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ถังกวนแบบแบตช์โดยใช้ใบกวนแบบเฮียร์มิกเซอร์

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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

DISSOLUTION OF SULPHUR POWDER IN CUTTING OIL WITHIN BATCH  
MIXING TANK USING SHEAR MIXER

MR.KITTIYAKORN SRISAWAM

สถาบันวิทยบริการ  
จุฬาลงกรณ์มหาวิทยาลัย

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Thesis Co-advisor	Geravat Sinthuvanichsaid, M.Sc.

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กิตติยากรณ์ ศรีสุวรรณ : การละลายของผงกำมะถันในน้ำมันที่ใช้ในงานตัดกลึงในถังกวนแบบ  
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ในงานวิจัยนี้ได้ทำการศึกษาอิทธิพลของภาวะการปฏิบัติการที่มีต่อการละลายของผงกำมะถัน  
 ในน้ำมันที่ใช้ในงานตัดกลึง โดยทำการหาเวลาที่จำเป็นในการผสม โดยใช้สารกำมะถันเป็นสารติดตาม  
 ใช้เครื่องมือ Ultra Violet Spectrophotometer ( UV Spectrophotometer ) และใช้ถังกวนผสมขนาด  
 มาตรฐาน ชนิดของใบพัดกวนเป็นแบบ High speed shear mixer โดยตัวแปรที่สำคัญที่ใช้ศึกษาคือ  
 ความเร็วรอบของใบพัดกวนในช่วง 300 ถึง 500 รอบต่อนาที ความหนืดของน้ำมันหล่อลื่นพื้นฐานที่  
 อุณหภูมิ 100 องศาซึ่งมีค่าอยู่ในช่วง 4.7, 11.2 และ 32.0 เซนติสโตค ช่วงอุณหภูมิที่ใช้ในการศึกษา  
 80, 100 และ 120 องศาเซลเซียส

จากผลการวิจัยที่ได้พบว่าเวลาที่จำเป็นในการผสมจะเป็นสัดส่วนผกผันกับจำนวนรอบและ  
 อุณหภูมิรวมถึงคุณสมบัติความหนืดของน้ำมันหล่อลื่นและจุดหลอมเหลวของกำมะถันผงด้วยนอกจาก  
 นี้ยังพบว่าที่ความเร็ว 400 รอบจะใช้เวลาของการผสมที่เหมาะสมที่สุดเนื่องจากจะสามารถประหยัดเวลา  
 และพลังงานในการกวนได้ทั้งนี้สามารถแสดงผลของการหาเวลาที่จำเป็นในการผสมในรูปของการ  
 วิเคราะห์เชิงมิติที่แต่ละอุณหภูมิและชนิดความหนืดของน้ำมันแล้วรวมทั้งสามารถนำเสนอสมการในการ  
 ผสมในกลุ่มของการวิเคราะห์เชิงมิติ ได้ดังนี้คือ

$$Sh_T = r Re_a^{0.378} Sc^{0.454}$$

โดยที่  $0.465 \times 10^3 < Re_a < 22.371 \times 10^3$  ,  $1.711 \times 10^{11} < Sc < 374.155 \times 10^{11}$

ภาควิชา.....วิศวกรรมเคมี..... ลายมือชื่อนิสิต.....  
 สาขาวิชา.....วิศวกรรมเคมี..... ลายมือชื่ออาจารย์ที่ปรึกษา.....  
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# # 4371518921 : MAJOR CHEMICAL ENGINEERING

KEY WORD: SULPHUR DISSOLUTION / LUBRICATING OILS / MIXING

KITTIYAKORN SRISAWAN: DISSOLUTION OF SULPHUR POWDER IN CUTTING OIL WITHIN BATCH MIXING TANK USING SHEAR MIXER. THESIS ADVISOR: ASSOCIATE PROFESSOR TAWATCHAI CHARINPANITKUL, D.Eng., THESIS COADVISOR : GERAVAT SINTHUVANICHSAID, M.Sc. 148 pp. ISBN 974-03-1082-6.

The aim of this work is to study influence of operating condition on dissolution of sulphur powder in cutting oil. The mixing time required for a standard configuration tank was measured from rate of sulphur dissolution by using UV Spectrophotometer. Mixing was carried out in a flat bottom cylindrical vessel with a high speed shear mixer. Rotation speeds of the impeller were varied between 300 to 500 rpm and viscosity of lubricating oil @100 °C were 4.7, 11.2 and 32.0 cSt. (approximately). Temperature under investigation were between 80 to 120 °C

The results showed that the required mixing time was inversely proportional to rotational speed and depended on temperature, viscosity of lubricating oil and melting point of sulphur powder. The optimum mixing condition was impeller rotational speed of 400 rpm at which the shortest mixing time and the lowest mixing energy were employed. The required mixing time can be correlated with various temperature and viscosity each type of base oil.

By employing the dimensional analysis, the empirical equation for the mass transfer coefficient was

$$Sh_T = r Re_a^{0.378} Sc^{0.454}$$

whereas  $0.465 \times 10^3 < Re_a < 22.371 \times 10^3$ ,  $1.711 \times 10^{11} < Sc < 374.155 \times 10^{11}$

Department.....Chemical Engineering..... Student's signature.....

