Sasolwax 5405 was used for next experiments. We prepared 500 ppm PPD2 in "5w/w% 5405 in Heptane" and then compared its spectra with 500 ppm PPD2 in "5w/w% FT wax in Toluene". Result is presented in figure 4.8.

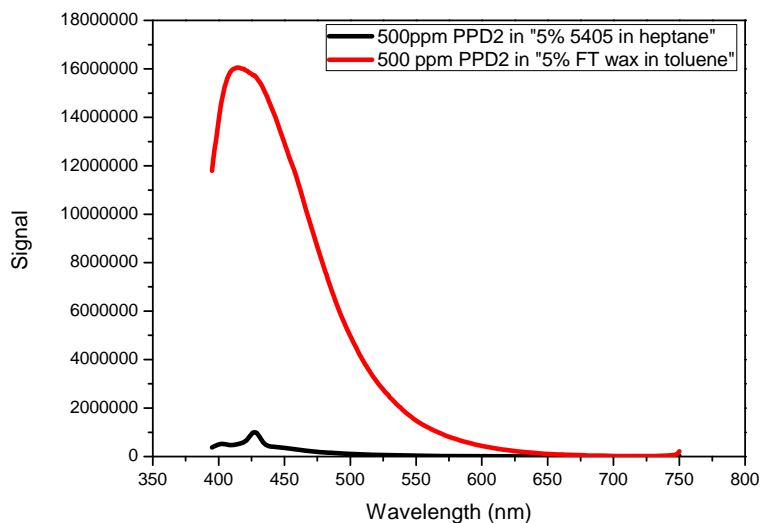


Figure 4.8: Steady-state spectra of “500ppm PPD2 in 5w/w% 5405 wax in heptane” and “500ppm PPD2 in 5w/w% FT wax in toluene”

Spectra of 500 ppm PPD2 in “5w/w% 5405 in heptane” is in the working range as compared to the other spectra. So we decided to use macro-crystalline 5405 wax in our Model wax oil.

4.4 Effect of solvents on steady-state spectra of Model oils

We employed three different solvents i.e., heptane, toluene and methyl cyclohexane. First of all, pure samples of all three solvents were analyzed using spectrofluorometer. Figure 4.9 presents the result.

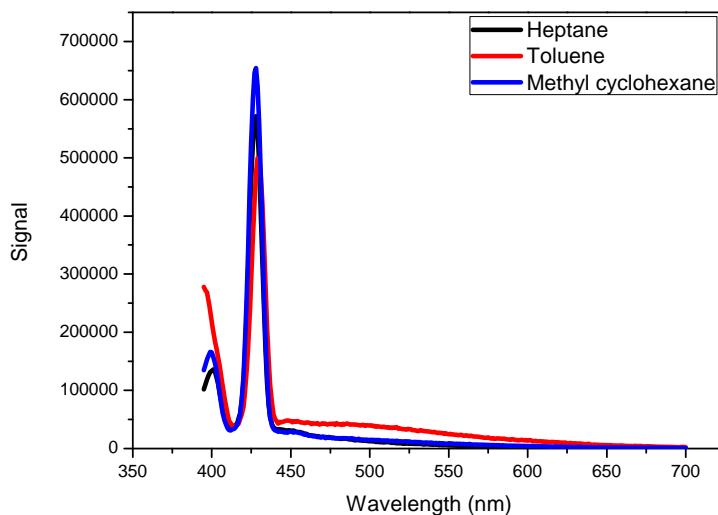


Figure 4.9: Steady-state spectra of heptane, toluene and methyl cyclohexane

All spectrums are in the reliable range and lies very close to each other; this indicates that these solvents can be used for our Model wax oil system.

Then both type of waxes i.e., FT wax and macro-crystalline wax 5405 were added in solvents. Following solutions were prepared and analyzed using spectrofluorometer. Due to toxicity reasons, for future experimental work toluene was not chosen as a solvent.

- 5w/w% FT wax in methyl cyclohexane
- 5w/w% FT wax in heptane
- 5w/w% 5405 in methyl cyclohexane
- 5w/w% 5405 in heptane

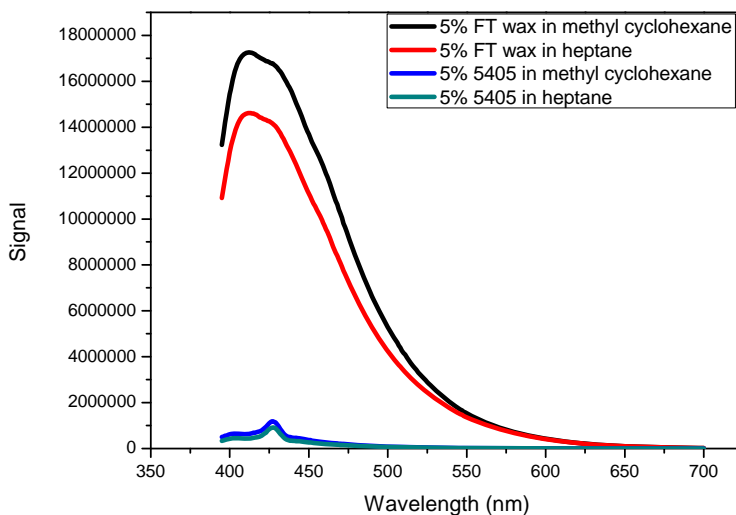


Figure 4.10: Steady-state spectra of 5w/w% FT wax in methyl cyclohexane, 5w/w% FT wax in heptane, 5w/w% 5405 in methyl cyclohexane, 5w/w% 5405 in heptane

Figure 4.10 shows that effect of both solvents are same even with waxes i.e., fluorescence signal does not change with the change of solvent. This graph reconfirms previous result about FT wax that this type of wax gives out of range spectra which is clearly shown in it. While for Sasolwax 5405, the spectra are in the range. As both solvents, heptane and methyl cyclohexane gives us adequate results with 5405 wax, so any solvent can be used. Further experiments were carried out with heptane.

4.5 Effect of PPDs on Model wax oil system

With the help of earlier results, it has been decided that in Model wax oil, macro-crystalline wax 5405 will be used in heptane. The concentration of wax is 5 w/w% and hence the model wax oil is “5 w/w% 5405 in heptane”. To see the effect of different PPDs on this model wax oil system, we added PPDs in it and then analyze them.

4.5.1 PPD2

Following solutions were prepared:

1. 5w/w% 5405 in Heptane
2. 500 ppm PPD2 in “5w/w% 5405 in Heptane”
3. 400 ppm PPD2 in “5w/w% 5405 in Heptane”
4. 300 ppm PPD2 in “5w/w% 5405 in Heptane”
5. 200 ppm PPD2 in “5w/w% 5405 in Heptane”
6. 100 ppm PPD2 in “5w/w% 5405 in Heptane”

The idea is to develop procedure for defining the mechanism of acting of the inhibitor PPD2. To perform this we need to be able to register changes in concentration of inhibitor between initial model oils and probes, which are taken during glass vessel experiments. For creating the calibration curve, above written solutions were prepared and then analyzed by spectrofluorometer. The steady-state spectra are shown in figure 4.11.

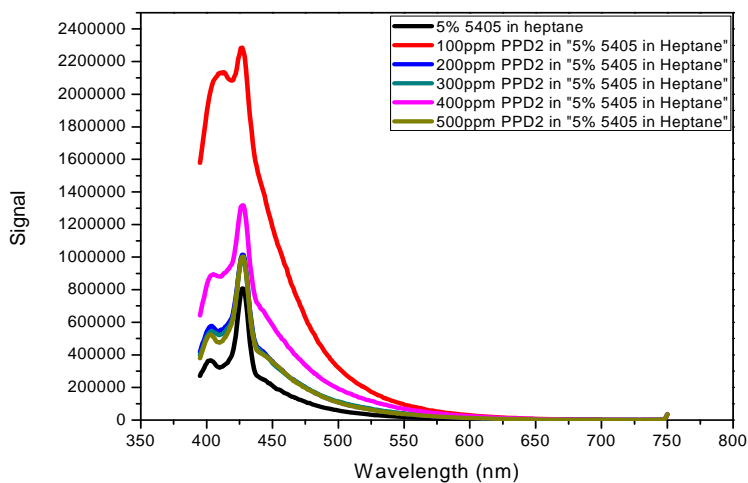


Figure 4.11: Steady-state spectra of 100-500 ppm PPD2 in “5 w/w% 5405 in Heptane”

Intensity signal supposed to be increasing while addition of PPD2 to 5w/w% 5405wax in heptane. For concentrated model oils above 100ppm, the picture is complex; the signal is changing with concentration non-linearly. Quenching of fluorescence could be the reason for concentration range where signal is lowering. A variety of processes can result in quenching, such as excited state reactions, energy transfer, complex-formation. Other possible reason might be molecular aggregation of PPD2.

For the concentration range of 20-100 ppm PPD2 in “5 w/w % 5405 in Heptane”, obtained spectra shows a step wise increase in signal from low concentration to high concentration as shown in figure 4.12. These diluted concentrations don't give rise to aggregation effects among PPD2 molecules.

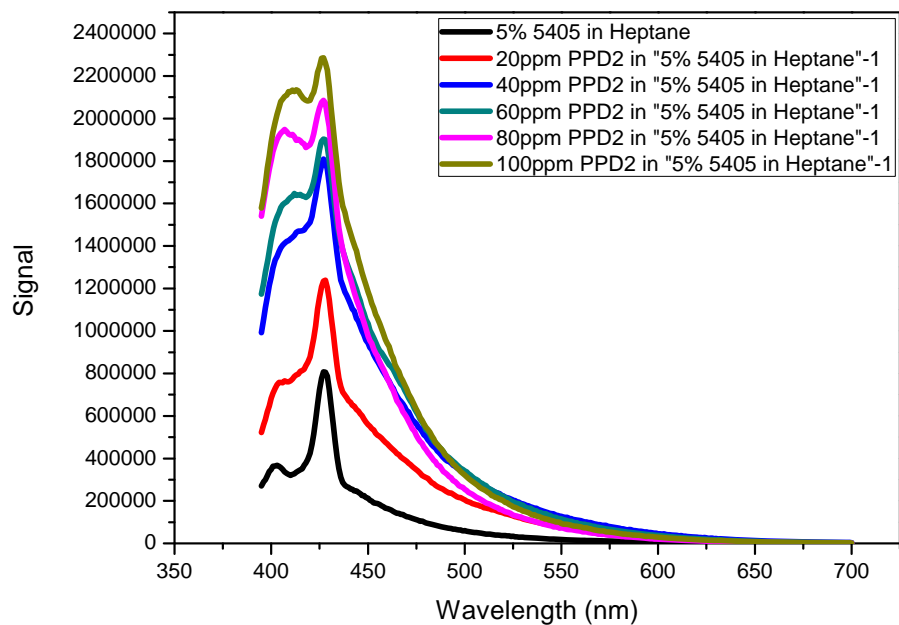


Figure 4.12: Steady-state spectra of 0-100 ppm PPD2 in “5 w/w% 5405 in Heptane”

In order to plot calibration curve for steady state spectra of 0-100 ppm PPD2 in “5 w/w% 5405 in Heptane”, integral fluorescence signal A in the interval between $\lambda_1 = 395\text{nm}$ and $\lambda_2 = 428\text{nm}$ was used instead of the signal on a fixed wavelength. The following equation is used to calculate area under the curves.

$$A = \int_{\lambda_1}^{\lambda_2} I(\lambda) d\lambda$$

Calculated integral signals, which were used in plotting of the calibration curve, are shown in Table 4.1. These integral signals were calculated using Easy Plot Software.

Table 4.1: Integral fluorescence signal for 0-100 ppm PPD2 in “5 w/w% 5405 in Heptane”

Concentration of PPD2 in “5 w/w% 5405 in Heptane” (ppm)	Integral signal (Integral Area) 395-428nm
0	13716900
20	27253600
40	47552800
60	52835600
80	62428300
100	67870200

In figure 4.7, points for calibration curve for the concentration range of 0-100 ppm PPD2 in “5 w/w % 5405 in Heptane” are shown, which gives almost linear trend in this concentration range of PPD2.

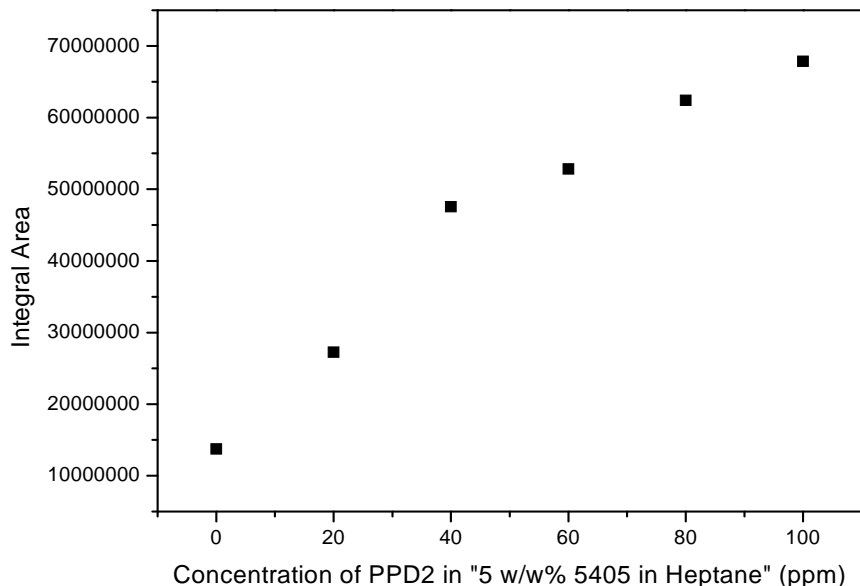


Figure 4.13: Calibration curve for concentration range 0-100 ppm PPD2 in "5w/w% 5405 in Heptane". Integral area of the spectra at wavelength range 395-428nm

At low concentrations from 0-100 ppm of PPD2, inhibitor is well dissolved in waxy solution and stays in solution as single molecules. After concentration of PPD2 reaches 100ppm aggregation process starts and it influence fluorescence spectra of concentrated solutions and non-linear signals are observed in the concentration range of 100-500 ppm PPD2 in waxy solution. Formation of aggregates results in a quenching of fluorescence.

Glass Vessel Experiments on PPD2

Glass vessel experiments were performed on 100 ppm, 40 ppm and 20 ppm PPD2 in "5 w/w % 5405 in Heptane" according to existing protocol and probes were taken at 4⁰C using filter of 0.45 μ m and then tested in spectrofluorometer. The figures 4.14, 4.15 and 4.16 below contain steady state spectra of all these probes with spectra of original inhibited waxy

solutions. By comparing these spectra of probes with spectra of original solutions, an increase in fluorescence signal was found. It means that in probes taken from glass vessel experiments the concentration of inhibitor is higher than in original solution, and hence the signal is increasing.

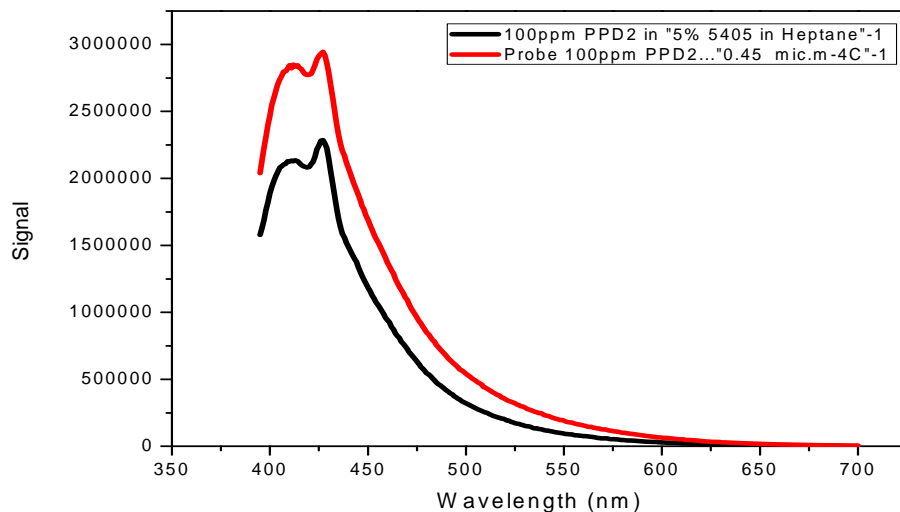


Figure 4.14: Steady-state spectra of 100 ppm PPD2 in “5 w/w% 5405 in Heptane” with its Probe at 0.45 μm filter-4⁰C

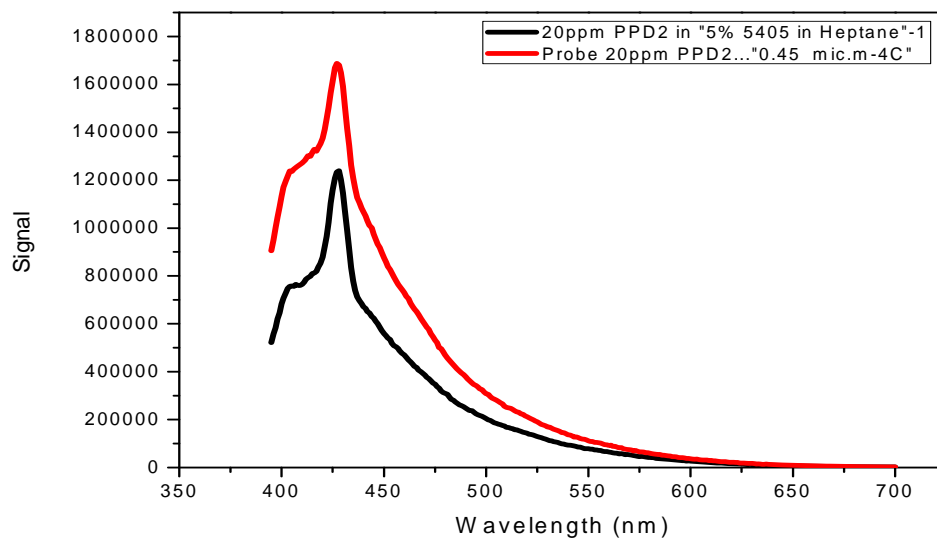


Figure 4.15: Steady-state spectra of 20 ppm PPD2 in “5 w/w% 5405 in Heptane” with its Probe at 0.45 μm filter-4⁰C

For 40ppm PPD2 in “5% 5405 in heptane” when used for glass vessel experiment, two probes were taken:

- probe at 4°C by 0.45 μm filter, and
- probe at 10°C by 0.2 μm filter.

For this experiment, we have taken one more probe named “filter solution”. Wax crystals remaining on both used filter (which was used for probe extracting) were dissolved by a portion of hot heptane, this type of probes were called “filter solution”.