Field of study...Chemical Engineering..... Advisor's signature.....

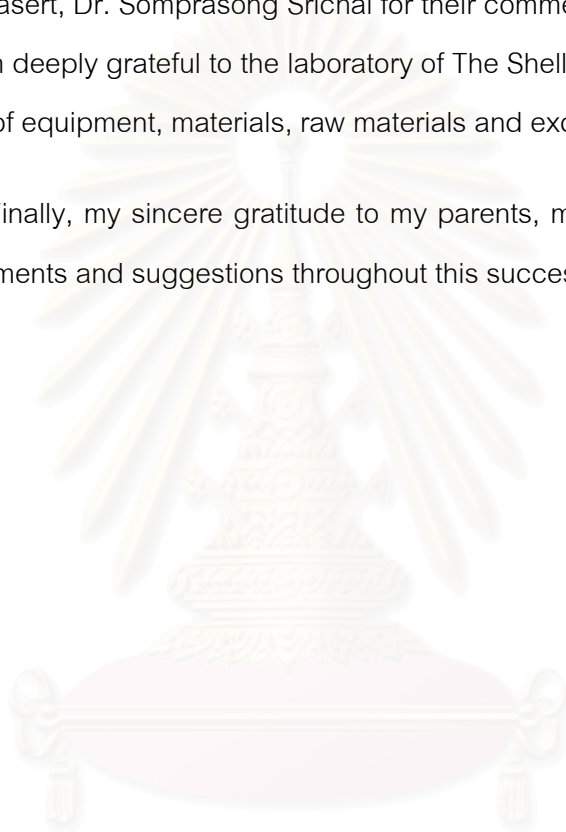
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## NOMENCLATURE

$A$	=Contract surface area of solid and liquid	$\text{cm}^2$
$C$	=Concentration at time $t$	$\text{g/cm}^3$
$C_s$	=Saturated concentration	$\text{g/cm}^3$
$D_i$	=Impeller diameter	m
$D_p$	=Diameter of particle	cm
$H_i$	=Impeller height from tank bottom	m
$H_l$	=Depth of liquid in tank	m
$K$	=Dissolution rate coefficient	cm/sec
$L$	=Impeller blade length	m
$n$	=Total of counts, Number of particle $\frac{6W}{d_p^3 \pi \rho_s}$	-
$N$	=Rotation speed of impeller	rpm
$P$	=Power consumption	
$Re$	=Reynolds number, $\frac{D_i^2 w \rho}{\mu}$	-
$p$	Variable exponent	-
$q$	Variable exponent	-
$Re_T$	=Reynolds number referred to tank	-
$Re_a$	=Reynolds number referred to agitator	-
$Sc$	=Schmidt number, $\frac{\mu}{\rho D_v}$	-
$Sh$	=Sherwood number, $\frac{KT}{D_v}$	-
$S_C$	= The perimeter of circle having the same area as the projected area of the particle	-
$S_A$	=The actual particle perimeter	-
$t$	=Time	sec
$t_m$	=Mixing time	min
$T$	=Diameter of tank	cm
$V$	=Volume of liquid	$\text{cm}^3$

$W$	=Impeller blade width	cm
$W$	=Mass of solid particles, The mass remaining at time $t$	g
$W_0$	= Initial mass of solid	g
$X$	=Number of counts	-
$\bar{X}$	=Mean value counts	-
$Z$	=liquid depth	cm
$\alpha_w$	=Shape factor relating the surface area with mass	-
$\rho$	=Density	$\text{g/cm}^3$
$\psi_c$	=Circularity	-
$\tau$	=Dimensionless mixing time, $t_m ND_i^2 S/V$	-
$\nu$	=Kinematics viscosity	$\text{cm}^2/\text{s}$
$u$	=Velocity	m/s
$\mu$	=Dynamic viscosity	Pa-s
$\sigma$	=Standard deviation	-



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# CHAPTER I

## INTRODUCTION

### 1.1 General

Mixing is one of the most common operations in the chemical and allied industries. However, in spite of long history of the mixing process, theory and principles are often ignored from practice, and the concepts of mixer design have not yet been systematized. A combination of physical motion of the fluid and diffusion causes mixing or transport of heat and mass in the fluid, and therefore has various effects on chemical processes. In spite of this, mixing has proved intractable to a rigid theoretical analysis. Thus in comparison with other chemical engineering operation, mixing is still regarded as state-of-the-art technology.

Mixing action is not only to produce uniform distribution of components but also to provide movement or transfer of materials from surfaces of one phase to the other. Agitated vessels are often used because they are effective in suspending solid particles, which can ensure that all the surface area available is utilized and leads to good dissolution rate.