Filter solutions, as well as probes from glass vessel experiments were tested by fluorescence procedure. The resulting spectra are shown in below figure 4.10 along with the steady state spectra of original 40 ppm PPD2 in “5 w/w% 5405 in Heptane”.

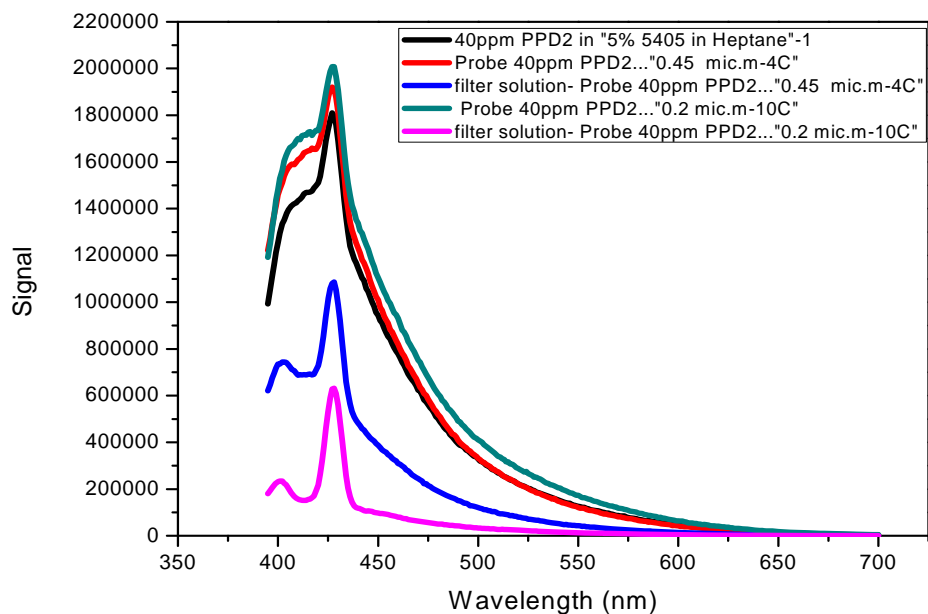


Figure 4.16: Steady-state spectra of 20 ppm PPD2 in “5 w/w% 5405 in Heptane” with its Probe and filter solutions at 4°C and 10°C

Both filter solutions show lower signal than corresponding probes, which means that crystals in the filter has residual amounts of PPD2. Probe at 10⁰C from filter 0.2 μm gives maximum fluorescent signal as compared to Probe at 4⁰C from filter 0.45 μm. The filter solution of filter 0.45 μm at 4⁰C gives a bit higher signal as compared to filter solution of filter 0.2 μm at 10⁰C.

Integral signal could be calculated for probes taken at 4⁰C from 100ppm, 40ppm and 20ppm PPD2 in “5 w/w% 5405 in Heptane” initial solutions. The integral fluorescence signal A in the interval between λ₁ = 395nm and λ₂ = 428nm was used and following equation was used to find out the area under the curves.

$$A = \int_{\lambda_1}^{\lambda_2} I(\lambda) d\lambda$$

By using Easy Plot Software, integral signals were calculated those are listed in Table below:

Table 4.2: Calculated integral signals of Probes at 4⁰C of 100, 40 and 20 ppm PPD2 in “5 w/w% 5405 in Heptane”

Probes at 4⁰C from Concentration of PPD2 in “5 w/w% 5405 in Heptane” (ppm)	Integral signal (Integral Area) 395-428nm
100	89762800
40	53235700
20	42969700

By plotting these points on Figure 4.13; Calibration curve for 0-100 ppm PPD2 in “5 w/w% 5405 in Heptane”, it was found that concentration of PPD2 has increased in probes (in comparison with original solution), which was established from calibration curve as shown in figure 4.17.

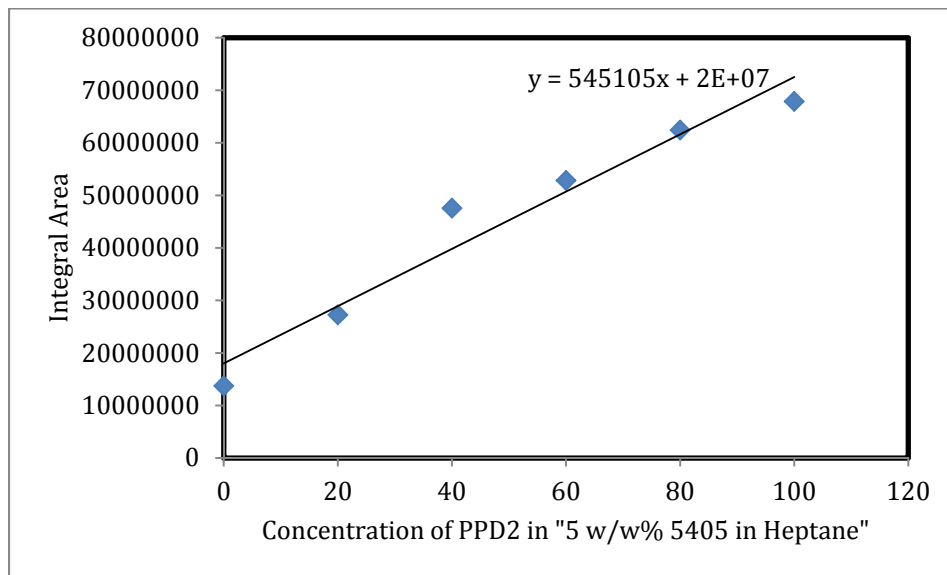


Figure 4.17: Trend line on Calibration curve for 0-100 ppm PPD2 in “5 w/w% 5405 in Heptane”

We have a straight trend line which was best suited and gives us minimum error. Polynomial trend line was also tried and the exponential curve does not fit over the points on the graph.

Table 4.3: Original and calculated concentration of PPD2 in “5 w/w% 5405 in Heptane”

<i>Concentration of PPD2 (ppm)</i>	
In original inhibited waxy solution	In probe taken at 4⁰C from Glass Vessel
100	128
40	61
20	42

We can see from the table that concentration of PPD2 increases in the probe.

4.5.2 PPD3

Following solutions were prepared and analyzed using spectrofluorometer.

- 20 ppm PPD3 in “5 w/w % 5405 Heptane”
- 40 ppm PPD3 in “5 w/w % 5405 Heptane”
- 60 ppm PPD3 in “5 w/w % 5405 Heptane”
- 80 ppm PPD3 in “5 w/w % 5405 Heptane”
- 100 ppm PPD3 in “5 w/w % 5405 Heptane”

When we plot steady-state spectra of solutions 0 – 100 ppm PPD3 in “5 w/w% 5405 in Heptane”, it goes linear from 0 to 40 ppm but after that from 60 to 100 it doesn't follow the same pattern and gives a low fluorescence signal, this is shown in figure 4.18. It might be due to aggregation effects at high enough concentrations.

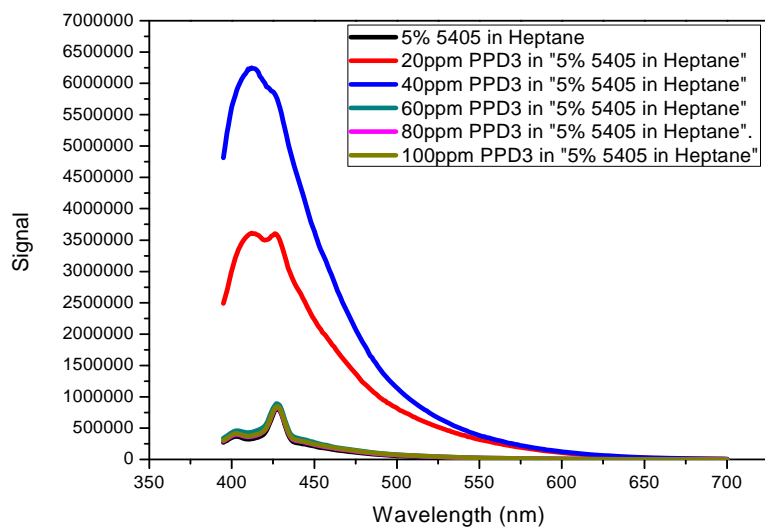


Figure 4.18: Steady-state spectra of 0-100 ppm PPD3 in “5 w/w% 5405 in Heptane”

Now as we are working in the range of 0-40 ppm PPD3 in “5 w/w% 5405 in Heptane”, two more solutions in this working range were prepared:

- 10 ppm PPD3 in “5 w/w % 5405 Heptane”
- 30 ppm PPD3 in “5 w/w % 5405 Heptane”

Steady state spectra give us increasing curve even with 10 ppm PPD3 and 30 ppm PPD3 spectra, shown in figure 4.19. This means that we can make calibration curve in this range.

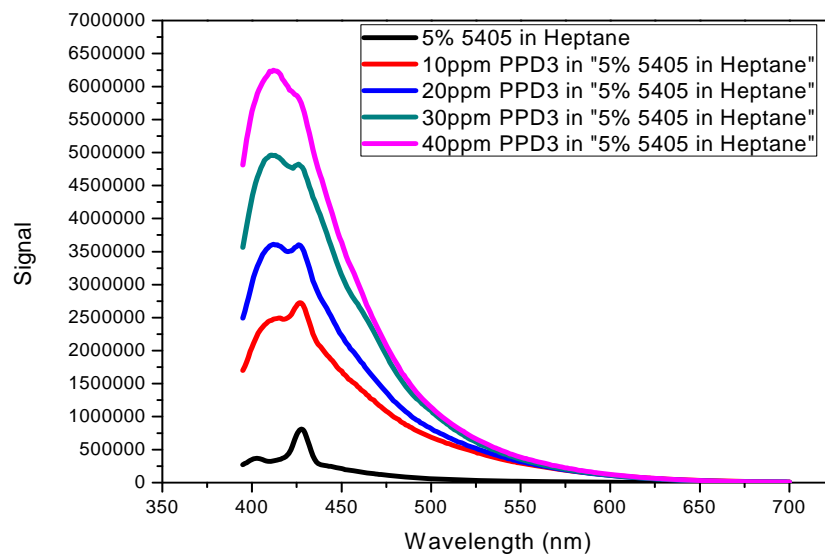


Figure 4.19: Steady-state spectra of 0-40 ppm PPD3 in “5 w/w% 5405 in Heptane”

In order to plot calibration curve for steady state spectra of 0-40 ppm PPD3 in “5 w/w% 5405 in Heptane”, integral fluorescence signal A in the interval between $\lambda_1 = 395\text{nm}$ and $\lambda_2 = 428\text{nm}$ was used instead of the signal on a fixed wavelength. Integral signals are calculated by using Easy Plot software and were used in plotting of the calibration curve, are shown in the following table.

Table 4.4: Integral fluorescence signal for 0-40 ppm PPD3 in "5 w/w% 5405 in Heptane"

<i>Concentration of PPD3 in "5 w/w% 5405 in Heptane" (ppm)</i>	<i>Integral Signal (Integral Signal) 395-428 nm</i>
0	13176900
10	78158200
20	111554000
30	154397000
40	194764000

In the following figure 4.20, points for calibration curve for the concentration range of 0-40 ppm PPD3 in "5 w/w% 5405 in Heptane" are plotted, which shows almost linear trend.

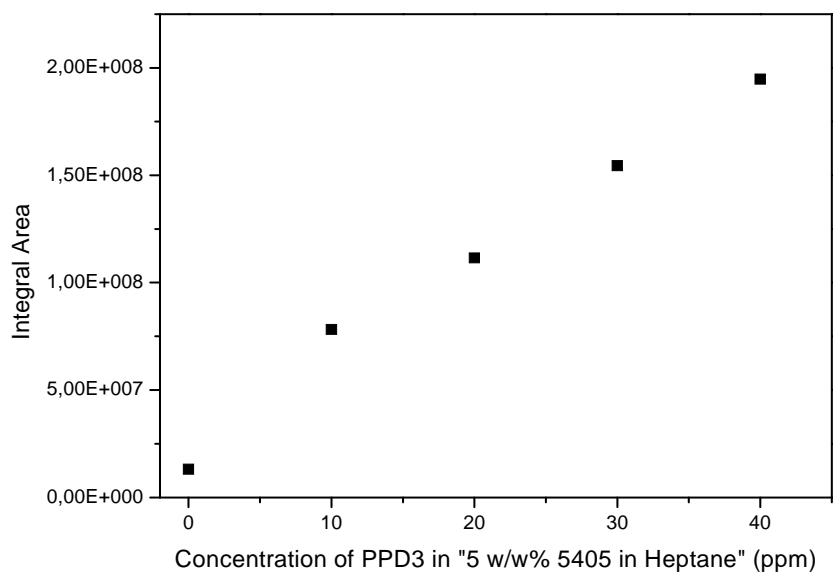


Figure 4.20: Calibration curve for concentration range 0-40 ppm PPD3 in "5 w/w% 5405 in Heptane". Integral area of the spectra at wavelength range 395-428nm

Glass Vessel Experiments on PPD3

Then we performed glass vessel experiments on *20 ppm and 10ppm PPD3 in “5 w/w% 5405 in Heptane”* and take probe at 4°C with $0.2\ \mu\text{m}$ filter. Also its filter solution has been collected. Then, repeated this experiment. Here are the results:

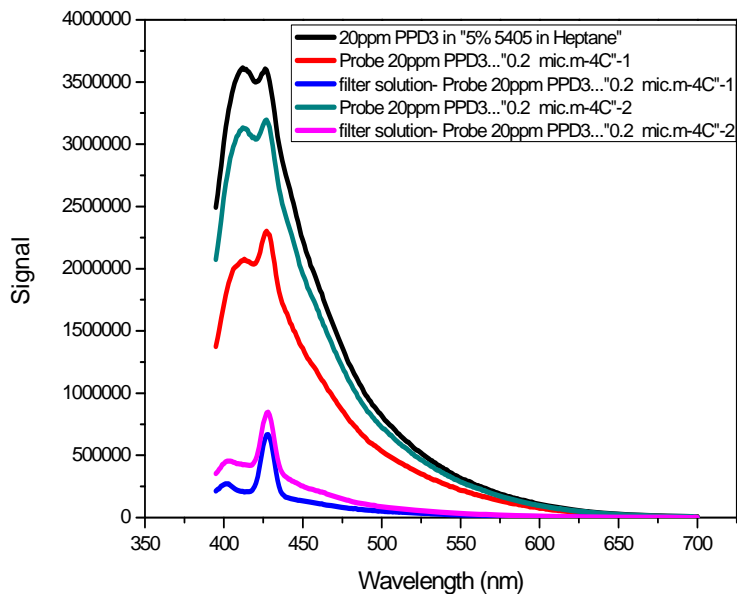


Figure 4.21: Steady-state spectra of 20 ppm PPD3 in “5 w/w% 5405 in Heptane” with its Probe and filter solution at 4°C with $0.2\ \mu\text{m}$ filter

It is clear from this figure 4.21 that steady-state spectra of probes goes down in comparison of spectra of 20ppm PPD3 in “5 w/w% 5405 in Heptane”, it means probes give a decreased fluorescence signal. It might be due to some different structure formation of gel took place during glass vessel experiment. Steady-state spectra of both filter solutions lie close to each other at the bottom of the figure with a lower fluorescence signal.

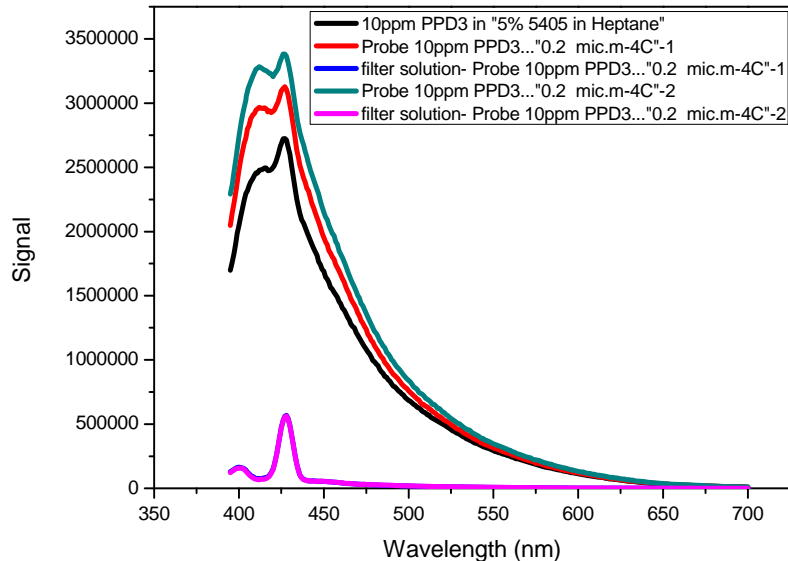


Figure 4.22: Steady-state spectra of 10 ppm PPD3 in “5 w/w% 5405 in Heptane” with its Probe and filter solution at 4°C with 0.2 μm filter

In the above figure 4.22, by comparing steady-state spectra of probes with that of original solution of 10 ppm PPD3 in “5 w/w% 5405 in Heptane”, an increase in fluorescence signal appeared. It might be possible that concentration of inhibitor is higher in probes taken from glass vessel experiments than in original solution, which gives us an increased signal.

Steady state spectra of filter solutions give a lower fluorescence signal as compared to original solution.

By adding trend line to the calibration curve earlier plotted for 0-40 ppm PPD3 in “5 w/w% 5405 in Heptane”, it might be able to explain the increase in fluorescence signal of 10 ppm PPD3.

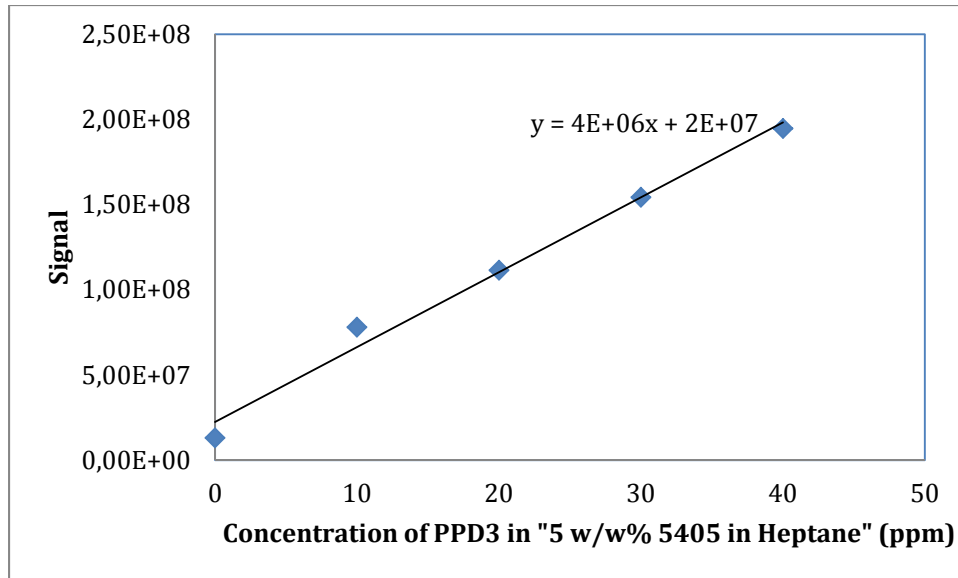


Figure 4.23: Trend line on Calibration curve for 0-40 ppm PPD3 in “5 w/w% 5405 in Heptane”

Table 4.5: Original and calculated concentration of PPD3 in “5 w/w% 5405 in Heptane”

<i>Concentration of PPD3 (ppm)</i>	
In original inhibited waxy solution	In probe taken at 4⁰C from Glass vessel
10	14.54
20	15.37
30	33.59
40	43.69

We can see from the table that concentration of PPD3 increases in the probe. But for 20ppm PPD3 in “5 w/w % 5405 in Heptane”, the concentration of PPD3 actually decreases in probe.

4.5.3 PPD4

Following solutions were prepared and analyzed in spectrofluorometer.

- 20 ppm PPD4 in “5 w/w % 5405 Heptane”
- 40 ppm PPD4 in “5 w/w % 5405 Heptane”
- 60 ppm PPD4 in “5 w/w % 5405 Heptane”
- 80 ppm PPD4 in “5 w/w % 5405 Heptane”
- 100 ppm PPD4 in “5 w/w % 5405 Heptane”

Figure 4.24 shows steady state spectra of solutions 20 – 100 ppm PPD4 in “5 w/w% 5405 in Heptane”, the steady state spectra of 20, 40 and 100 ppm lies over each other while 60 ppm and 80 ppm goes up in blue and green color respectively. This shows that it is not possible to have calibration curve in this concentration range of PPD4.

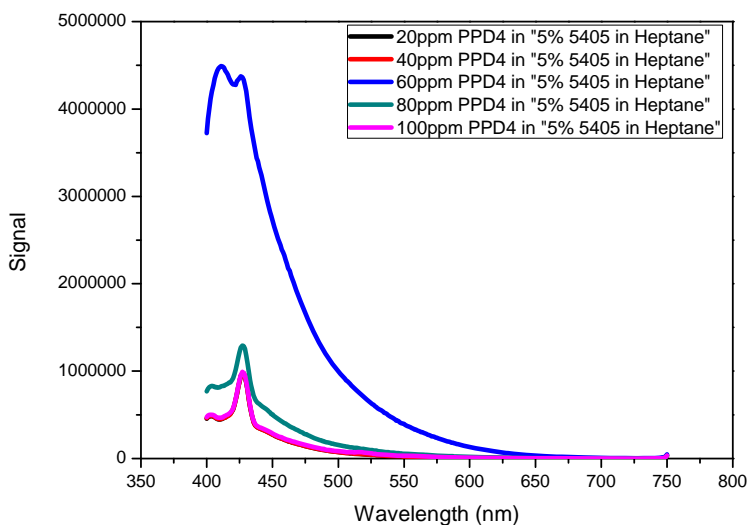


Figure 4.24: Steady-state spectra of 20-100 ppm PPD4 in “5 w/w% 5405 in Heptane”

A glass vessel experiment is performed on 20 ppm PPD4 in “5w/w% 5405 in Heptane”. In figure 4.25, the steady state spectra of probe and the solution almost lies over each other.

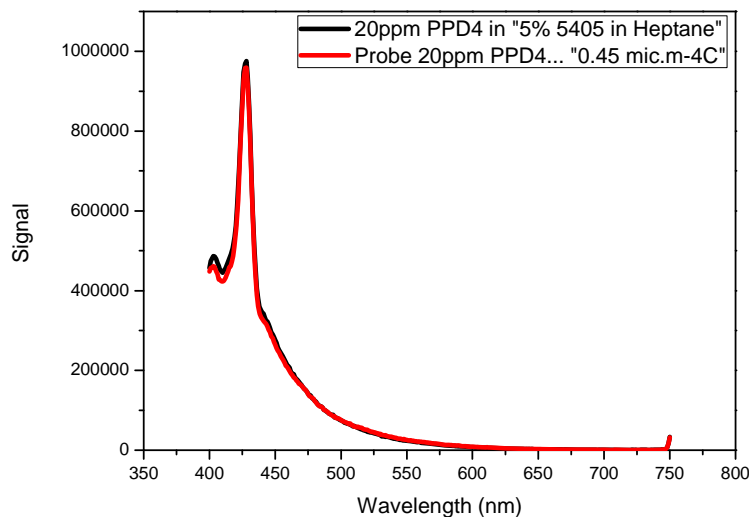


Figure 4.25: Steady-state spectra of 20 ppm PPD4 in “5 w/w% 5405 in Heptane” with its Probe at 4°C with 0.2 µm filter

4.5.4 PPD5

Following solutions were prepared and analyzed in spectrofluorometer.

- 20 ppm PPD5 in “5w/w% 5405 Heptane”
- 40 ppm PPD5 in “5w/w% 5405 Heptane”
- 60 ppm PPD5 in “5w/w% 5405 Heptane”
- 80 ppm PPD5 in “5w/w% 5405 Heptane”
- 100 ppm PPD5 in “5w/w% 5405 Heptane”
- 120 ppm PPD5 in “5w/w% 5405 Heptane”

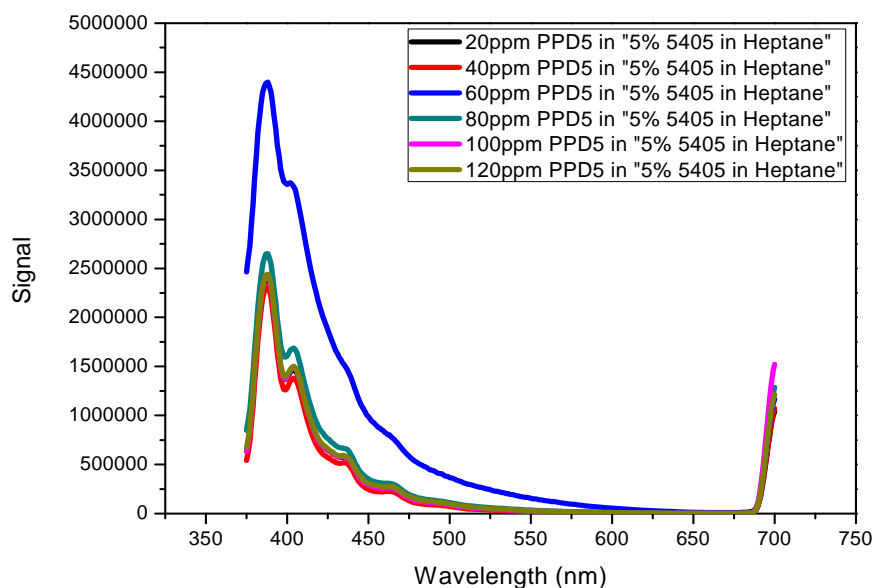


Figure 4.26: Steady-state spectra of 20-120 ppm PPD5 in “5 w/w% 5405 in Heptane”

Obtained spectra show complex behavior of fluorescent species in concentration range 20-120 ppm PPD5. Possibly aggregation is already taken place at this low concentrations, which influence non-linearity of fluorescence signal changes.

Glass vessel experiment was performed on 20 ppm PPD5 in “5 w/w% 5405 in Heptane” and probe was taken at 4⁰C using 0.45 μ m filter. The steady-state spectra are shown in figure 4.27 below.

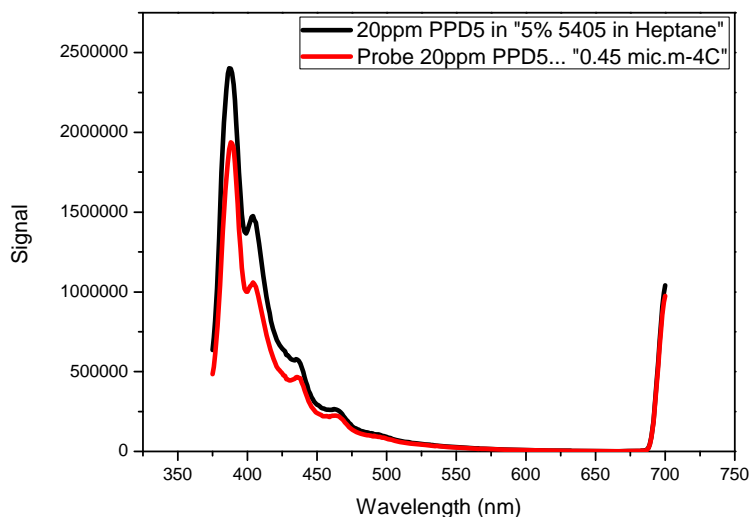


Figure 4.27: Steady state spectra of 20 ppm PPD5 in “5 w/w% 5405 in Heptane” with its Probe at 4°C with 0.45 μm filter

Decrease of the signal of the probe in comparison to original solution doesn't allow us to state that concentration of PPD5 in the probe has decreased. It might be due to impossibility of plotting adequate calibration curve in this concentration range. Previous work on PPD2 shows that in order to work in this concentration range with PPD5, it should be understood how to make a calibration curve.

Higher concentrations of PPD5 in “5 w/w% 5405 in Heptane” were tested to see whether there is linearity in behavior of fluorescence signal (100 – 500 ppm, 700 ppm and 1000 ppm PPD5 in “5% w/w 5405 in Heptane”). Steady state spectra are shown in figure 4.28.

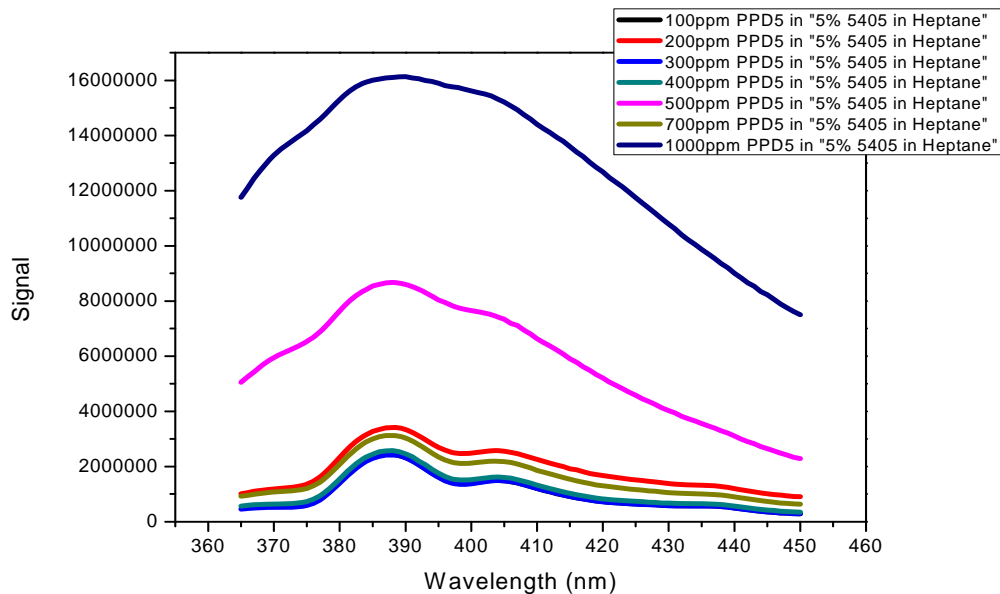


Figure 4.28: Steady state spectra of 100-500,700 & 1000 ppm PPD5 in “5 w/w% 5405 in Heptane”

Signal change from concentration range 100-1000ppm PPD5 appeared to be nonlinear. So, it is not possible to have calibration curve for PPD5 by presented technique.

4.6 NMR experiments on Model wax oil systems

(1) 5w/w% 5405 in Deuterated Toluene, with and without PPD2

Two samples of Sasolwax 5405 immersed in deuterated toluene were prepared;

1. 5 w/w % 5405 in Deuterated Toluene
2. 100 ppm PPD2 in 5 w/w % 5405 in Deuterated Toluene

These were analyzed by LF-NMR at elevated temperatures, from 30 to 4⁰C. Cooling rate was manually controlled.

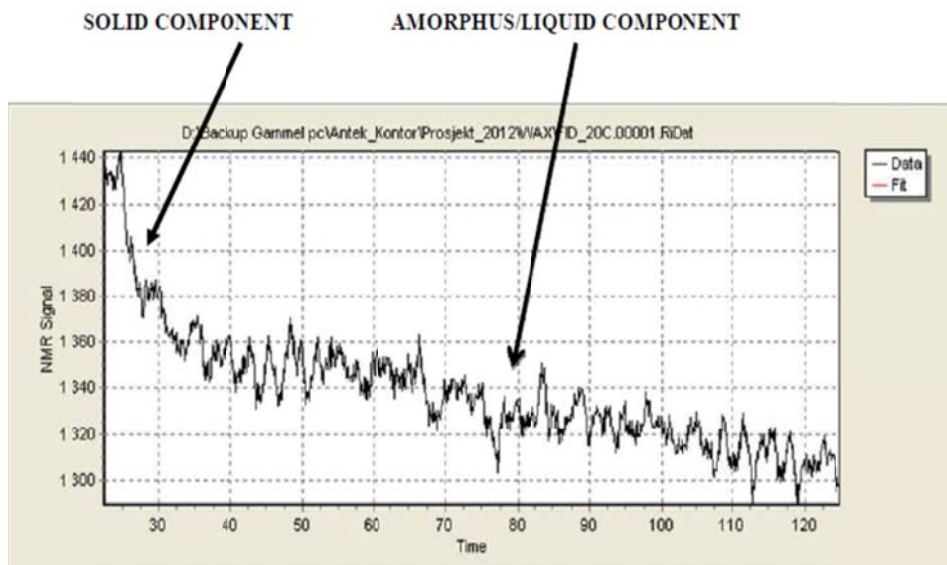


Figure 4.30: The NMR signal arising from FID below transition temperature. The sharp initial decay is due to crystallized phase of wax, i.e., the solid component

At 30⁰C the sample was pure liquid and there was no sign of any particles forming in the liquid. The T_2 distribution in figure 4.31 also shows a sharp peak at 1.1 seconds, which supports the picture of a homogeneous liquid. As the temperature was lowered down to approximately 21⁰C by steps of 2.5 degrees, there was only a small shift in the distributions towards shorter T_2 -values. However, the peaks were still quite sharp and visual inspection did not show any loss in transparency of the liquid.

From 21⁰C and downwards the increments was 1 degree, and the results are displayed in figure 4.31 and 4.32. When the temperature was below 19⁰C, the pure sample became less transparent, and shift in the T_2 distribution towards shorter T_2 's started to appear. However, the shift was not as strong as in the previous test, where the cooling rate was much higher.

The shift in T_2 distributions towards shorter T_2 -values, and the loss of NMR signal is due to the loss of signal from the crystallized wax, which will have a T_2 of less than 100 μs . Then it is not detectable by the CPMG experiment which records the T_2 . For the sample containing 100 ppm PPD2, the loss of signal became apparent at a temperature of 16-17 $^{\circ}\text{C}$, i.e. ~ 2 degrees lower. What is also interesting to note the less broadening of the T_2 -distribution and less shift towards shorter T_2 values for the sample containing 100 ppm PPD2. This means that the liquid component keeps its “liquidity” at lower temperatures compared to the sample of pure 5405.

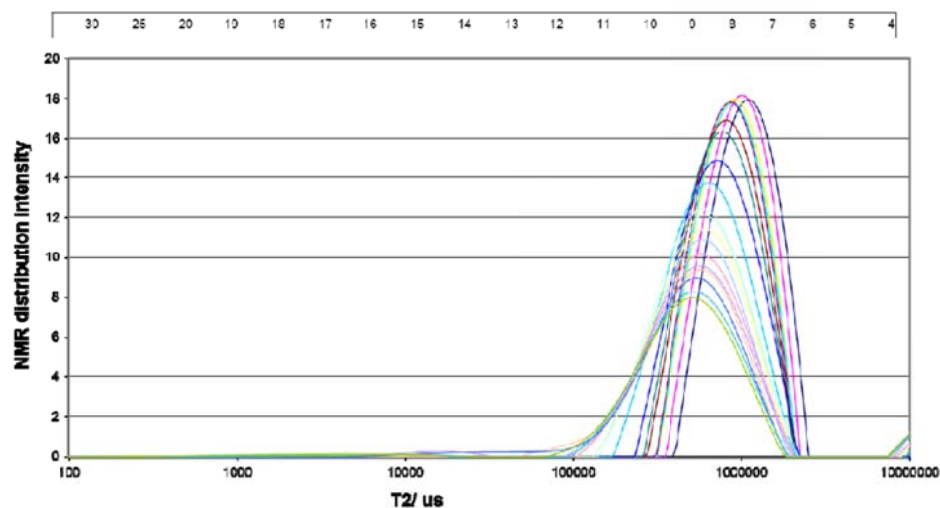


Figure 4.31: T_2 distribution as a function of temperature for 5 w/w % 5405 in Deuterated Toluene

The solid signal may be found in the FID experiment, where the first measuring point is at 30 μs . Then we are able to monitor the tail of the solid component, as shown in figure 4.30. In figure 4.33 the solid fraction is plotted against the monitoring temperature for the two samples. (In order to do so, the NMR signal, which is temperature dependent, must be calibrated against a known sample which stays as a liquid in the temperature range 4 - 30 $^{\circ}\text{C}$. Water was used for this purpose.)