There is very limited work reported in the literature where either the theoretical or the experimental aspects of solid-liquid dissolution in agitated vessel. There are various researches working on this problem, especially about requirement of mixing time in blending of different viscosity and temperature, Hixson and Baum<sup>(8)</sup>, Nagata and Yamagushi<sup>(19)</sup>, Barker and Treybal<sup>(2)</sup>, Johnson and Chen-Jung<sup>(12)</sup>, Hariot<sup>(5)</sup>, Kevin Hool<sup>(14)</sup>, Mashelkar and Chavan<sup>(16)</sup> are representatives of there investigating for agitated vessels. Hixson and Baum studied the rate of mass transfer from solid particle in liquid agitated by a four-pitched-blade paddle and a marine propeller in unbaffled vessels of various diameters. Barker and Treybal studied the rate of solution of solid particles suspended in water baffled vessels agitated by a six-flat-blade turbine. However, a detailed analysis indicated that there exists a wide divergence of theoretical and experimental results.



## 1.2 Lubricating oils

In lubricating oil blending process (Figure 1.1) mixing time is very important for obtaining homogeneous lubricating oil mixture. The problem in this process is that some lubricant additive is in powder form and difficult to dissolve in the oil, This rate of homogeneity was one factor for analyze blending time. So, the rate of homogeneity has affected on performance and power consumption in the production of lubricating oil product.

In general, lubricating oils consist of basic oil and additives as described below:-

### 1.2.1 Base oils

Three major chemical obtained from crude oil are generally employed as the base component for lubricating oil.

#### a) Naphthenic Oils

They are saturated hydrocarbons but made up of Methylene group ( $\text{CH}_2$ ) arranged in ring formation. Those with one such closed carbon ring are known as monocyclic bodies and when more than one is present in the molecule, it is called as Polycyclic. Paraffinic side chains may be attached to carbon atom of ring and proportion of carbon atom in side chain to those ring have affects to their properties.

#### b) Paraffinic Oils

These contain saturated, straight chain or branched hydrocarbons. The branched paraffins, are found in large quantities in lubricating oil fraction from paraffinic crudes. The straight chain paraffins with high molecular weight raise the pour point of oils therefore it should be removed by dewaxing.

#### c) Aromatic

Hydrocarbons of this type have closed carbon ring of semi-unsaturated character. Side chains can be attached to any carbon atom, ring structures are joined


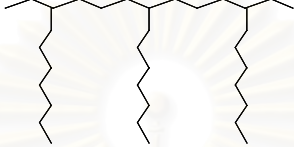
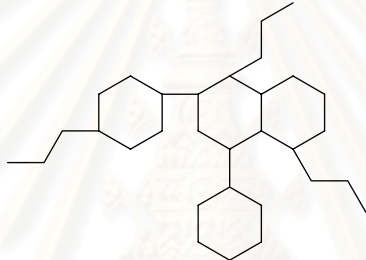
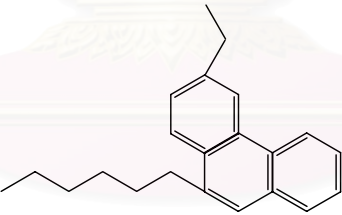
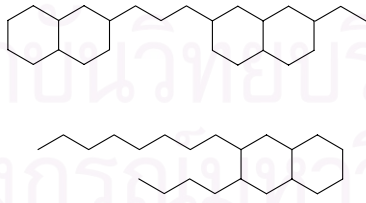
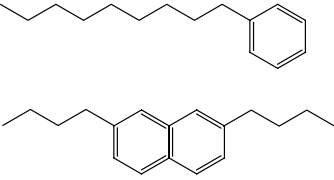
together and an enormous number of compounds exist. The structure is more easily attached than either paraffins or naphthene groups which, in service, lead to formation of resinous, asphaltic and corrosive by products. Their density is quite high and viscosity indexes low. Aniline point was the minimum equilibrium solution temperature for equal volumes of aniline and sample. Their aniline point is very low and their solvent power is high, but these are of little interest due to the above indicated flaws. Lastly, their low interfacial tension makes it easier to form emulsions with water.

The high viscosity index associated with paraffinic oils is generally preferred for premium quality industrial gear oil applications because of the tolerance for wide temperature range. Maintenance of a continuous lubricant film is also important to the load carrying capabilities of lubricants. Very low viscosity could lead to failures in field service. The desired viscosity can usually be obtained by combining a heavy bright stock with light neutral oil in the proper ratios. The same additives may show a different response in different base stocks.

Table 1.1 gives the relations between properties and main type of hydrocarbon structures which are present in current lubricating oils; the under lined structures correspond to research properties.

Synthesis lubricants are receiving attention where temperature extremes demand a high viscosity index and thermal stability. Principles classes of synthetic lubricants crude Polyolefins, Diesters, Polyglycols, Halogenated hydrocarbons, Phosphate ester and Silicone type polymers. The polyolefins and esters appear to be receiving the greatest attention for industrial gear applications. The viscosity of the oil

Table 1.1 Relations between properties and structures of lubricating oil

Hydrocarbon Types	Example Structure	Main Properties
Straight Chain Paraffin.		Viscosity varies little with temperature. Good oxidation resistance. High pour point.
Branched Chain Paraffin.		Viscosity varies little with temperature. Good oxidation resistance. May have low pour point.
Naphtha Rings with short Paraffin Side Chain.		Good oxidation resistance. Low pour point. Viscosity varies greatly with temperature.
Aromatic Rings with short Paraffin Side Chain.		Pour point varies according to structure. Good thermal stability. Viscosity varies greatly with temperature. Easily oxidizes.
Naphtha Ring with long Paraffin Side Chain.		Viscosity varies little with temperature. Good oxidation resistance. May have low pour point.
Aromatic Ring with long Paraffin Side Chain.		Viscosity varies little with temperature. May have good oxidation resistance if cycles are not numerous.

must allow the formulation of an adequate protective film under the existing conditions of load, speed and temperature. Low viscosity gear oil are usually chosen for high speed operations where contact periods between teeth or meshing gears are minimal and where loading is light. Heavier oils are suitable to low speed/high load operations where contact periods are longer.