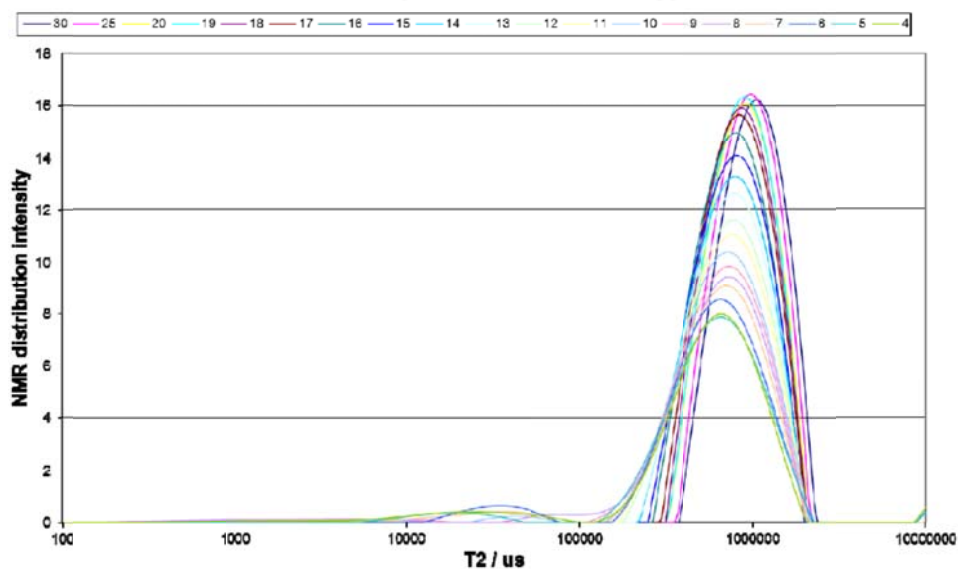


Figure 4.32: T_2 distribution as a function of temperature for 100 ppm PPD2 in 5 w/w % 5405 in Deuterated Toluene

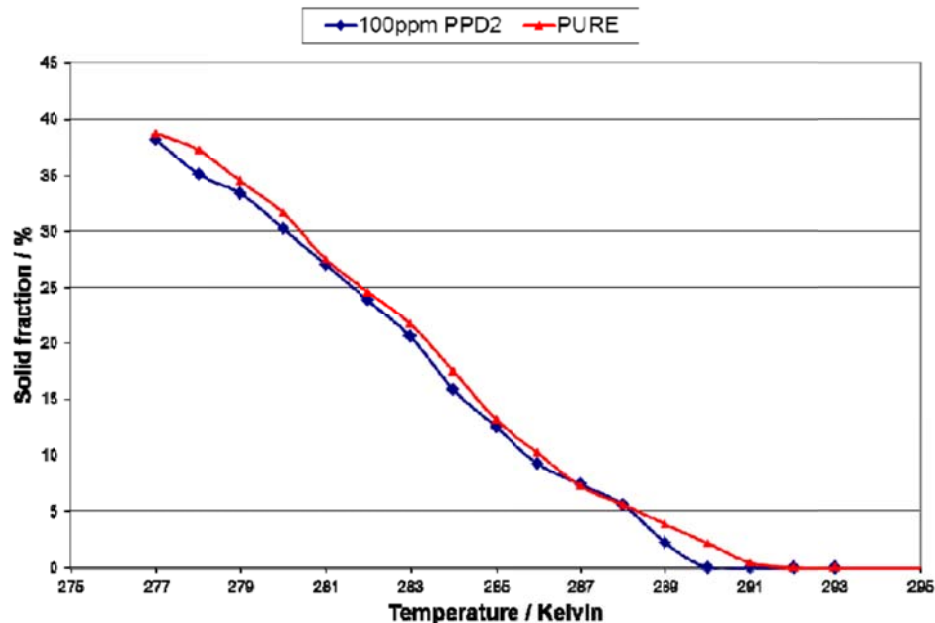


Figure 4.33: The fraction of solid component as a function of Temperature

As seen in figure 4.33 the solid component starts to appear at a slightly lower temperature for the sample containing 100 ppm PPD2, and the solid fraction continues to stay below the value of the pure sample down to 4°C. The difference is not large, but it is significant enough to conclude that the introduction of 100 ppm PPD2 reduces the fraction of solid component at the same temperature. In the liquid phase the average T_2 is longer for the sample containing 100 ppm PPD2, which supports the picture of a sample that is more mobile at lower temperature once 100 ppm PPD2 is added.

(2) 10 w/w% 5405 in Deuterated Toluene

A sample containing 10% wax and 90% deuterated toluene was placed in the low field NMR (LF-NMR) instrument at a temperature of 50⁰C.

At 50⁰C the sample was pure liquid and there was no sign of any articles forming in the liquid. The T₂ distribution in figure 4.34 also shows a sharp peak at 1.2 seconds, which supports the picture of a homogeneous liquid. As the temperature was lowered down to approximately 25⁰C there was only a small shift in the distributions towards shorter T₂ -values. However, the peaks were still quite sharp and visual inspection did not show any loss in transparency of the liquid. When the temperature approached 20⁰C, the liquid became less transparent, and a sudden shift in the T₂ distribution towards much shorter T₂'s, from 900 ms down to 300 ms.

In addition the shape of the T₂ distribution changed as it became broader and even lost some signal. This is expected when a solidification process is taking place. Less homogeneous system leads to broader T₂ distributions at shorter T₂-values, and the loss of NMR signal is due to the loss of signal from the crystallized wax, which will have a T₂ of less than 100 us, which is not detectable in the CPMG experiment. However, the solid signal may be found in a so called FID experiment, where the first measuring point is at 30 us, and then we are able to monitor the tail of the solid component, as shown in previous figure 4.30.

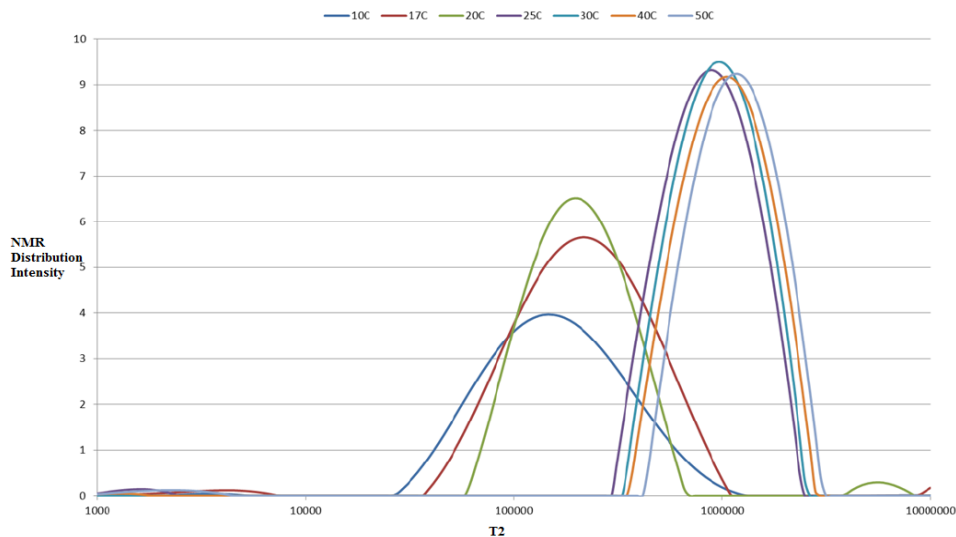


Figure 4.34: T_2 distribution as a function of temperature for 10 w/w % 5405 in Deuterated Toluene

As the temperature is further decreased, the T_2 distribution is further broadened and even more signal is lost. If there was no solidification, one should expect the NMR signal to increase as a function of lowering the temperature. Thus the system continues to solidify as the temperature is lowered, and at 10⁰C probably more than 50 % of the wax is in a solid phase. No exact quantitative measurements were done at this stage.

Finally the sample was tempered to 20⁰C again, and a NMR diffusion experiment was performed. This experiment does not measure the solid signal, only the amorphous phase. At 50⁰C we would expect the diffusion coefficient of the liquid to more or less coincide with the brine, but at 20⁰C we do see a significant difference between the brine and the wax diffusion.

This is due to the presence of the solid phase which prevents the “liquid” wax to move freely around. Thus one would expect a similar temperature dependency on the diffusion as for the T₂ distributions.

It might be feasible to study this transition indirectly by monitoring the diffusion coefficient of the non-deuterated solvent.

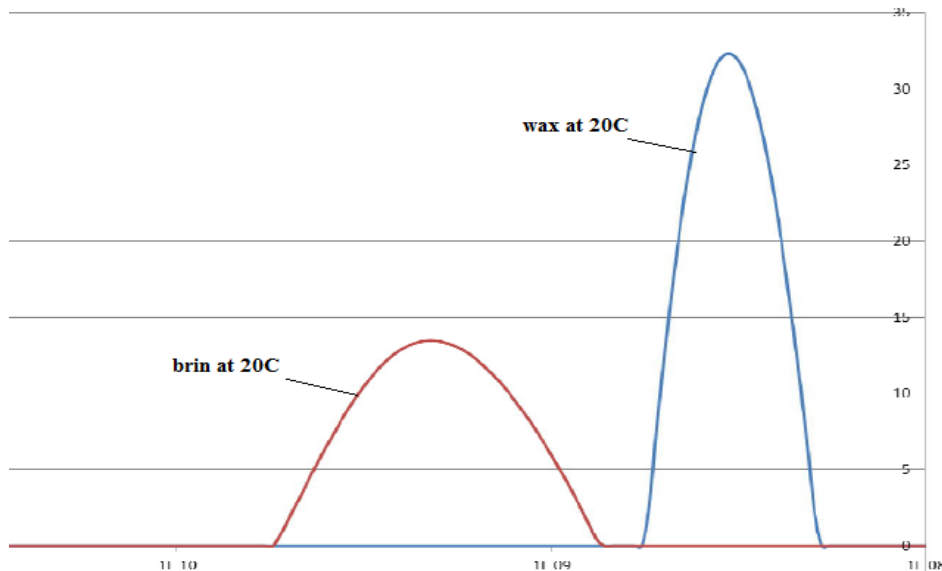


Figure 4.35: Diffusion distribution from wax at 20⁰C and compared against brine at 20⁰C

4.7 GC experiments on Model wax oil systems

Following samples were prepared and sent to Champion Technologies, Netherlands for GC analysis:

1. 5 w/w % 5405 in Heptane
2. 40 ppm PPD2 in “5 w/w% 5405 in Heptane”
3. Probe at 4⁰C with 0.45 μ m filter from - 40 ppm PPD2 in “5 w/w% 5405 in Heptane”
4. Filter solution of - Probe at 4⁰C with 0.45 μ m filter from 40 ppm PPD2 in “5 w/w% 5405 in Heptane”

5. Probe at 10⁰C with 0.2 μm filter from - 40 ppm PPD2 in “5 w/w% 5405 in Heptane”
6. Filter solution of - Probe at 10⁰C with 0.2 μm filter from 40 ppm PPD2 in “5 w/w% 5405 in Heptane”

Here are the results of GC analysis for these samples. All the samples were measured three times and these results contain the average values from their respective experimental runs. Details of the data are further presented in Appendix A4.

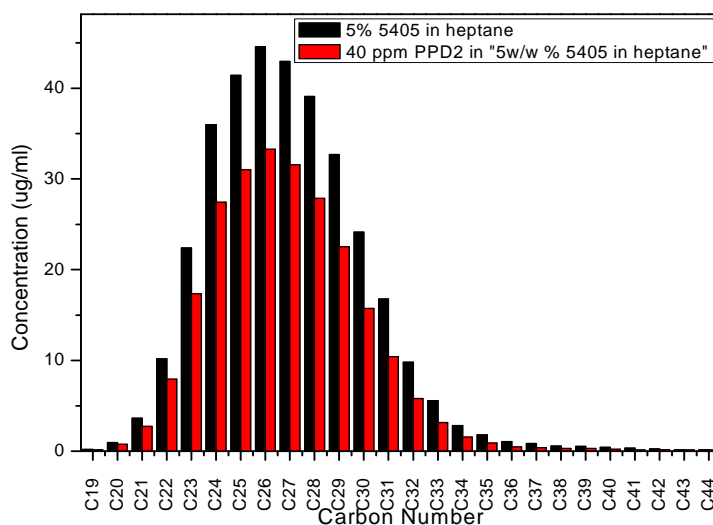


Figure 4.36: GC analysis of 5w/w% 5405 in Heptane and 40 ppm PPD2 in 5w/w% 5405 in Heptane

The concentration of each carbon number reduces when we add PPD2 in model wax oil system i.e., 5w/w% 5405 in Heptane. This could happen due to that GC analysis is carrying at 400⁰C. This temperature is closed to temperature at which alkanes could be cracked. PPD can act at this temperature in that way that it supports alkane cracking.

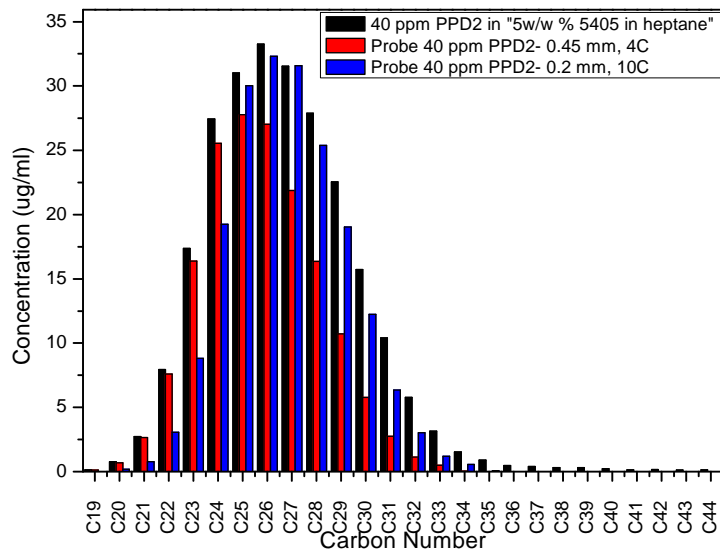


Figure 4.37: GC analysis of 40 ppm PPD2 with probes at 10°C with 0.2 μm filter and at 4°C with 0.45 μm filter

The carbon number distribution goes down when we take probe at 10°C from 40 ppm PPD2 in 5w/w% 5405 in Heptane. Carbon number distribution goes further down for the probe at 4°C. According to figure 4.36, one can see shift to low molecular alkanes when analyzing initial solution “40 ppm PPD2 in 5w/w% 5405 in Heptane” and probe, which were taken from it at 4°C. Analysis shows that high molecular alkanes crystallize first upon cooling of waxy oil systems while low molecular alkanes are still present in inter-crystal space in liquid phase.

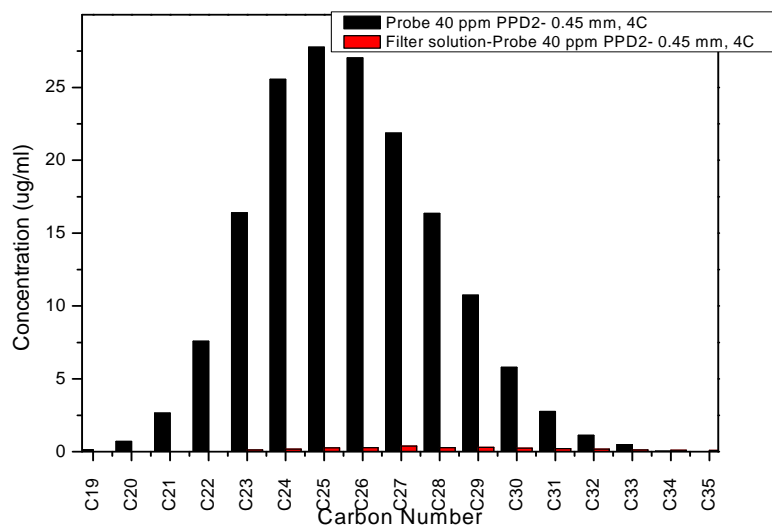


Figure 4.38: GC analysis of probe and filter solution at 4C with 0.45 μ m filter for 40 ppm PPD2 in 5w/w% 5405 in Heptane

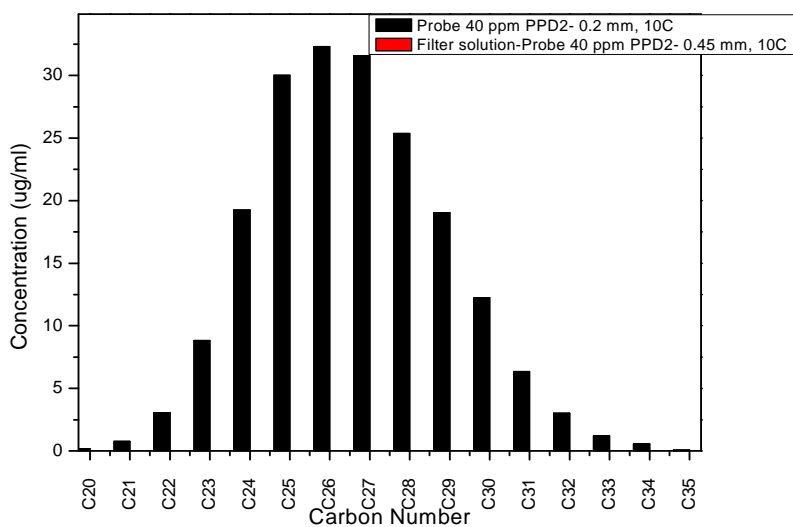


Figure 4.39: GC analysis of probe and filter solution at 10C with 0.2 μ m filter for 40 ppm PPD2 in 5w/w% 5405 in Heptane

The filter solution doesn't give any significant carbon number distribution as compared to their respective probes.

5 CONCLUSIONS

This research work aims to present the impact of cloud point depressants (pour point depressants) on wax-oil gel structure and its characteristics.

Glass vessel protocol for analyzing inhibited model wax oil systems was developed specifically for two inhibitors named PPD2 and PPD3 in macro crystalline wax using heptane as solvent.

Protocol for spectrofluorometer was also developed for analyzing model oil systems with inhibitors (PPD2, PPD3, PPD4 and PPD5) containing fluorescence species. This protocol works well for two inhibitors PPD2 and PPD3 in model wax oil systems. Results clearly indicate the increase in concentration of PPD2 and PPD3 in probes taken from glass vessel experiment as compared to the concentration in original inhibited model wax oil systems.

Low field NMR can be used to monitor the formation of solid/amorphous phase in a liquid containing wax, and it may distinguish between systems and their Cloud Point. A deuterated solvent is the best option, but it might be possible to monitor most of the changes using a non-deuterated solvent as well.

According to GC analysis, at subsea temperature, low-molecular alkanes are present in liquid phase, while high-molecular alkanes are participating in forming wax crystals.

PPDs action mechanism

Following are the effects which PPD2 and PPD3 have on Model wax oil system:

- Decreasing cloud point to a small extent (about 2 degrees)
- Glass vessel probes gives increased fluorescence signal as compared to their original solution

Small decrease in cloud point suggests that PPDs are not changing wax crystal structure in model oils of presented composition; molecules of inhibitor are trapped between forming paraffin crystals.

Probes give increased signal because filters were used to take probe, so large crystals (high molecular weight) were blocked by filter. Probes contain dissolved paraffins of lower values (as explained by GC analysis) along with PPDs. So PPD molecules have more freedom to move and they give high fluorescence signals. In case of original waxy solution with PPDs, fluorescence signal is lower as compared to probes; the reason might be that PPD molecules are not free to move, they are attached/adsorbed on paraffins which changes their fluorescence ability.