### 1.2.2 Additives

Modern machinery places high demands on lubricants. In order to meet these demands on lubricants contain small quantities of additives are employed to enhance the properties of base oils.

There are many different types of additives. Some of which may fulfill several different functions. The combinations of additives used in a lubricant depend on the use to which the lubricant is to be put.

It is convenient to divide additives into three categories:

- Additives for modifying the performance of the lubricant. These include viscosity index improvers and pour point dispersants.

- Additives for protecting the lubricant. These include anti-oxidants and anti-foaming agents.

- Additives for protecting the lubricated surface. These include corrosion inhibitors, rust inhibitors, detergents, dispersants and anti-wear additives.

From various properties and functions of additives, sulphur powder is an additive that we use for corrosion inhibitor in metal cutting process. Sulphur after dissolve is linked organically with hydrocarbons in the lubricating oil, giving compounds of very similar molecular weight. Hence a very small percentage. In this blending process sulphur powder is difficult to dissolve in lubricating oil and long blending time.

### 1.3 Objective for this study

The main objective of this study is to obtain understanding of sulphur particles dissolution in mineral oil then the optimal mixing time will be explored for predicting the solid dissolution rate of sulphur in mineral oil will also be investigated. The study will be conducted in standard configuration tank using high speed shear mixer impeller.

### 1.4 Scope of work

1. The mixing systems used in this study are standard configuration flat bottom cylinder tanks with high speed shear impeller is set at 1/3 of liquid depth from the vessel bottom.

2. Particle size of sulphur powder used in this study has size in range 37-44 micron (80-200 Mesh)

3. The important parameters for solid dissolution, such as impeller speed, temperature, viscosity will be studied and correlated in terms of dimensionless number of Sherwood, Reynolds, and Schmidt. The various mixing conditions to be investigated are

- Impeller speed in a range of 300 – 500 rpm.
- Liquid temperature in a range of 80 – 120 °C
- Viscosity of Parafinic base oil are 4.7, 11.2 and 32.0 cSt. (approximately)

4. The physicochemical properties of sulphur in mineral oil will be studied and include

- Dissolution rate coefficient
- Solubility

5. A comparison with correlation of other investigation will be conducted

## CHAPTER II

### LITERATURE REVIEW

One of the important factors influencing the rates of mass transfer is the molecular diffusion coefficient of the solute in the solvent. The rate of solid dissolution is controlled by the relative velocity of solid to liquid and the rate of renewal of liquid layer which depend on the intensity of turbulence around the solid particles.

#### 2.1 Hixson and Crowell <sup>(7)</sup>

Hixson studied the suspension of sand in water using an unbaffled vessel of 18 cm in diameter agitated by 4-pitched-blade turbine. They proposed a mixing index when the liquid is in excess. When the mixing index reaches 90%, the effect of an increase in agitator speed reaches a maximum and the mixing index does not change further. The mixing index is larger when the liquid viscosity is increased at an equal agitator speed.

Rate of dissolution of solid particle in liquid can be expressed as follows:

$$-\frac{dW}{Vdt} = K \frac{A}{V} (C_s - C) \quad (2.1)$$

whereas	$W$	:	mass of solid particles [g]
	$V$	:	volume of liquid [cm <sup>3</sup> ]
	$K$	:	dissolution rate coefficient [cm/sec]
	$A$	:	contact surface area of solid and liquid [cm <sup>2</sup> ]
	$C_s$	:	saturated concentration [g/cm <sup>3</sup> ]
	$C$	:	concentration at time $t$ [g/cm <sup>3</sup> ]

By integrating Eq. (2.1) Hixson and Crowell could obtain the following relations:

$$Kt = \left( \frac{V}{\alpha_w m^{2/3}} \right) \left\{ \frac{3^{0.5} \tan^{-1} (2\sqrt{3} m^{1/3} (W_0^{1/3} - W^{1/3}))}{3m^{2/3} + (2W_0^{1/3} - m^{1/3})(2W^{1/3} - m^{1/3})} + 1.1513 \log \frac{(m^{1/3} + W_0^{1/3})^2}{(m^{1/3} + W^{1/3})^2} \right\} \\ \times \frac{\{m^{2/3} - m^{1/3}W^{1/3} + W^{2/3}\}}{\{m^{2/3} - m^{1/3}W_0^{1/3} + W_0^{2/3}\}} \quad (2.2)$$

$$\begin{aligned}
 A &= \alpha_w W^{2/3} & C_s &= \frac{W_s}{V} \\
 C &= \frac{(W_0 - W)}{V} & m &= W_s - W_0
 \end{aligned}$$

whereas

$\alpha_w$	=	Shape factor relating the surface area with mass
$W_s$	=	Mass needed to saturate the liquid [g]
$W_0$	=	Total mass charged [g]
$K$	=	Dissolution rate coefficient [cm/sec]
$t$	=	Time [sec]
$W$	=	Mass of solid particles [g]
$V$	=	Volume of liquid [cm <sup>3</sup> ]
$A$	=	Contact surface area of solid and liquid [cm <sup>2</sup> ]
$C_s$	=	Saturated concentration [g/cm <sup>3</sup> ]
$C$	=	Concentration at time t [g/cm <sup>3</sup> ]

$\alpha_w$  is a shape factor relating the surface area with mass,  $W_s$  which is needed for the saturation condition  $W_0$  is the total mass charged into the oil. Eq.(2.2) shows the relation between the time  $t$  and the cube root of the weight of solid at that time therefore it is called, "cube root law". In the range where the dissolution rate coefficient  $K$  and shape factor  $\alpha_w$  are assumed constant, the rate of dissolution is obtained experimentally at any time and the value  $K$  is determined when the value of  $\alpha_w$  or  $A$  is given.

## 2.2 Wilhelm et al. <sup>(7)</sup>

By using the follow relations and notations,

$$W_d = W_0 - W, \quad X = \frac{W_d}{W_s}, \quad Y = \frac{W_0}{W_s}$$

$$A = \alpha_v n^{1/3} \frac{W^{2/3}}{\rho_s^{2/3}}, \quad V = (1 + \alpha X)V_0, \quad V_s = (1 + \alpha)V_0$$

Eq.(2.1) is transformed to an integral form;

$$Z = \int_0^x \frac{dX}{(Y - X)^{2/3} \left( \frac{1}{1 + \alpha} \right) - \left( \frac{X}{1 + \alpha X} \right)} = \frac{KA_s t}{V} \quad (2.3)$$

By graphical integration, Eq.(2.3) is calculated and is used to evaluate  $K$  when the relation between  $Z$  and  $X$  is drawn on a graph using  $Y$  as a parameter. Wilhelm et al. showed a diagram for the system in the case of the dissolution of sodium chloride in water. From the measurement of  $W_d$  at time  $t$ , the dissolution rate coefficient  $K$  volumetric change can be neglected. Wilhelm et al. considered the change of liquid volume is proportional to the ratio of saturation.

### 2.3 Hixson and Baum's Correlation <sup>(8)</sup>

Hixson and Baum studied the rate of mass transfer from solid particle of 2.5 cm in diameter in a liquid which is agitated by a turbine or by a marine propeller equipped in unbaffled vessels of various diameters. They obtained a dimensionless equation as follows,

$$\frac{KT}{D_v} = \frac{T}{Z} = r \left[ \frac{T^2 N \rho_l}{\mu} \right]^p \left[ \frac{\mu}{\rho_l D_v} \right]^q \quad (2.4)$$

$$Sh_T = r Re^p Sc^q \quad (2.5)$$

The experimental data are correlated as shown in Figure 2.1(a) and 2.1(b) and the following equations could be drawn from the figures:

For Turbine

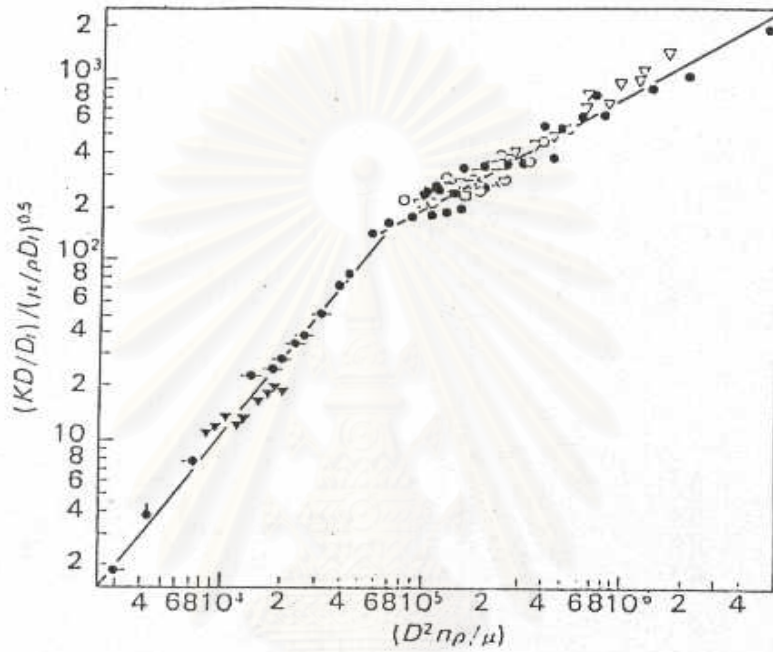
$$Re < 6.7 \times 10^4 \quad ; \quad Sh_T = 2.7 \times 10^{-5} Re^{1.4} Sc^{0.5} \quad (2.6)$$

$$Re > 6.7 \times 10^4 \quad ; \quad Sh_T = 0.16 Re^{0.63} Sc^{0.5} \quad (2.7)$$