6 FUTURE RECOMMENDATIONS

A more detailed research on the mechanism of cloud point depressants should be carried out. For research in future, experiments can be performed by varying model oil composition in order to get closer to real crude oil composition, and hence the performance of PPDs could be seen brightly. Investigations of concentration dependence of PPDs content in Model wax oil systems on carbon number distribution of alkanes are suggested by GC analysis.

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A3 List of Symbols

Symbols	Description
A	Integral Area
D	Diffusion coefficient
E	Energy level of hydrogen's nuclear spin
ΔE	Difference between two energy levels
g	Magnetic field gradients
$g(t'')$	Total magnetic field gradient, external and internal
h	Planck's constant
H_0	Homogenous magnetic field
H_1	Rotating magnetic field
$H(t)$	External magnetic field
I	Spin operator
I_0	Initial intensity of the NMR-signal
k	Boltzmann's constant
M^+	Transverse magnetization
M_0^+	Integration constant on initial conditions
n_{upper}	Population at upper level
n_{lower}	Population at lower level
t_1	Duration the NMR-signal is influenced by transverse relaxation processes
t_2	duration the NMR-signal is influenced by longitudinal relaxation processes
T	Absolute temperature
T_1	Characteristic longitudinal relaxation time
T_2	Characteristic transverse relaxation time
v	velocity
μ	Nuclear magnetic moment
ω	frequency
γ	Gyromagnetic ratio

A4 GC analysis data

Wax Concentration	5%5405 heptane	40ppm PPD2 in...	Probe at 4C	filter soln at 4C	Probe at 10C	filter soln at 10C
C18						
C19	0,203315682	0,155712868	0,133806458			
C20	0,965357808	0,769873133	0,697729013		0,191417512	
C21	3,630012656	2,724236145	2,647850849		0,779554198	
C22	10,16843091	7,93893398	7,581779977		3,068099247	
C23	22,40461254	17,36913792	16,38910161	0,12597047	8,815712286	
C24	35,99933688	27,44786955	25,54988014	0,183355909	19,2509774	
C25	41,43357436	31,01461257	27,77265773	0,264701434	30,02040037	
C26	44,59247124	33,27507783	27,02141922	0,289308817	32,31825748	
C27	42,93470886	31,55219203	21,8665751	0,377847581	31,57621554	
C28	39,08373986	27,88208734	16,3605634	0,283418147	25,37607699	
C29	32,69055282	22,54640518	10,73306771	0,291509242	19,03256805	
C30	24,14997743	15,7355571	5,789709498	0,245385545	12,23751016	
C31	16,77848486	10,41589007	2,759606795	0,20724207	6,358161956	
C32	9,800260103	5,792725855	1,12539097	0,174032408	3,030559743	
C33	5,592666661	3,147949032	0,494376087	0,114157825	1,200111396	
C34	2,813642663	1,536314145	0,042185413	0,112855784	0,577407026	
C35	1,799880352	0,9052503		0,088053509	0,083641537	
C36	1,062938579	0,478095242				
C37	0,83792725	0,400204392				
C38	0,553595468	0,304952188				
C39	0,523533178	0,317723501				
C40	0,423244538	0,225309286				
C41	0,327548219	0,154194952				
C42	0,24028516	0,17222798				
C43	0,180276077	0,133708695				
C44	0,159817098	0,154024438				
C45						

Wax (% of C18+ Paraffins)	5%5405 heptane	40ppm PPD2 in...	Probe at 4C	filter soln at 4C	Probe at 10C	filter soln at 10C
C18						
C19	0,060411744	0,064240803	0,080327985		0,098671785	
C20	0,284931121	0,317456063	0,418321697		0,402047327	
C21	1,068715815	1,123105183	1,585794229		1,582299317	
C22	2,992597967	3,273183667	4,541014624	4,556441656	4,546343444	
C23	6,598290408	7,161096788	9,816519193	6,581041616	9,928325657	
C24	10,60128956	11,31636414	15,30252515	9,749634854	15,48372873	
C25	12,20234676	12,7867491	16,63219054	10,61665257	16,6683213	
C26	13,13050091	13,71896601	16,18356984	13,70110079	16,28495058	
C27	12,64775415	13,00836372	13,09703439	10,29754348	13,08645077	
C28	11,51974829	11,49521297	9,798151142	10,4919158	9,813775946	
C29	9,639528171	9,295481384	6,428408884	8,861668265	6,308871739	
C30	7,121633978	6,487689192	3,468145422	7,513208462	3,276661191	
C31	4,949991285	4,294308871	1,653538329	6,319888806	1,561155215	
C32	2,893628432	2,388330818	0,673898264	4,075573559	0,618097629	
C33	1,653463248	1,297882772	0,295440736	4,00329369	0,297426752	
C34	0,831022135	0,633376223	0,025119573	3,232036452	0,042872608	
C35	0,530746035	0,373120557				
C36	0,314135735	0,197080008				
C37	0,247562259	0,165005727				
C38	0,163902538	0,125772745				
C39	0,156154376	0,130984796				
C40	0,123411931	0,092949047				
C41	0,096631784	0,063570067				
C42	0,070612948	0,07107386				
C43	0,05371213	0,055103946				
C44	0,047276289	0,063531552				
C45						

NTNU	Risikovurdering	utarbeidet av	Nummer	Dato	
		HMS-avd.	HMSRV/2603	4.3.2010	
HMS/KS		godkjent av	side	Erstatter	
		Rektor	2 av 3	9.2.2010	

Sannsynlighet

1. Svært liten
2. Liten
3. Middels
4. Stor
5. Svært stor

Konsekvens

- A. Svært liten
- B. Liten
- C. Moderat
- D. Alvorlig
- E. Svært alvorlig

Risikoverdi (beregnes hver for seg):

Menneske = Sannsynlighet x Konsekvens Menneske

Ytre miljø = Sannsynlighet x Konsekvens Ytre miljø

Økonomi/materiell = Sannsynlighet x Konsekvens Øk/materiell



Omdømme = Sannsynlighet x Konsekvens Omdømme

Sannsynlighet vurderes etter følgende kriterier:

Svært liten 1	Liten 2	Middels 3	Stor 4	Svært stor 5
1 gang pr 50 år eller sjeldnere	1 gang pr 10 år eller sjeldnere	1 gang pr år eller sjeldnere	1 gang pr måned eller oftere	Skjer ukentlig

Konsekvens vurderes etter følgende kriterier:

Gradering	Menneske	Ytre miljø Vann, jord og luft	Øk/materiell	Omdømme
E Svært Alvorlig	Død	Svært langvarig og ikke reversibel skade	Drifts- eller aktivitetsstans >1 år.	Troverdighet og respekt betydelig og varig svekket
D Alvorlig	Alvorlig personskade. Mulig uførhet.	Langvarig skade. Lang restitusjonstid	Driftsstans > ½ år Aktivitetsstans i opp til 1 år	Troverdighet og respekt betydelig svekket
C Moderat	Alvorlig personskade.	Mindre skade og lang restitusjonstid	Drifts- eller aktivitetsstans < 1 mnd	Troverdighet og respekt svekket
B Liten	Skade som krever medisinsk behandling	Mindre skade og kort restitusjonstid	Drifts- eller aktivitetsstans < 1 uke	Negativ påvirkning på troverdighet og respekt
A	Skade som krever førstehjelp	Ubetydelig skade og kort	Drifts- eller aktivitetsstans <	Liten påvirkning på troverdighet

NTNU	Risikovurdering	utarbeidet av	Nummer	Dato	
		HMS-avd.	HMSRV2603	4.3.2010	
HMS/KS		godkjent av	side	Erstatter	
		Rektor	3 av 3	9.2.2010	

Svært liten		restitusjonstid	1 dag	og respekt
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Risikoverdi = Sannsynlighet x Konsekvens

Beregn risikoverdi for Menneske. Enheten vurderer selv om de i tillegg vil beregne risikoverdi for Ytre miljø, Økonomi/materiell og Omdømme. I så fall beregnes disse hver for seg.

Til kolonnen ”Kommentarer/status, forslag til forebyggende og korrigerende tiltak”:

Tiltak kan påvirke både sannsynlighet og konsekvens. Prioriter tiltak som kan forhindre at hendelsen inntreffer, dvs. sannsynlighetsreducerende tiltak foran skjerpet beredskap, dvs. konsekvensreducerende tiltak.



EEC-Safety data sheet

SYNTHETIC PARAFFIN WAXES

page 1/2

Version: 7

Date: 2007.05.29

Revision: 2005.05.02

1. Identification of the substance and company

Sasolwax 5000

Manufacturer/supplier:

Sasol Wax GmbH
Worthdamm 13 - 27, D-20457 Hamburg
Tel.: +49-40-7 81 15 - 0, Fax: +49-40-7 81 15 - 415
Emergency telephone:
Safety engineer, Tel.: +49-40-7 81 15 -254 (24-hr-service)

2. Composition/Information on ingredients

Chemical characterisation:

Solid saturated hydrocarbons,
molecular formula C_nH_{2n+2}

CAS number: 8002-74-2

EINECS number: 232-315-6

Hazardous ingredients: None

3. Hazards identification

EEC-labelling: None

Potential dangers to health and environment:

Solid: None

Liquid: May cause burns as a result of the temperatures

Powder/vapour: May be irritating to the respiratory system

4. First-aid measures

Solid: None

Liquid: Same action to be taken as in the case of burns

Inhalation:

Powder/vapour: Take affected person to fresh air

Eye contact:

Liquid: Seek medical attention immediately

Skin contact:

Liquid: After contact with molten product remove soaked clothing immediately and cool the skin quickly with cold water. Consult a doctor if necessary. Do not pull solidified product from skin.

When swallowed:

Liquid: Same action to be taken as in the case of burns.

5. Fire-fighting measures

Suitable extinguishing media:

Carbon dioxide, foam, dry powder, sand

Extinguishing media that must not be used for safety reasons: Water

Special exposure hazards arising from the substance, its combustion products or from resulting gases:

In the case of fire, combustion gases are formed: Carbon monoxide (CO), carbon dioxide (CO₂), smoke

Special protective equipment for fire fighting:

Use self-contained breathing apparatus

Additional information:

Cool endangered containers externally with water

Fire class according to DIN-EN 2: B

6. Accidental release measures

Personal precautions:

Liquid: Remove persons to safety

Environmental precautions:

Solid/liquid: Do not allow to enter drains/surface waters/groundwater

Methods for cleaning up:

Liquid/solid: Allow to solidify / remove mechanically

Additional information:

Liquid: In case of the leakage of a large amount inform the responsible authorities

7. Handling and storage

Handling

Hints on safe handling:

No special measures necessary if used correctly.

Hints for protection against fire and explosion:

Product mist/dust can form an explosive mixture when in contact with air

Storage

Requirements for storage rooms and containers:

Solid: Store under cool, dry and light protected conditions

Liquid: Store in heatable tanks/containers.

Prolonged storage 20°C above the congealing point may interfere with product quality

Additional instructions for technical installations:

Liquid: Install containers in such a way, that filling containers is carried out without aerosol formation; earth containers well, prevent ignition sources.

Additional information:

Liquid: Product is not allowed to contain water at temperatures above 100°C (delay in boiling).

VCI storage class: 11

8. Exposure controls / personal protection

Ingredients with occupational exposure limits which should be monitored:

1. Germany/Switzerland/Austria:

There is no MAK-value for this product

2. Germany:

For aerosols and fine dust MAK-value of 6 mg/m³ has to be adhered to.

3. USA

TLV-value for paraffin vapours: 2 mg/m³

Personal protective equipment:

Hand protection:

Liquid: Heat resistant gloves

Eye protection:

Liquid: Safety goggles

Body protection:

Liquid: Protective clothing

Additional information:

When exceeding Maximum Work Place Concentration (MAK-value) for fine dust use appropriate breathing apparatus



EEC-Safety data sheet

page: 2/2

9. Physical and chemical properties

Physical state: At room temperature solid
Colour: White/whitish
Odour: Practically odourless
pH-value: Aqueous extract: Neutral
Congealing range (DIN-ISO 2207): 60 - 75°C
Flash point (DIN-ISO 2592): > 150°C
Lower explosion limit:
 Product dust/powder: > 15 g/m³
Upper explosion limit:
 Product dust/powder: > 1000 g/m³
Vapour pressure at 20°C: < 0,01 hPa
Density at 20°C (DIN 51 757): About 900 kg/m³
Solubility at 20°C
 - in water: Practically insoluble
 - in chlorinated hydrocarbons: Soluble
 - in benzene: Limited solubility
Viscosity at 100°C (DIN 51 562): 2,5 - 10 mm²/s

10. Stability / reactivity

Conditions to avoid:
 Under normal conditions no hazardous reactions likely
Material to avoid: Strong oxidising agents
Decomposition products:
 In case of incomplete combustion and/or thermal decomposition carbon monoxide, carbon dioxide and smoke may be formed.

11. Toxicological information

Acute oral toxicity: Rat non-toxic
Irritant effect on rabbits' skin: Non-irritant
Irritant effect on rabbits' eye: Non-irritant

12. Ecological information

The product is insoluble in water. It can be removed mechanically in a purification plant.
Mobility: In solid state the product cannot be dispersed into the environment. In liquid state, when discharged into the environment, the product spreads out over the surface and solidifies.
Information on elimination (persistence and degradability):
 The product is not readily biodegradable.
 The product has no detrimental effects on plants, animals or micro-organisms when used properly.

13. Disposal considerations

Product: After re-conditioning the product may be used again or thermally employed
GERMANY:
 Can be disposed off after consultation with the responsible authorities according to the following waste disposal codes (European Waste Catalogue):

EWC-Code	Description
05 01 06	Sludge from plant, equipment and maintenance operations
07 01 99	Wastes not otherwise specified
12 01 12	Spent waxes and fats

Packaging:

Pallets: Can be returned to supplier via the haulier/ forwarding agent
Cartons: Cartons with the "Resy" symbol may be returned to the associated used paper collecting point
Paper bags: Paper bags containing the "Repa-Sack"-symbol and "Chemical Classification 4" may be returned to the associated used paper collecting point
Big-Bags: Re-usable packaging can be returned to supplier
Drums: Drums can be returned to supplier

14. Transport information

(ADR/RID; BinSchV/ADNR/ADN; IMDG/GGVSee; ICAO-TI und IATA-DGR):
 Products like this one are not hazardous goods at temperatures < 100°C.
 In case of transport of liquids with temperatures of ≥100°C hazardous goods class 9, number 20c
UN: 99/3257 applies
IMDG-Code: 9027-1
Powder/pastilles transport: At temperatures above 25 °C cold transport is recommended

15. Regulatory information

The product does not require a hazard warning label in accordance with the EEC directives
National regulations
Germany:
Classification according to VbF: Not applicable
TA Luft:
 Threshold limit value TA Luft 150 mg/m³
 Mass flow 3 kg/h or more
 Substance class III, amendment E
Water hazard class (WGK): Not water endangering substance according to Annex I VwVwS (KBwS classification, identification no. 268)
StörfallVO: Not applicable for paraffin wax
 Observe principles of the trade association

16. Other information

None

This safety data sheet contains only safety related information. For specific data see product data sheet.

Although the information contained herein is presented in good faith and to the best of Sasol Wax's knowledge and experience, it is made without any warranty or guarantee whatsoever.

In case any questions should arise please call the number given in point 1.

Sasol Wax GmbH





PRODUCT DATA SHEET

SASOLWAX 5000

Date: 29.05.2007

Revision: 09.07.2001

Property	Method	Unit	Specification
Congealing point	ASTM D 938	°C	48,0 - 54,0
Oil content	ASTM D 721	%	0,00 - 4,20
Needle penetration at 25°C	ASTM D 1321	1/10mm	25 - 45
Colour Saybolt	ASTM D 156	-	28 - 30
Gas chromatogram	ASTM D 4626		pass

NOTICE: This product information is indicative and does not include any guarantee.

Sasol Wax GmbH

Worthdamm 13-27, 20457 Hamburg, Postanschrift: Postfach 11 02 49, 20402 Hamburg
Telefon: +49 40 78115-0 Telefax: +49 40 78115-777 wax@de.sasol.com www.sasolwax.com

Geschäftsführung: Helmuth Schulze-Trautmann, William Anthony Cook
Vorsitzender des Aufsichtsrats: Daniel Johan du Preez
Registergericht Hamburg: HRB 73963 USt-IdNr./VAT No.: DE 118 922 039 Steuer-Nr./Tax-No. 27/164/00096



**Safety data sheet**
according to 1907/2006/EC, Article 31

Printing date 15.02.2011

Revision: 15.02.2011

1 Identification of the substance/mixture and of the company/undertaking**Product identifier****Trade name: Sasolwax 5405****Relevant identified uses of the substance or mixture and uses advised against****Application of the substance / the preparation Additive****Details of the supplier of the safety data sheet****Manufacturer/Supplier:**

Sasol Wax GmbH

Worthdamm 13-27

D - 20457 Hamburg

Germany

Tel.: +49 (0)40 78115-450

Further information obtainable from: Product safety department, e-mail: norbert.petereit@de.sasol.com**Emergency telephone number: +49 (0) 40 78115 - 247****2 Hazards identification****Classification of the substance or mixture****Classification according to Directive 67/548/EEC or Directive 1999/45/EC Not applicable.****Information concerning particular hazards for human and environment:****Warning! Hot melt.****The product does not have to be labelled due to the calculation procedure of the "General Classification guideline for preparations of the EU" in the latest valid version.****Label elements****Labelling according to EU guidelines:****Observe the general safety regulations when handling chemicals.****The product is not subject to identification regulations under EU Directives and the Ordinance on Hazardous Materials (German GefStoffV).****Special labelling of certain preparations:****Safety data sheet available for professional user on request.****Other hazards****Results of PBT and vPvB assessment****PBT: Not applicable.****vPvB: Not applicable.****3 Composition/information on ingredients****Chemical characterization: Substances****CAS No. Description**

CAS: 8002-74-2 Paraffin waxes and hydrocarbon waxes

50-100%

EINECS: 232-315-6

Chemical characterization: Mixtures**Description: Mixture of substances listed below with nonhazardous additions.****Dangerous components: Void****4 First aid measures****Description of first aid measures****General information: No special measures required.****After inhalation: Supply fresh air; consult doctor in case of complaints.****After skin contact: After contact with the molten product, cool rapidly with cold water.****After eye contact: Rinse opened eye for several minutes under running water.****After swallowing: If symptoms persist consult doctor.**



Safety data sheet
according to 1907/2006/EC, Article 31

Printing date 15.02.2011

Revision: 15.02.2011

Trade name: Sasolwax 5405

(Contd. of page 2)

Penetration time of glove material

The exact break through time has to be found out by the manufacturer of the protective gloves and has to be observed.
Eye protection: Not required.