For Marine Propeller

$$3,300 < Re < 330,000 ; \quad Sh_T = 3.5 \times 10^{-4} Re^{1.0} Sc^{0.5} \quad (2.8)$$



- : Benzoic acid-water,                      ○ : Benzoic acid-benzene,
- : Benzoic acid-sperm oil,                ● : Benzoic acid cotton seed oil,
- : Benzoic acid-rape seed oil,           ● : Benzoic acid-40% sucrose,
- ▼ : Benzoic acid-ethylene glycol,        ▽ : Barium chloride-water,
- : Rock salt-water,                        ✕ : Naphthalene-methanol,

Figure 2.1(a) : Correlation of mass transfer coefficients.<sup>(12)</sup>

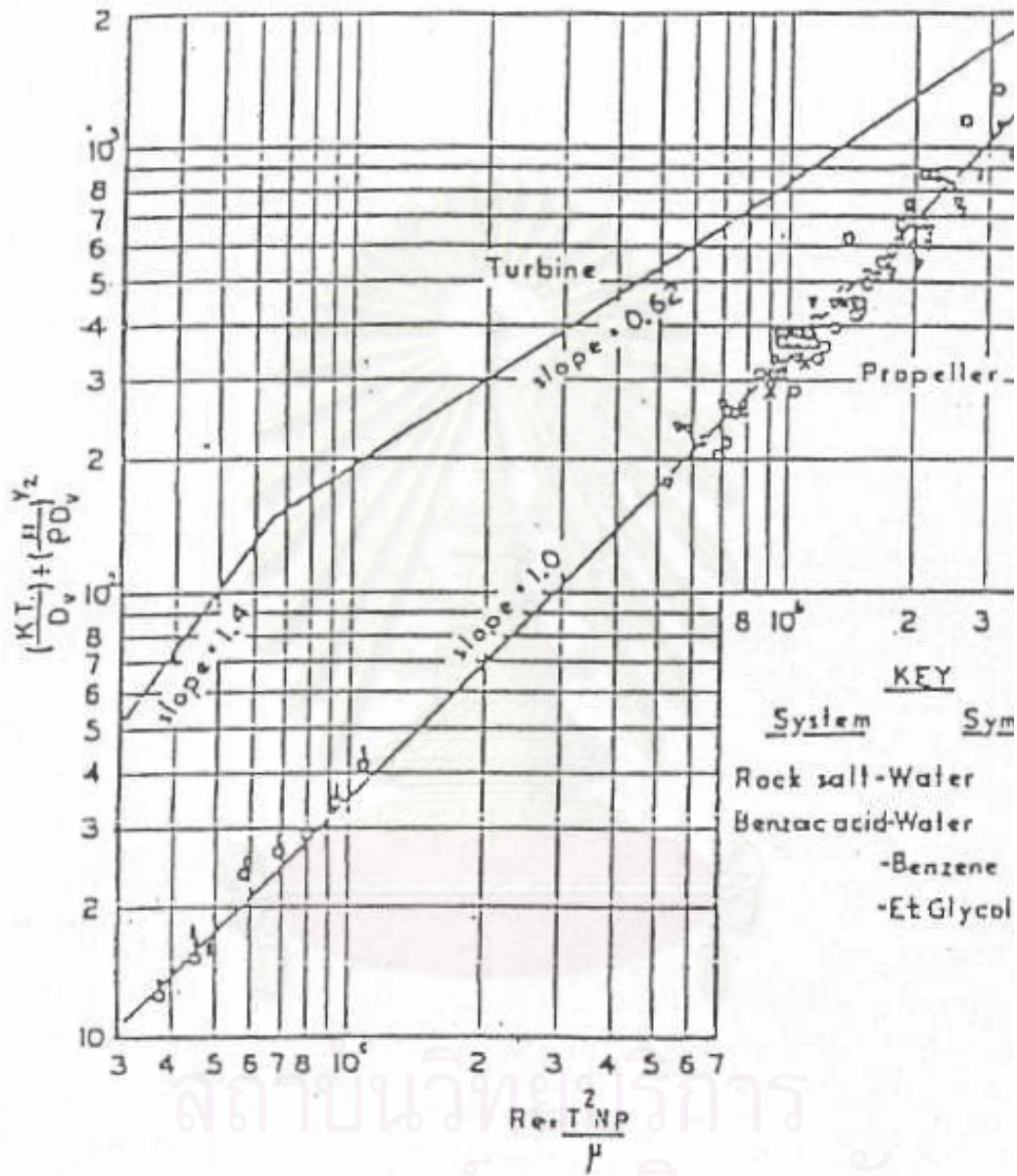


Figure 2.1(b) : Graphical determination of constant exponents of general equation for mass transfer.<sup>(12)</sup>