9 Physical and chemical properties

Information on basic physical and chemical properties

General Information

Appearance:

Form:	Solid
Colour:	White
Odour:	Odourless
Odour threshold:	Not determined.
pH-value:	Not applicable.

Change in condition

Melting point/Melting range:	Undetermined.
Boiling point/Boiling range:	Undetermined.
Congeaing point:	53-55 °C

Flash point: > 200°C

Flammability (solid, gaseous): Not determined.

Ignition temperature:

Decomposition temperature: Not determined.

Self-igniting: Product is not selfigniting.

Danger of explosion: Product does not present an explosion hazard.

Explosion limits:

Lower:	Wax dust / powder > 15 g/m ³
Upper:	Wax dust / powder: > 1000 g/m ³

Vapour pressure: Not applicable.

Density:	Not determined.
Relative density	Not determined.
Vapour density	Not applicable.
Evaporation rate	Not applicable.

Solubility in / Miscibility with water: Insoluble.

Segregation coefficient (n-octanol/water): Not determined.

Viscosity:

Dynamic:	Not applicable.
Kinematic:	Not applicable.

Other information: No further relevant information available.

10 Stability and reactivity

Reactivity

Chemical stability

Thermal decomposition / conditions to be avoided: No decomposition if used according to specifications.

Possibility of hazardous reactions: No dangerous reactions known.

Conditions to avoid: No further relevant information available.

Incompatible materials: No further relevant information available.

(Contd. on page 4)



Safety data sheet
according to 1907/2006/EC, Article 31

Printing date 15.02.2011

Revision: 15.02.2011

Trade name: Sasolwax 5405

(Contd. of page 4)

UN proper shipping name: ERWÄRMTER FLÜSSIGER STOFF, N.A.G.
Tunnel restriction code D
Remarks: Products < 100°C are no hazardous goods / class 9 does not apply.

Maritime transport IMDG:



IMDG Class: 9
UN Number: 3257
Label: 9
EMS Number: F-A, S-P
Marine pollutant: No
Proper shipping name: ELEVATED TEMPERATURE LIQUID, N.O.S.
Remarks: Products < 100°C are no hazardous goods / class 9 does not apply.

Air transport ICAO-TI and IATA-DGR:

ICAO/IATA Class: -
Special precautions for user Warning: Miscellaneous dangerous substances and articles.
Transport in bulk according to Annex II of MARPOL73/78 and the IBC Code Not applicable.

15 Regulatory information

Safety, health and environmental regulations/legislation specific for the substance or mixture

Labelling according to EU guidelines:

Observe the general safety regulations when handling chemicals.

The product is not subject to identification regulations under EU Directives and the Ordinance on Hazardous Materials (German GefStoffV).

Special labelling of certain preparations:

Safety data sheet available for professional user on request.

Chemical safety assessment: A Chemical Safety Assessment has not been carried out.

16 Other information

This information is based on our present knowledge. However, this shall not constitute a guarantee for any specific product features and shall not establish a legally valid contractual relationship.

Department issuing MSDS: Product safety department, Sasol Wax GmbH, Hamburg, Germany

* Data compared to the previous version altered.



SAFETY DATA SHEET
Paraffin Treatment Chemical 5% Solution

1 IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND OF THE COMPANY/UNDERTAKING

PRODUCT NAME Paraffin Treatment Chemical 5% Solution
 PRODUCT NO. PARAFFIN TRTMT
 SUPPLIER Champion Technologies
 Minto Avenue
 Altens Industrial Estate
 Aberdeen, AB12 3JZ
 00 44 1224 879022
 00 44 1224 876022
 eh.productstewardship@champ-tech.com
 EMERGENCY TELEPHONE [24Hr] 00 44 (0)1224 879022 OUT OF OFFICE HOURS ASK FOR HEALTH AND SAFETY

2 HAZARDS IDENTIFICATION

CLASSIFICATION (1999/45) N;R51/53. R66, R67.
 CLASSIFICATION (EC 1272/2008)
 Physical Not classified.
 Health EUH066;STOT Single 3 - H336
 Environmental Aquatic Chronic 2 - H411

LABEL IN ACCORDANCE WITH (EC) NO. 1272/2008



SIGNAL WORD Warning

HAZARD STATEMENTS

EUH066 Repeated exposure may cause skin dryness or cracking.
 H336 May cause drowsiness or dizziness.
 H411 Toxic to aquatic life with long lasting effects.

PRECAUTIONARY STATEMENTS

P273 Avoid release to the environment.
 P501a Dispose of contents/container to hazardous waste collection point in accordance with local authority requirements.

3 COMPOSITION/INFORMATION ON INGREDIENTS

Solvent naphtha (petroleum), heavy aromatic	90-100%
CAS-No.: 64742-94-5	EC No.: 265-198-5
CLASSIFICATION (EC 1272/2008) EUH066 STOT Single 3 - H336 Asp. Tox. 1 - H304 Aquatic Chronic 2 - H411	CLASSIFICATION (67/548) Xn;R65. N;R51/53, R66,R67.

The Full Text for all R-Phrases and Hazard Statements are Displayed in Section 16

4 FIRST-AID MEASURES

Paraffin Treatment Chemical 5% Solution

INHALATION

Remove victim immediately from source of exposure. Perform artificial respiration if breathing has stopped. Keep the affected person warm and at rest. Get prompt medical attention.

INGESTION

NEVER MAKE AN UNCONSCIOUS PERSON VOMIT OR DRINK FLUIDS! Rinse mouth thoroughly. Drink plenty of water. DO NOT induce vomiting. Get medical attention immediately. Provide rest, warmth and fresh air. If vomiting occurs, keep head low so that stomach content doesn't get into the lungs.

SKIN CONTACT

Promptly flush contaminated skin with soap or mild detergent and water. Promptly remove clothing if penetrated and flush the skin with water. Get medical attention if any discomfort continues.

EYE CONTACT

Make sure to remove any contact lenses from the eyes before rinsing. Promptly wash eyes with plenty of water while lifting the eye lids. Continue to rinse for at least 15 minutes and get medical attention.

5 FIRE-FIGHTING MEASURES

EXTINGUISHING MEDIA

Water spray. Foam. Dry chemicals. Carbon dioxide (CO2).

SPECIAL FIRE FIGHTING PROCEDURES

Avoid breathing fire vapours. Keep up-wind to avoid fumes. Cool containers exposed to flames with water until well after the fire is out. Avoid water in straight hose stream; will scatter and spread fire. Keep run-off water out of sewers and water sources. Dike for water control.

UNUSUAL FIRE & EXPLOSION HAZARDS

May ignite at high temperature.

SPECIFIC HAZARDS

Fire or high temperatures create: Oxides of: Carbon.

PROTECTIVE MEASURES IN FIRE

Wear full protective clothing. Use air-supplied respirator during fire fighting.

6 ACCIDENTAL RELEASE MEASURES

PERSONAL PRECAUTIONS

Wear protective clothing as described in Section 8 of this safety data sheet. In case of inadequate ventilation, use respiratory protection. Do not smoke, use open fire or other sources of ignition. Avoid contact with skin and eyes.

ENVIRONMENTAL PRECAUTIONS

Do not discharge into drains, water courses or onto the ground.

SPILL CLEAN UP METHODS

Runoff or release to sewer, waterway or ground is forbidden. Absorb in vermiculite, dry sand or earth and place into containers. Collect and reclaim or dispose in sealed containers in licensed waste.

7 HANDLING AND STORAGE

USAGE PRECAUTIONS

Container must be kept tightly closed. Provide good ventilation. Avoid spilling, skin and eye contact. Keep away from heat, sparks and open flame.

STORAGE PRECAUTIONS

Use container made of: Carbon steel. Stainless steel. Suitable plastic material.

8 EXPOSURE CONTROLS/PERSONAL PROTECTION

Name	Std	TWA - 8 hrs		STEL - 15 min		Notes
Solvent naphtha (petroleum), heavy aromatic	SUP	17 ppm	100 mg/m3			

INGREDIENT COMMENTS

SUP = Supplier's recommendation.

PROTECTIVE EQUIPMENT

Paraffin Treatment Chemical 5% Solution



ENGINEERING MEASURES

Provide adequate general and local exhaust ventilation.

RESPIRATORY EQUIPMENT

Respiratory protection must be used if air contamination exceeds acceptable level. Wear full face mask supplied with: Gas cartridge suitable for organic substances.

HAND PROTECTION

Use protective gloves made of: Nitrile. The most suitable glove must be chosen in consultation with the gloves supplier, who can inform about the breakthrough time of the glove material. Be aware that the liquid may penetrate the gloves. Frequent change is advisable.

EYE PROTECTION

Use approved safety goggles or face shield.

OTHER PROTECTION

Wear appropriate clothing to prevent any possibility of skin contact.

HYGIENE MEASURES

When using do not eat, drink or smoke. **DO NOT SMOKE IN WORK AREA!** Wash at the end of each work shift and before eating, smoking and using the toilet. Use appropriate skin cream to prevent drying of skin.

9 PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE	Liquid		
ODOUR	Aromatic.		
SOLUBILITY	Immiscible with water Soluble in:	Organic solvents	
BOILING POINT (°C)	178 - 209	MELTING POINT (°C)	Pour point - < -20
RELATIVE DENSITY	0.89 @ 20 °c	VAPOUR PRESSURE	1.0 hPa @ 20 °c
pH-VALUE, DILUTED SOLUTION	Not determined	VISCOSITY	~ 2 mPas @ 20 °c
FLASH POINT (°C)	~ 63	AUTO IGNITION TEMPERATURE (°C)	> 450
FLAMMABILITY LIMIT - LOWER(%)	0.6	FLAMMABILITY LIMIT - UPPER(%)	7.0

10 STABILITY AND REACTIVITY

STABILITY

Stable under normal temperature conditions and recommended use.

CONDITIONS TO AVOID

Avoid heat, flames and other sources of ignition.

MATERIALS TO AVOID

Strong acids. Strong oxidising substances.

HAZARDOUS DECOMPOSITION PRODUCTS

Fire or high temperatures create: Oxides of: Carbon.

11 TOXICOLOGICAL INFORMATION

GENERAL INFORMATION

Prolonged and repeated contact with solvents over a long period may lead to permanent health problems.

INHALATION

Vapour may affect central nervous system and cause headache, discomfort, vomiting or intoxication. Vapours may cause drowsiness and dizziness.

INGESTION

Harmful: may cause lung damage if swallowed. Pneumonia may be the result if vomited material containing solvents reaches the lungs.

SKIN CONTACT

Prolonged contact may cause dryness of the skin. Acts as a defatting agent on skin. May cause cracking of skin, and eczema.

EYE CONTACT

No specific health warnings noted. Spray and vapour in the eyes may cause irritation and smarting.

12 ECOLOGICAL INFORMATION

Paraffin Treatment Chemical 5% Solution

RISK PHRASES IN FULL

R65	Harmful: may cause lung damage if swallowed.
R66	Repeated exposure may cause skin dryness or cracking.
R51/53	Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
R67	Vapours may cause drowsiness and dizziness.

HAZARD STATEMENTS IN FULL

H304	May be fatal if swallowed and enters airways.
H336	May cause drowsiness or dizziness.
EUH066	Repeated exposure may cause skin dryness or cracking.
H411	Toxic to aquatic life with long lasting effects.

DISCLAIMER

The information provided in this SDS is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal, and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process unless specified in the text.



SAFETY DATA SHEET

according to Regulation (EC) No. 1907/2006

Revision Date 05.11.2010

Version 1.4

1. Identification of the substance/mixture and of the company/undertaking

1.1 Product identifier

Catalogue No. 104379
Product name n-Heptane for analysis EMSURE® Reag. Ph Eur
REACH Registration Number A registration number is not available for this substance as the substance or its use are exempted from registration according to Article 2 REACH Regulation (EC) No 1907/2006, the annual tonnage does not require a registration or the registration is envisaged for a later registration deadline.

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses Reagent for analysis, Chemical production
For additional information on uses please refer to the Merck Chemicals portal (www.merck-chemicals.com).

1.3 Details of the supplier of the safety data sheet

Responsible Department EQ-RS * e-mail: prodsafe@merckgroup.com
Regional representation: Merck Chemicals Ltd * Boulevard Industrial Park * Padge Road *
Beeston * Nottingham * NG9 2JR * Tel. 01159 430840 *
information@merckchem.co.uk.

1.4 Emergency telephone number +49 (0) 6151 722440

2. Hazards identification

2.1 Classification of the substance or mixture

Classification (REGULATION (EC) No 1272/2008)

Flammable liquid, Category 2, H225

Aspiration hazard, Category 1, H304

Skin irritation, Category 2, H315

Specific target organ toxicity - single exposure, Category 3, H336

Acute aquatic toxicity, Category 1, H400

Chronic aquatic toxicity, Category 1, H410

For the full text of the H-Statements mentioned in this Section, see Section 16.

Classification (67/548/EEC or 1999/45/EC)

F; R11

Xi; R38

Xn; R65

N; R50/53

For the full text of the R-phrases mentioned in this Section, see Section 16.

SAFETY DATA SHEET
according to Regulation (EC) No. 1907/2006

Catalogue No. 104379
Product name n-Heptane for analysis EMSURE® Reag. Ph Eur

2.2 Label elements

Labelling (REGULATION (EC) No 1272/2008)

Hazard pictograms



Signal word
Danger

Hazard statements

H225 Highly flammable liquid and vapour.
H304 May be fatal if swallowed and enters airways.
H315 Causes skin irritation.
H336 May cause drowsiness or dizziness.
H410 Very toxic to aquatic life with long lasting effects.

Precautionary statements

P210 Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
P273 Avoid release to the environment.
P301 + P310 IF SWALLOWED: Immediately call a POISON CENTER or doctor/ physician.
P331 Do NOT induce vomiting.
P302 + P352 IF ON SKIN: Wash with plenty of soap and water.
P403 + P235 Store in a well-ventilated place. Keep cool.

Reduced labelling (≤125 ml)

Hazard pictograms



Signal word
Danger

Hazard statements

H304 May be fatal if swallowed and enters airways.

Precautionary statements

P301 + P310 IF SWALLOWED: Immediately call a POISON CENTER or doctor/ physician.

Index-No. 601-008-00-2

Labelling (67/548/EEC or 1999/45/EC)

Symbol(s)	F Xn N	Highly flammable Harmful Dangerous for the environment
R-phrases(s)	11-38-50/53-65-67	Highly flammable. Irritating to skin. Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. Harmful: may cause lung damage if swallowed. Vapours may cause drowsiness and dizziness.
S-phrases(s)	9-16-29-33-60-61-62	Keep container in a well-ventilated place. Keep away from sources of ignition - No smoking. Do not empty into drains. Take precautionary measures against static discharges. This material and its container must be disposed of as hazardous

SAFETY DATA SHEET
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Product name n-Heptane for analysis EMSURE® Reag. Ph Eur

waste. Avoid release to the environment. Refer to special instructions/ Safety data sheets. If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.

EC-No. 205-563-8

EC Label

Reduced labelling (≤125 ml)

Symbol(s) F
Xn
N
R-phrases) 65
S-phrases) 62

Highly flammable
Harmful
Dangerous for the environment
Harmful: may cause lung damage if swallowed.
If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.

2.3 Other hazards
None known.

3. Composition/Information on ingredients

Formula	CH ₃ (CH ₂) ₆ CH ₃	C ₇ H ₁₆ (Hill)
CAS-No.	142-82-5	
Index-No.	601-008-00-2	
EC-No.	205-563-8	
Molar mass	100,2 g/mol	

4. First aid measures

4.1 Description of first aid measures

After inhalation: fresh air. Consult doctor if feeling unwell.

After skin contact: wash off with plenty of water. Remove contaminated clothing.

After eye contact: rinse out with plenty of water with the eyelid held wide open. Call in ophthalmologist if necessary.

If swallowed Caution Aspiration hazard Keep respiratory tract clear. Call a physician immediately. In case of spontaneous vomiting: Risk of aspiration. Pulmonary failure possible. Call in physician.

4.2 Most important symptoms and effects, both acute and delayed

irritant effects, Drowsiness, Unconsciousness, narcosis, Headache, drowsiness, Vertigo, death

4.3 Indication of immediate medical attention and special treatment needed

No information available.

5. Fire-fighting measures

5.1 Extinguishing media

Suitable extinguishing media

Carbon dioxide (CO₂), Foam, Dry powder

Unsuitable extinguishing media

For this substance/mixture no limitations of extinguishing agents are given.

5.2 Special hazards arising from the substance or mixture

Combustible material, Vapours are heavier than air and may spread along floors.

Forms explosive mixtures with air at ambient temperatures.

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Pay attention to flashback.
Development of hazardous combustion gases or vapours possible in the event of fire.

5.3 Advice for firefighters

Special protective equipment for fire-fighters

Stay in danger area only with self-contained breathing apparatus. Prevent skin contact by keeping a safe distance or by wearing suitable protective clothing.

Further information

Suppress (knock down) gases/vapours/mists with a water spray jet. Prevent fire extinguishing water from contaminating surface water or the ground water system. Remove container from danger zone and cool with water.

6. Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Advice for non-emergency personnel: Avoid substance contact. Do not breathe vapours, aerosols. Ensure adequate ventilation. Evacuate the danger area, observe emergency procedures, consult an expert.

Advice for emergency responders: Protective equipment see section 8.

6.2 Environmental precautions

Do not empty into drains. Risk of explosion.

6.3 Methods and materials for containment and cleaning up

Cover drains. Collect, bind, and pump off spills.

Observe possible material restrictions (see sections 7.2 and 10.5).

Take up with liquid-absorbent material (e.g. Chemisorb®). Dispose of properly. Clean up affected area.

6.4 Reference to other sections

Indications about waste treatment see section 13.

7. Handling and storage

7.1 Precautions for safe handling

Work under hood. Do not inhale substance. Avoid generation of vapours/aerosols.

Advice on protection against fire and explosion

Keep away from open flames, hot surfaces and sources of ignition. Take precautionary measures against static discharge.

7.2 Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place. Keep away from heat and sources of ignition.

Storage temperature: no restrictions.

7.3 Specific end uses

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated.

8. Exposure controls/personal protection

8.1 Control parameters

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Components with workplace control parameters

Components

Basis	Value	Threshold limits	Ceiling Limit Value, Remarks
<i>n-heptane (142-82-5)</i>			
ECTLV	Time Weighted Average (TWA):	500 ppm 2.085 mg/m ³	
EH40 WEL	Time Weighted Average (TWA):	500 ppm	
ECTLV	Time Weighted Average (TWA):	500 ppm 2.085 mg/m ³	Indicative

Recommended monitoring procedures

Methods for measurement of the workplace atmosphere have to correspond to the requirements of norms DIN EN 482 and DIN EN 689.

8.2 Exposure controls

Engineering measures

Technical measures and appropriate working operations should be given priority over the use of personal protective equipment.

See section 7.1.

Individual protection measures

Protective clothing needs to be selected specifically for the workplace, depending on concentrations and quantities of the hazardous substances handled. The chemical resistance of the protective equipment should be enquired at the respective supplier.

Hygiene measures

Immediately change contaminated clothing. Apply preventive skin protection. Wash hands and face after working with substance. Work under hood. Do not inhale substance.

Eye/face protection

Safety glasses

Hand protection

full contact:

Glove material:	Nitrile rubber
Glove thickness:	0,40 mm
Break through time:	> 480 min

splash contact:

Glove material:	polychloroprene
Glove thickness:	0,65 mm
Break through time:	> 60 min

The protective gloves to be used must comply with the specifications of EC Directive 89/686/EEC and the related standard EN374, for example KCL 730 Camatril® -Velours (full contact), KCL 720 Camapren® (splash contact).

The breakthrough times stated above were determined by KCL in laboratory tests acc. to EN374 with samples of the recommended glove types.

This recommendation applies only to the product stated in the safety data sheet (>,<) supplied by us and for the designated use. When dissolving in or mixing with other substances and under conditions deviating from those stated in EN374 please contact the supplier of CE-approved gloves (e.g. KCL GmbH, D-36124 Eichenzell, Internet: www.kcl.de).

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Other protective equipment:

Flame retardant antistatic protective clothing

Respiratory protection

required when vapours/aerosols are generated.

Recommended Filter type: Filter A (acc. to DIN 3181) for vapours of organic compounds

The entrepreneur has to ensure that maintenance, cleaning and testing of respiratory protective devices are carried out according to the instructions of the producer. These measures have to be properly documented.

Environmental exposure controls

Do not empty into drains.

Risk of explosion.

9. Physical and chemical properties

9.1 Information on basic physical and chemical properties

Form	liquid
Colour	colourless
Odour	characteristic
Odour Threshold	No information available.
pH	No information available.
Melting point	-90,5 °C
Boiling point/boiling range	97 - 98 °C at 1.013 hPa
Flash point	-4 °C Method: c.c.
Evaporation rate	No information available.
Flammability (solid, gas)	No information available.
Lower explosion limit	1 %(V)
Upper explosion limit	7 %(V)
Vapour pressure	48 hPa at 20 °C
Relative vapour density	3,46
Relative density	0,68 g/cm ³ at 20 °C
Water solubility	0,05 g/l at 20 °C

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Product name n-Heptane for analysis EMSURE® Reag. Ph Eur

Partition coefficient: n-octanol/water log Pow: 4,66
Method: (experimental)
(Lit.) A remarkable bioaccumulation potential is expected (log Po/w >3).

Autoignition temperature No information available.

Decomposition temperature No information available.

Viscosity, dynamic 0,42 mPa.s
at 20 °C

Explosive properties No information available.

Oxidizing properties No information available.

9.2 Other data

Ignition temperature 215 °C

10. Stability and reactivity

10.1 Reactivity

Vapours may form explosive mixture with air.

10.2 Chemical stability

The product is chemically stable under standard ambient conditions (room temperature) .

10.3 Possibility of hazardous reactions

Risk of ignition or formation of inflammable gases or vapours with:

Strong oxidizing agents

phosphorus

Chlorine

10.4 Conditions to avoid

Warming.

A range from approx. 15 Kelvin below the flash point is to be rated as critical.

10.5 Incompatible materials

rubber, various plastics

10.6 Hazardous decomposition products

no information available

11. Toxicological information

11.1 Information on toxicological effects

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according to Regulation (EC) No. 1907/2006

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Product name n-Heptane for analysis EMSURE® Reag. Ph Eur

Acute oral toxicity

LD50 rat

Dose: > 2.000 mg/kg

(External MSDS)

Symptoms: drowsiness, Drowsiness, Headache, Vertigo, Unconsciousness, Irritations of mucous membranes in the mouth, pharynx, oesophagus and gastrointestinal tract., Risk of aspiration upon vomiting., Aspiration may cause pulmonary oedema and pneumonitis., Pulmonary failure possible after aspiration of vomit.

Acute inhalation toxicity

LC50 rat

Dose: 103 g/m³, 4 h

(RTECS)

Symptoms: Drowsiness, drowsiness, Irritation symptoms in the respiratory tract.

Acute dermal toxicity

LD50 rabbit

Dose: 3.400 mg/kg

(IUCLID)

Skin irritation

Irritations

Repeated or prolonged exposure may cause skin irritation and dermatitis, due to degreasing properties of the product.

Causes skin irritation.

Eye irritation

rabbit

Result: No eye irritation

(IUCLID)

Genotoxicity in vitro

Ames test

Salmonella typhimurium

Result: negative

(IUCLID)

Specific target organ toxicity - single exposure

May cause drowsiness or dizziness.

Specific target organ toxicity - repeated exposure

The substance or mixture is not classified as specific target organ toxicant, repeated exposure.

Aspiration hazard

Aspiration hazard, Aspiration may cause pulmonary oedema and pneumonitis.

11.2 Further information

Further information

If swallowed

narcosis, death

Other information

It generally applies for aliphatic hydrocarbons with 6 - 18 carbon atoms that they may cause pneumonia, in some cases also pulmonary oedema, upon direct inhalation, i.e. in conditions that can occur only in very special circumstances (nebulizations, spraying, inhalation of aerosols and similar). After absorption of very large quantities: narcosis.

Further data:

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Product name n-Heptane for analysis EMSURE® Reag. Ph Eur

Handle in accordance with good industrial hygiene and safety practice.

12. Ecological information

12.1 Toxicity

Toxicity to fish

LC50

Species: Carassius auratus (goldfish)

Dose: 4 mg/l

Exposure time: 24 h

Method: ASTM D1345

(Lit.)

Toxicity to daphnia and other aquatic invertebrates.

EC50

Species: Daphnia magna (Water flea)

Dose: 1,5 mg/l

Exposure time: 48 h

(Lit.)

12.2 Persistence and degradability

Biochemical Oxygen Demand (BOD)

1.920 mg/g (5 d)

(IUCLID)

Theoretical oxygen demand (ThOD)

3.500 mg/g

(Lit.)

Ratio BOD/ThBOD

BOD5 55 %

(Lit.)

12.3 Bioaccumulative potential

Partition coefficient: n-octanol/water

log Pow: 4,66

Method: (experimental)

(Lit.) A remarkable bioaccumulation potential is expected (log Po/w >3).

12.4 Mobility in soil

No information available.

12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted.

12.6 Other adverse effects

Henry constant

208678 Pa·m³/mol

Method: (calculated)

(Lit.) Distribution preferentially in air.

Additional ecological information

Do not allow to run into surface waters, wastewater, or soil.

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according to Regulation (EC) No. 1907/2006

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Product name n-Heptane for analysis EMSURE® Reag. Ph Eur

13. Disposal considerations

Waste treatment methods

Waste material must be disposed of in accordance with the Directive on waste 2008/98/EC as well as other national and local regulations. Leave chemicals in original containers. No mixing with other waste. Handle uncleaned containers like the product itself.

See www.retrologistik.com for processes regarding the return of chemicals and containers, or contact us there if you have further questions.

14. Transport information

ADR/RID

UN 1206 HEPTANES, 3, II

IATA

UN 1206 HEPTANES, 3, II

IMDG

UN 1206 HEPTANES, 3, II

EmS

F-E S-D

The transport regulations are cited according to international regulations and in the form applicable in Germany. Possible national deviations in other countries are not considered.

15. Regulatory information

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture

EU regulations

Major Accident Hazard Legislation 96/82/EC
Highly flammable
7b
Quantity 1: 5.000 t
Quantity 2: 50.000 t

96/82/EC
Dangerous for the environment
9a
Quantity 1: 100 t
Quantity 2: 200 t

Occupational restrictions Take note of Dir 94/33/EC on the protection of young people at work.

National legislation

Storage class VCI 3 Flammable Liquids

15.2 Chemical Safety Assessment

For this product a chemical safety assessment was not carried out.

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Product name n-Heptane for analysis EMSURE® Reag. Ph Eur

16. Other information

Full text of H-Statements referred to under sections 2 and 3.

H225	Highly flammable liquid and vapour.
H304	May be fatal if swallowed and enters airways.
H315	Causes skin irritation.
H336	May cause drowsiness or dizziness.
H400	Very toxic to aquatic life.
H410	Very toxic to aquatic life with long lasting effects.

Full text of R-phrases referred to under sections 2 and 3

R11	Highly flammable.
R38	Irritating to skin.
R50/53	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
R65	Harmful: may cause lung damage if swallowed.

Training advice

Provide adequate information, instruction and training for operators.

Key or legend to abbreviations and acronyms used in the safety data sheet
Used abbreviations and acronyms can be looked up at www.wikipedia.org.

The information contained herein is based on the present state of our knowledge. It characterises the product with regard to the appropriate safety precautions. It does not represent a guarantee of any properties of the product.

SAFETY DATA SHEET

according to Regulation (EC) No. 1907/2006

Version 4.1 Revision Date 03.12.2011

Print Date 25.06.2012

1. IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING**1.1 Product identifiers**

Product name : Methylcyclohexane

Product Number : 259691
Brand : Sigma-Aldrich
Index-No. : 601-018-00-7
CAS-No. : 108-87-2**1.2 Relevant identified uses of the substance or mixture and uses advised against**

Identified uses : Laboratory chemicals, Manufacture of substances

1.3 Details of the supplier of the safety data sheetCompany : Sigma-Aldrich Company Ltd.
The Old Brickyard
NEW ROAD, GILLINGHAM
Dorset
SP8 4XT
UNITED KINGDOMTelephone : +44 (0)1747 833000
Fax : +44 (0)1747 833313
E-mail address : eurtechserv@sial.com**1.4 Emergency telephone number**

Emergency Phone # : +44 (0)1747 833100

2. HAZARDS IDENTIFICATION**2.1 Classification of the substance or mixture****Classification according to Regulation (EC) No 1272/2008 [EU-GHS/CLP]**Flammable liquids (Category 2)
Aspiration hazard (Category 1)
Skin irritation (Category 2)
Specific target organ toxicity - single exposure (Category 3)
Chronic aquatic toxicity (Category 2)**Classification according to EU Directives 67/548/EEC or 1999/45/EC**

Highly flammable. Harmful: may cause lung damage if swallowed. Irritating to skin. Vapours may cause drowsiness and dizziness. Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

2.2 Label elements**Labelling according Regulation (EC) No 1272/2008 [CLP]**

Pictogram



Signal word : Danger

Hazard statement(s)

H225 : Highly flammable liquid and vapour.
H304 : May be fatal if swallowed and enters airways.
H315 : Causes skin irritation.
H336 : May cause drowsiness or dizziness.

H411	Toxic to aquatic life with long lasting effects.
Precautionary statement(s)	
P210	Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
P261	Avoid breathing dust/ fume/ gas/ mist/ vapours/ spray.
P273	Avoid release to the environment.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER or doctor/ physician.
P331	Do NOT induce vomiting.
Supplemental Hazard Statements	none

According to European Directive 67/548/EEC as amended.

Hazard symbol(s)



R-phrases(s)	
R11	Highly flammable.
R38	Irritating to skin.
R51/53	Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
R65	Harmful: may cause lung damage if swallowed.
R67	Vapours may cause drowsiness and dizziness.
S-phrases(s)	
S 9	Keep container in a well-ventilated place.
S16	Keep away from sources of ignition - No smoking.
S33	Take precautionary measures against static discharges.
S61	Avoid release to the environment. Refer to special instructions/ Safety data sheets.
S62	If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.

2.3 Other hazards - none

3. COMPOSITION/INFORMATION ON INGREDIENTS

3.1 Substances

Synonyms	:	Hexahydrotoluene
Formula	:	C ₇ H ₁₄
Molecular Weight	:	98.19 g/mol

Component	Concentration
Methylcyclohexane	
CAS-No.	108-87-2
EC-No.	203-624-3
Index-No.	601-018-00-7

4. FIRST AID MEASURES

4.1 Description of first aid measures

General advice

Consult a physician. Show this safety data sheet to the doctor in attendance.

If inhaled

If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact

Wash off with soap and plenty of water. Consult a physician.

In case of eye contact

Flush eyes with water as a precaution.

If swallowed

Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed

prolonged or repeated exposure can cause:; narcosis

4.3 Indication of any immediate medical attention and special treatment needed

no data available

5. FIREFIGHTING MEASURES**5.1 Extinguishing media****Suitable extinguishing media**

Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture

Carbon oxides

5.3 Advice for firefighters

Wear self contained breathing apparatus for fire fighting if necessary.

5.4 Further information

Use water spray to cool unopened containers.

6. ACCIDENTAL RELEASE MEASURES**6.1 Personal precautions, protective equipment and emergency procedures**

Use personal protective equipment. Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.

6.2 Environmental precautions

Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up

Contain spillage, and then collect with an electrically protected vacuum cleaner or by wet-brushing and place in container for disposal according to local regulations (see section 13).

6.4 Reference to other sections

For disposal see section 13.

7. HANDLING AND STORAGE**7.1 Precautions for safe handling**

Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.

Keep away from sources of ignition - No smoking. Take measures to prevent the build up of electrostatic charge.

7.2 Conditions for safe storage, including any incompatibilities

Store in cool place. Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

7.3 Specific end uses

no data available

8. EXPOSURE CONTROLS/PERSONAL PROTECTION**8.1 Control parameters****Components with workplace control parameters**

Contains no substances with occupational exposure limit values.

8.2 Exposure controls

Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection

Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

Body Protection

Complete suit protecting against chemicals, Flame retardant antistatic protective clothing, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection

Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

9. PHYSICAL AND CHEMICAL PROPERTIES

9.1 Information on basic physical and chemical properties

- | | |
|---|--|
| a) Appearance | Form: liquid
Colour: colourless |
| b) Odour | no data available |
| c) Odour Threshold | no data available |
| d) pH | no data available |
| e) Melting point/freezing point | Melting point/range: -126 °C - lit. |
| f) Initial boiling point and boiling range | 101 °C - lit. |
| g) Flash point | -4.0 °C - closed cup |
| h) Evaporation rate | no data available |
| i) Flammability (solid, gas) | no data available |
| j) Upper/lower flammability or explosive limits | Upper explosion limit: 6.7 %(V)
Lower explosion limit: 1.1 %(V) |
| k) Vapour pressure | 110.9 hPa at 37.7 °C
49.3 hPa at 20.0 °C |
| l) Vapour density | no data available |
| m) Relative density | 0.77 g/cm ³ at 25 °C |
| n) Water solubility | no data available |
| o) Partition coefficient: n-octanol/water | no data available |

- | | |
|------------------------------|-------------------|
| p) Autoignition temperature | 283.0 °C |
| q) Decomposition temperature | no data available |
| r) Viscosity | no data available |
| s) Explosive properties | no data available |
| t) Oxidizing properties | no data available |

9.2 Other safety information

no data available

10. STABILITY AND REACTIVITY

10.1 Reactivity

no data available

10.2 Chemical stability

no data available

10.3 Possibility of hazardous reactions

no data available

10.4 Conditions to avoid

Heat, flames and sparks. Extremes of temperature and direct sunlight.

10.5 Incompatible materials

Strong oxidizing agents

10.6 Hazardous decomposition products

Other decomposition products - no data available

11. TOXICOLOGICAL INFORMATION

11.1 Information on toxicological effects

Acute toxicity

LD50 Oral - mouse - 2,250 mg/kg

LC50 Inhalation - mouse - 2 h - 41,500 mg/m³

Skin corrosion/irritation

no data available

Serious eye damage/eye irritation

no data available

Respiratory or skin sensitization

no data available

Germ cell mutagenicity

no data available

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

Reproductive toxicity

no data available

Specific target organ toxicity - single exposure

May cause drowsiness or dizziness.

Specific target organ toxicity - repeated exposure

no data available

Aspiration hazard

May be fatal if swallowed and enters airways.

Potential health effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation. Vapours may cause drowsiness and dizziness.
Ingestion	May be harmful if swallowed. Aspiration hazard if swallowed - can enter lungs and cause damage.
Skin	May be harmful if absorbed through skin. Causes skin irritation.

Signs and Symptoms of Exposure

prolonged or repeated exposure can cause: , narcosis

Additional Information

RTECS: GV6125000

12. ECOLOGICAL INFORMATION

12.1 Toxicity

Toxicity to fish LC50 - other fish - 5.8 mg/l - 96.0 h

Toxicity to daphnia and other aquatic invertebrates. Immobilization EC50 - Daphnia magna (Water flea) - 1.47 mg/l - 48 h

12.2 Persistence and degradability

no data available

12.3 Bioaccumulative potential

no data available

12.4 Mobility in soil

no data available

12.5 Results of PBT and vPvB assessment

no data available

12.6 Other adverse effects

Toxic to aquatic life.

13. DISPOSAL CONSIDERATIONS

13.1 Waste treatment methods

Product

Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging

Dispose of as unused product.

14. TRANSPORT INFORMATION

14.1 UN number

ADR/RID: 2296

IMDG: 2296

IATA: 2296

14.2 UN proper shipping name

ADR/RID: METHYLCYCLOHEXANE

IMDG: METHYLCYCLOHEXANE

IATA: Methylcyclohexane

14.3 Transport hazard class(es)

ADR/RID: 3

IMDG: 3

IATA: 3

14.4 Packaging group

ADR/RID: II

IMDG: II

IATA: II

14.5 Environmental hazards

ADR/RID: yes

IMDG Marine pollutant: yes

IATA: no

14.6 Special precautions for user
no data available

15. REGULATORY INFORMATION

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture
no data available

15.2 Chemical Safety Assessment
no data available

16. OTHER INFORMATION

Further information

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Sikkerhetsbrosjyre ifølge Forordning (EC) nr. 1907/2006 (REACH)

Revisjonsdato: 8-Aug-2011

Versjonsnummer: 2

1. Beskrivelse av stoffet/blandingen og det aktuelle selskapet

1.1 Produktidentifikator

Produktnavn:/betegnelse	Toluen AnalaR NORMAPUR® ACS, ISO, Reag.Ph.Eur. analytisk reagens
Produkt-nr.	28676 (VWR International, Prolabo)
Stoff-navn	Toluen
CAS-nr.	108-88-3
EU-identifikasjonsnummer	601-021-00-3
REACH registreringsnr.	Not yet communicated down the supply chain.
Andre betegnelser	

1.2 Relevante identifiserte bruksmåter for stoffet eller blandingen og bruksmåter det advares mot

Identifiserte relevante bruksmåter for laboratorier og kjemisk produksjon.