## 2.4 Nagata's Correlation <sup>(19)</sup>

A more general correlation for geometrically similar, unbaffled vessels, with spherical and granular solids is

$$\frac{kT}{D_v} = 3.60 \times 10^{12} \left( \frac{T^2 N \rho_l}{\mu} \right)^p (\rho_l D_v)^q \left( \frac{D_v^2}{T^3} \right)^{0.627} \left( \frac{d_p}{T} \right)^{3.08} \left( \frac{\Delta \rho}{\rho_l} \right)^{-2.82} \quad (2.9)$$

whereas:

$$p = 0.0802 \left( \frac{T^3 g \rho_l^2}{\mu^2} \right)^{0.0772} \left[ \log \left\{ \left( \frac{\Delta \rho}{\rho_l} \right) + 0.043 \right\} + 1.35 \right]^{\frac{-13.52 d_p}{T}}$$

$$q = 14.4 \frac{d_p}{T} + 1.84 \left( \frac{\Delta \rho}{\rho_l} \right) 0.116$$

As shown in the above equation, the exponent's  $p$  increase with the increase in density difference between liquid and solid and with the decrease in particle size,  $d_p$ . The effect of shape is not significant. It is rather difficult to determine the coefficient and exponents so Nagata derived the following equation from experimental data using combinations of solids and liquids. This equation is applicable only in the range,  $\left( \frac{d_p}{D} \right)^2 > 4.10^{-5}$  and is not applicable for smaller particles.

For influencing factors on  $p$  <sup>(12)</sup>. When dissolution velocity  $\left( \frac{-dW}{Vdt} \right)$  accompanied by an increase in agitator speed ( $N$ ) has two distinct blending points,  $N_f$  and  $N_a$  as shown in Fig 2.2  $N_a$  is a speed at which air is sucked into the liquid and  $N_f$  is an agitator speed.

In Fig.2.2(a)  $\left( \frac{KD}{D_f} \right)$  vs.  $\left( \frac{D^2 n \rho}{\mu} \right)$  are used instead of  $\left( \frac{-dW}{Vdt} \right)$  vs.  $N$  for generalization, but the slope of the curves is equal in both diagrams. Let  $p_1$  denote the slope in the range of agitator speed less than  $N_f$ ,  $p_2$  denotes that between  $N_f$  and  $N_a$ , and  $p_3$  that in the range of speed larger than  $N_a$ . Usually they are in the order,  $p_1 > p_2 > p_3$ , and

these value vary with  $(\rho-\rho_l)$  and  $d_p$ . The Nagata correlated  $p$  with various factors; density difference, particle diameter and shape factor in the case of slight amount of solid dispersed (2-5 g of solid particles in 800 cc liquid).

$$p = f \left\{ \left( \frac{\rho_s - \rho_l}{\rho_l} \right), \left( \frac{d_p}{D} \right), \phi_s \right\} \quad (2.10)$$

A large number of reports have been presented on the solid-liquid mass transfer coefficient of suspended solid particle in agitated liquids. This is illustrated in Table 2.1

As can be seen from Table 2.1 there is a wide divergence of the experimental results and correlations. This is because different approaches have been used to predict mass transfer from suspended solids, for instance dimensional analysis, the slip velocity theory proposed by Harriott<sup>(5)</sup>, the non-steady state model according to Higbie's penetration theory<sup>(22)</sup> and the Kolmogoroff's theory of local isotropic turbulence<sup>(13)</sup>

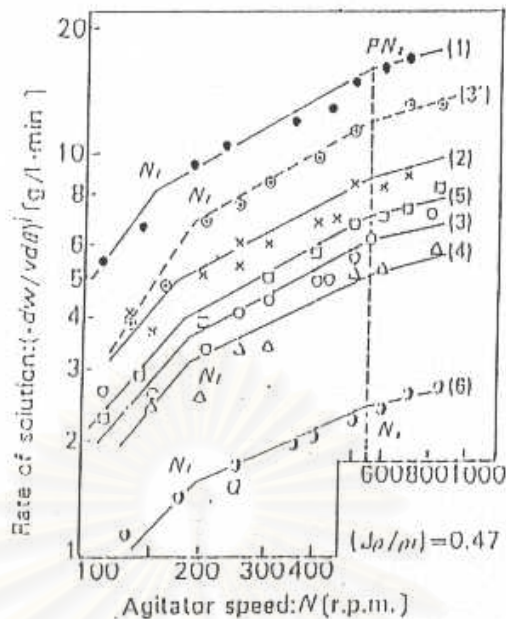


Fig 2.2(a) : Rate of solution vs. agitator speed ( $H_3BO_3$ -water system)  
 Solid dissolved : (1)-(6) 2.000g (3) 4.00 g; sphere : (1) 60-100#, (2) 45-60#, (3), (3') 28-45# ( $d_p = 0.519$  mm), (4) 16-28# ( $d_p = 0.737$  mm); rhombic crystal : (5) 28-46# ( $d = 0.557$  mm), (6) 16-28# ( $d_p = 0.858$  mm), Temp.:  $25^\circ C$  constant<sup>(6)</sup>