1.3 Informasjon om leverandøren av sikkerhetsdatabladet

leverandør (produsent/importør/etterordnet bruker/forhandler)

VWR International AS

Gate	Haavard Martinsens vei 30
Postnummer/Sted	0978 Oslo
Land	Norge
Telefon	+47 02290
Telefax	
E-post (kompetent person)	vwrds@eu.vwr.com

1.4 Nødnummer

Telefon (+47) 22591300 (Giftinformasjonen)

2. Farlige egenskaper

2.1 Klassifisering av stoffet eller blandingen

2.1.1 klassifisering ifølge forordning (EF) nr. 1272/2008 [CLP]

fareklasser og farekategorier	Hensvisninger om fare	innordningsmetode	bemerkning
Flammable liquid, category 2	H225		
Reproductive toxicity, category 2	H361d		

Aspiration hazard, category 1	H304		
Specific target organ toxicity (repeated exposure), category 2	H373		
Skin irritation, category 2	H315		
Specific target organ toxicity (single exposure), category 3, narcotic effect	H336		

2.1.2 Plassering etter direktiv 67/548/EØS eller 1999/45/EF

Faresymboler:	R-settninger
F	R11
Xi	R38
Xn	R48/20
Xn	R63
Xn	R65
	R67

2.2 Kjennetegnselementer

2.2.1 Kjennetegn ifølge forordning (EF) nr. 1272/2008 [CLP]

Farepiktogrammer:



Signalord

Danger

Hensvisninger om fare

H225	Meget brannfarlig væske og damp.
H361d	Mistenkes for å kunne gi fosterskader.
H304	Kan være dødelig ved svelging om det kommer ned i luftveiene.
H373	Kan forårsake skade på organer ved langvarig eller gjentatt eksponering.
H315	Irriterer huden.
H336	Kan forårsake døsighet eller svimmelhet.

Sikkerhetsanvisninger

P201	Innhent særskilt instruks før bruk.
P210	Holdes vekk fra varme/gnister/åpen flamme/varme overflater. - Røyking forbudt.
P243	Treff tiltak mot statisk elektrisitet.
P281	Bruk påkrevd personlig verneutstyr.
P301+P331	VED SVELGING: IKKE fremkall brekning.
P302+P352	VED HUDKONTAKT: Vask med mye såpe og vann.
P304+P340	VED INNÅNDING: Flytt personen til frisk luft og sørg for at vedkommende hviler i en stilling som letter åndedrettet.
P309+P310	Ved eksponering eller ubehag: Kontakt umiddelbart et GIFTINFORMASJONSSENTER eller lege.

2.2.2 Merking (67/548/EEC eller 1999/45/EU)

Faresymboler:

F, Xn

R-settninger

R11	Meget brannfarlig.
R38	Irriterer huden.
R48/20	Farlig: Alvorlige helsefare ved lengre tids påvirkning ved innånding.
R63	Mulig fare for fosterskader.
R65	Farlig: kan forårsake lungeskade ved svelging.
R67	Damp kan forårsake dødsighet og svimmelhet.

Sikkerhets-setning

S36/37	Bruk egnede verneklær og vernehansker.
S46	Ved svelging, kontakt lege omgående og vis denne beholderen eller etiketten.
S62	Ved svelging må ikke brekning fremkalles: Kontakt lege omgående og vis denne etikett eller emballasje.

2.3 Andre farer

ingen

3. Opplysninger om innhold sammensetning

Formel:	$C_6H_5CH_3$
Molarmasse:	92,14 g/mol
CAS-nr.	108-88-3
EU-nummer	203-625-9
EU-identifikationsnummer	601-021-00-3

4. Første hjelp

4.1 Generell informasjon

Ved eksponering: Kontakt umiddelbart et GIFTINFORMASJONSSENTER eller lege. Ved bevisstløshet: bring personen i stabil liggestilling på siden og kontakt lege. Aldri gi noe gjennom munnen til en ubevisst person eller til en som har krampes. Bytt tilsølt, gjennomvåt bekledding. Impliserte må ikke være uten tilsyn.

4.2 Etter innånding

Kontakt umiddelbart et GIFTINFORMASJONSSENTER eller lege. Personen føres ut i frisk luft og holdes varm og i ro. Ved pusteproblemer eller pustestans begynn kunstig ånderett.

4.3 Ved hudkontakt

Får man stoff på huden, vask straks med store mengder vann og såpe. Ta av tilmussede eller kontaminerte klær umiddelbart. Legbehandling er nødvendig straks, fordi etseskader som ikke blir behandlet, fører til sår som vanskelig gror.

4.4 Etter øyekontakt

Ved øyekontakt vaskes øynene øyeblikkelig med rennende vann i 10 til 15 minutter mens øyelokkene holdes fra

hverandre, konsulter deretter en øyelege. Beskytt det uskadde øyet. Fjern eventuelle kontaktlinser dersom dette enkelt lar seg gjøre. Fortsett skyllingen.

4.5 Etter svelging

Kontakt umiddelbart et GIFTINFORMASJONSSENTER eller lege. Forhindere oppkast. Skyll munnen grundig med vann. Gi ikke mat eller drikke.

4.6 Selvbeskyttelse for førstehjelper

Førstehjelp: bruk verneutstyr!

4.7 Informasjon til legen.

Symptomer	Ingen data tilgjengelige
Faretype	Ingen data tilgjengelige
Behandling	Ingen data tilgjengelige

5. Forhandsregler ved brann

5.1 Egnede løsemiddel

ABC-pulver Kuldiksyd (CO₂). Tørr sand Sement.

5.2 Av sikkerhetsmessige årsaker uegnet som slukningsmiddel.

Vann Skum.

5.3 Spesielle farer med stoffet eller blandingen

Ved brann kan oppstå: Kuldiksyd (CO₂). Kullmonoksyd.

5.4 Anvsining for brannslukking

IKKE bekjemp brannen når den når eksplosive varer. Ved brann: Ventilasjonsuavhengig åndedrettsvernapparat må brukes.

5.5 Ytterligere opplysninger fra produsent eller avsender

Ikke la slukkevannet havne i avløpet, jorden eller vassdrag. Ikke pust inn eksplosjons- og branngasser.

6. Forholdsregler ved uforutsatt utslipp

6.1 Personlige forholdsregler, verneutstyr og nødprosedyrer

Unngå innånding av gass/damp/sprøytetåke. Unngå kontakt med hud, øyne og klær. Det må sørges for tilstrekkelig lufting.

6.2 Miljøbeskyttelsestiltak

Må ikke komme i kloaksystemet eller i vassdrag. Eksplosjonsfare.

6.3 Metoder og materialer for oppbevaring og rengjøring

Må opptaes med væskebindende material (sand, kiselgur, syrebinder, universalbinder). Samles sammen i egnede, lukkede beholder og avfallshåndteres. Rengjør grundig skitne flasker.

6.4 Ytterligere opplysninger fra produsent eller avsender

Ingen data tilgjengelige

7. Håndtering og lagring

7.1 Beskyttelsestiltak for sikker håndtering

Holdes vekk fra antennelseskilder - Røyking forbudt. Ta forholdsregler mot utladning av statisk elektrisitet. Sikkerhetsforskrifter for sikker behandling Benytt avtrekk (laboratorium). Unngå innånding av gass/røyk/damp/sprøytetåke. Unngå: Gassdannelse

7.2 Betingelser for sikker lagring med henblikk på inkompatibiliteter

Lagertemperatur 15-25°C

Emballasjen skal holdes tett lukket og oppbevares på et kjølig, godt ventilert sted. Oppbevares/lagres isolert fra brennbare materialer.

7.3 Spesifikk sluttbruk

Ingen data tilgjengelige

8. Begrensning og overvåking av ekposisjonen/ Personlig beskyttelsesutrustning

8.1 Kontrollparametre

Reseptur (Betegnelse)	Grenseverditype (opprinnelsesland):	Grenseverdi	Rettsforskrifter
Toluen	TWA (EU)	50 ppm, 192 mg/m ³	2006/15/EC
Toluen	STEL (EU)	100 ppm, 384 mg/m ³	2006/15/EC

8.2 Egnede tekniske styringskontrollmekanismer

Tekniske tiltak og bruken av egnet arbeidsmåte er viktigere enn bruken av personlig verneutstyr. Ved åpen omgang/håndtering må man bruke anretning med lokalt avsug.

8.3 Personlig beskyttelsesutrustning

Bruk egnede verneklær.

8.3.1 Øye-/ansiktsbeskyttelse

Vernebriller med sidebeskyttelse DIN-/EN-standard: DIN EN 166

8.3.2 Hudvern

Ved omgang med kjemiske arbeidsstoffer kan man bare bruke kjemikalievernhandsker med CE-merking inklusivt firesifret kontrollnummer. Anbefalte hanskefabrikat DIN-/EN-standard: DIN EN 374 Ved planlagt gjenbruk av hanskene må man rengjøre dem før man tar dem av, og oppbevare dem ved god ventilasjon.

Ved nylig håndkontakt

Egnet material:	Ingen data tilgjengelige
Tykkelse på hanskematerialet	Ingen data tilgjengelige
Penetrasjonstid (maksimal varighet)	Ingen data tilgjengelige

Ved hyppig hudkontakt

Egnet material:	Ingen data tilgjengelige
Tykkelse på hanskematerialet	Ingen data tilgjengelige
Penetrasjonstid (maksimal varighet)	Ingen data tilgjengelige

8.3.3 Vernebekledning

Det er nødvendig å beskytte seg mot umiddelbar hudkontakt ved å bruke kroppsbeskyttelse i tillegg til vanlige arbeidsklær.

8.3.4 Pustebeskyttelse

Pustemaske er nødvendig ved: Aerosol- eller tåkedannelse.

Egnet åndedrettsvern:	Ingen data tilgjengelige
Egnet material:	Ingen data tilgjengelige

8.4 Ytterligere informasjon

For pausene og ved arbeidsslutt må hendene vaskes. Unngå kontakt med huden og øynene. Det må ikke spises, drikkes eller røykes under bruk. Øyendusjene er klargjort og plasseringen av dem er tydelig avmerket.

9. Fysiske og kjemiske egenskaper

9.1 Informasjon om de grunnleggende fysiske og kjemiske egenskapene

a) Utseende	
Form	flytende
Farge	farveløs
b) Lukt	Ingen data tilgjengelige
c) Lukte grense	Ingen data tilgjengelige

Sikkerhetsrelevante data

d) pH-verdi	Ingen data tilgjengelige
e) Smeltepunkt/Frysepunkt	-95°C
f) Kokepunkt	110,6°C (1013 hPa)
g) Brannpunkt	4°C
h) Fordampingshastighet	Ingen data tilgjengelige

i) Antennelighet (fast, gassformet)	Meget brannfarlig væske og damp.
j) Eksplosjonsgrenser (UEG, OEG)	
Nedre eksplosjonsgrense (Vol%)	1,2
Ovre eksplosjonsgrense (Vol%)	8
k) Damptrykk	36,7 mmHg (30°C)
l) Damptetthet	3,14 (20°C)
m) Relativ densitet	0,867 g/cm ³ (20°C)
n) Oppløslighet	
Oppløslighet i vann (g/l)	0,52 g/l (20°C)
ved/hos °C:	20
Oppløselig (g/l) i	Ingen data tilgjengelige
o) Fordelingskoeffisient: n-octanol/vann	2,73 (20°C)
p) Selvantennelsestemperatur	535°C
q) Spaltningstemperatur	Ingen data tilgjengelige
r) Viskositet	
Kinetisk viskositet	Ingen data tilgjengelige
Dynamisk viskositet	0,6 mPa*s (20°C)
s) Eksplosive egenskaper	ikke anvendelig
t) Oksiderende egenskaper	ikke anvendelig

9.2 Ovrige opplysninger

Bulktetthet	Ingen data tilgjengelige
brytningsindeks	1,4967 (589 nm, 20°C)
Dissosiasjonskonstant	Ingen data tilgjengelige
Overflatespenning	Ingen data tilgjengelige
Henry-Konstante	Ingen data tilgjengelige

10. Stabilitet og reaktivitet

10.1 Reaktivitet

Ingen data tilgjengelige

10.2 Kjemisk stabilitet

Ingen data tilgjengelige

10.3 Mulighet for farlige reaksjoner

Ingen data tilgjengelige

10.4 Omstendigheter som bør unngås.

Ingen data tilgjengelige

10.5 Inkompatible materialer

Ingen data tilgjengelige

10.6 Farlige nedbrydningsprodukter

Ingen data tilgjengelige

10.7 Ytterligere opplysninger fra produsent eller avsender

Ingen data tilgjengelige

11. Opplysninger om toxicologie

11.1 Informasjon om toksikologiske effekter

Akutt virkning

Akut oral toksitet

Virkedosering

LD50: 636 mg/kg

regnear: Rotte.

Eksponeeringstid

bemerkning

kilde

IUCLID

Akutt hudtoksisitet

Virkedosering

LD50: 12124 mg/kg

regnear: Kaniner

Eksponeeringstid

bemerkning

kilde

IUCLID

Akutt inhaleringstoksisitet

Virkedosering

Ingen data tilgjengelige

regnear: Ingen data tilgjengelige

Eksponeeringstid

bemerkning

kilde

Irritasjon og etsevirkning

I første rekke hudirriterende

Eksponeeringstid

regnear:

Resultat

Øyeirritasjon

Eksponeeringstid

regnear:

Resultat

Irritasjon av luftveiene

Eksponeeringstid

regnear:

Resultat

sensibilisering

Ved hudkontakt
Etter innånding

Ikke sensibiliserende.
Ikke sensibiliserende.

Spesifisk målorgan-toksisitet (engangs eksponering)

Kan forårsake dødsighet eller svimmelhet.

Spesifisk målorgan-toksisitet (gjentatt eksponering)

Kan forårsake skade på organer ved langvarig eller gjentatt eksponering.

CMR-virkninger (kreftfremkallende, arvestoff- forandrende og forplantningstruende virkning)

Cancerogenitet

Ingen indikasjon på karsinogene effekter på mennesker.

Mutagenitet i kimcellene/Gentoksisitet

Ingen henvisninger til kimcellemutagenitet hos mennesker finnes.

Reproduksjonstoksisitet

Mistenkes for å kunne gi fosterskader.

Innåndingsfare

Kan være dødelig ved svelging om det kommer ned i luftveiene.

11.2 Andre skadelige følger

Ingen data tilgjengelige

11.3 Ytterligere informasjon

Ingen data tilgjengelige

12. Miljøbetinget informasjon

12.1 Okotoxiske virkninger

Akutt (kortsiktig) fisketoksisitet

LC50: Ingen data tilgjengelige
EC50
regneart:
Eksponeringstid

Kronisk (langsigtig) fisketoksisitet

LC50: Ingen data tilgjengelige

EC50

regneart:

Eksponeringstid

Akutt (kortsiktig) daphnitoksisitet

LC50: Ingen data tilgjengelige

EC50

regneart:

Eksponeringstid

Kronisk (langsigtig) daphnitoksisitet

LC50: Ingen data tilgjengelige

EC50

regneart:

Eksponeringstid

Akutt (kortsiktig) algetoksisitet

LC50: Ingen data tilgjengelige

EC50

regneart:

Eksponeringstid

Kronisk (langsigtig) algetoksisitet

LC50: Ingen data tilgjengelige

EC50

regneart:

Eksponeringstid

12.2 Opplysning om eliminering

Ingen data tilgjengelige

12.3 Bioakkumulasjonspotensial

o) Fordelingskoeffisient: n-octanol/vann 2,73 (20°C)

12.4 Mobilitet i grunnen

Ingen data tilgjengelige

12.5 Resultat av undersøkelsen av PBT-egenskapene

Ingen data tilgjengelige

12.6 Andre skadelige følger

Ingen data tilgjengelige

13. Opplysninger om Destruksjon.

13.1 Avfallshåndteringsmetoder

Avfallshåndtering / Produkt

Dispose of waste according to applicable local, state, and federal regulations.

Avfallnøkkel produkt

07 01 04 (other organic solvents, washing liquids and mother liquors)

Avfallshåndtering / Emballasje

Fjern i samsvar med lokale myndigheters bestemmelser.

13.2 Ytterligere informasjon

Ingen data tilgjengelige

14. Opplysninger om transport

14.1 Vejtransport (ADR/RID)

UN-Nummer	1294
Offisiell benevnelse for transporten	Ingen data tilgjengelige
Klasse(r)	3
Klassifiseringskode:	F1
Innpakningsgruppe	II
Fareseddel	3

14.2 Sjøfart (IMDG)

UN-Nummer	1294
Offisiell benevnelse for transporten	Ingen data tilgjengelige
Klasse(r)	3
Klassifiseringskode:	F1
Innpakningsgruppe	II
Marine pollutant	
Delingsgruppe	

14.3 Luftfart (ICAO-TI / IATA-DGR)

UN-Nummer	1294
Offisiell benevnelse for transporten	Ingen data tilgjengelige
Klasse(r)	3
Klassifiseringskode:	F1
Innpakningsgruppe	II

14.4 Ytterligere informasjon

Ingen data tilgjengelige

15. Rettsforskrifter

15.1 Forskrifter om sikkerhet, helse og miljø/spesifikke regler for stoffet eller blandingen

Vannfare-klasse (WGK)

2

15.2 Stoffsikkerhetsbedømmelse

Ingen data tilgjengelige

16. Øvrige opplysninger

16.1 Ordlyd i R-, H- og EUH-setningene (Nummer og fulltekst)

R11	Meget brannfarlig.
R38	Irriterer huden.
R48/20	Farlig: Alvorlige helsefare ved lengre tids påvirkning ved innånding.
R63	Mulig fare for fosterskader.
R65	Farlig: kan forårsake lungeskade ved svelging.
R67	Damp kan forårsake døsighet og svimmelhet.

H225	Meget brannfarlig væske og damp.
H361d	Mistenkes for å kunne gi fosterskader.
H304	Kan være dødelig ved svelging om det kommer ned i luftveiene.
H373	Kan forårsake skade på organer ved langvarig eller gjentatt eksponering.
H315	Irriterer huden.
H336	Kan forårsake døsighet eller svimmelhet.

16.2 Ytterligere opplysninger fra produsent eller avsender

Endringsindikasjoner

generell oppdatering

Opplysningene i dette sikkerhetsdatabladet beskriver våre kunnskaper ved trykking etter vår beste viten. Denne informasjonen skulle gi Dem holdepunkter for sikker omgang ved lagring, bearbeidelse, transport og fjerning av det produktet som dette sikkerhetsdatabladet nevner. Opplysningene er ikke overførbare til andre produkter. Hvis produktet blir blandet eller bearbeidet med andre materialer, er opplysningene i dette databladet ikke uten videre overførbare til det da ferdige nye materialet.