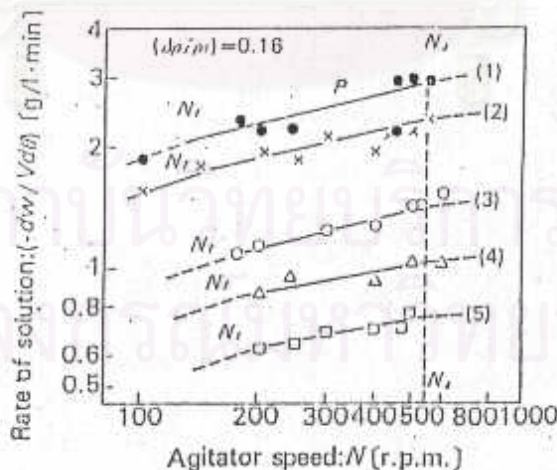


Fig 2.2(b) : Correlation of  $(-dw/Vd\theta)$  vs.  $N$  for the crotonic acid-water System. solid used: 0.500g; size: (1) 28-45#, (2), (3) 16-28#, (4), (5) 10-16#; shape : cubical monoclinic (3), (5), flat monoclinic (1), (2), (4); temp. :  $25^\circ C$  <sup>(6)</sup>

Table 2.1: Dimensionless-Type Correlation Form Various References <sup>(11)</sup>

System	Type of Impellers	Baffled	Form of solid particles	Schmidt number	Reynolds number	Correlation	Ref
Water-benzoic acid Water-barium chlorine Water-Naphthalene	Marine Propeller Turbine incurved with 45°	No	Tablet	486-2.56x10 <sup>6</sup>	2.94x10 <sup>3</sup> -5.3x10 <sup>6</sup> <6.7x10 <sup>4</sup> >6.7x10 <sup>4</sup>	Sh <sub>T</sub> =3.5x10 <sup>-4</sup> Re <sub>T</sub> Sc <sup>0.50</sup> Sh <sub>T</sub> =2.7x10 <sup>-5</sup> Re <sub>T</sub> <sup>1.40</sup> Sc <sup>0.50</sup> Sh <sub>T</sub> =0.16 Re <sub>T</sub> <sup>0.62</sup> Sc <sup>0.50</sup>	8
Water-benzoic acid Methylate-benzoic acid Benzene-benzoic acid Water-Sodium chloride Water-barium chloride	Turbine incurved with 45° Marine Propeller Turbine with 4-blade	No	Sphere		2.5x10 <sup>3</sup> -5.0x10 <sup>6</sup>	Sh <sub>T</sub> =A(Re <sub>p</sub> ) <sup>p</sup> (Sc) <sup>0.50</sup> (d <sub>p</sub> /T) <sup>g</sup> 0.2<p<0.67 -0.8<g<-0.32	19
Water-benzoic acid Water-boric acid Sucrose Sol benzoic acid Sucrose Sol boric acid	Standard Turbine	Yes	Granular	735-55000	10 <sup>4</sup> – 10 <sup>6</sup>	Sh <sub>T</sub> =0.052 Re <sub>a</sub> <sup>0.833</sup> Sc <sup>0.50</sup>	2
Water-benzoic acid	Turbine with 6-blade Turbine incurved with 45° Marine Propeller	Yes	Tablet		24,000-120,000 40,000-120,000 40,000-180,000	Sh <sub>T</sub> =3.30 Re <sub>a</sub> <sup>0.55</sup> Sc <sup>0.30</sup> Sh <sub>T</sub> =0.66 Re <sub>a</sub> <sup>0.667</sup> Sc <sup>0.30</sup>	13
Water-O-phenol	Agitator with 2-blade	No	Sheer	1240	43,000-125,000	Sh <sub>T</sub> =0.943 Re <sub>a</sub> <sup>0.80</sup> Sc <sup>0.33</sup>	17
Water benzoic acid	Turbine with 4-blade	Yes	Ball	-	>50,000	Sh <sub>T</sub> =8.48 Re <sub>a</sub> <sup>0.397</sup> Sc <sup>0.333</sup> Sh <sub>T</sub> =0.0267(2+1.1Re <sub>p</sub> <sup>0.5</sup> Sc <sup>0.33</sup> )N <sup>0.63</sup>	23
Water benzoic acid	Standard Turbine	Yes	Sphere	307-1990	1x10 <sup>4</sup> -3x10 <sup>4</sup>	Sh <sub>p</sub> =0.0446 Re <sub>p</sub> <sup>0.283</sup> Ga <sup>0.173</sup> T <sup>-0.011</sup> (T/d <sub>p</sub> ) <sup>0.019</sup> Sc <sup>0.461</sup>	4

## CHAPTER III

### THEORY

#### 3.1 Solid-Liquid Mass Transfer<sup>(11)</sup>

In general on study solid-liquid mass transfer says attention on the effect of mixing on the surface area of the solids

Three major correlations methods have been tried by various investigators to find out the relationship among  $k$  and other equation variables. The first method, using dimensional analysis, usually takes the form :

$$\frac{kT}{D_v} = r(\text{Re}_T)^p (\text{Sc})^q \quad (3.1)$$

or

$$\frac{kT}{D_v} = r(\text{Re}_a)^p (\text{Sc})^q \quad (3.2)$$

whereas  $\text{Re}_T$  = Reynolds number referred to tank  
 $\text{Sc}$  = Schmidt number  
 $\text{Re}_a$  = Reynolds number referred to agitator

Several investigators have tried using this equation in their correlations, and have found that the constant  $r$  and exponents  $p$  and  $q$  vary with impeller type and system geometry. Therefore, other geometric ratios and groups are needed, as well as the functional relationships between correlating parameters.

The second method involves the particle Sherwood number  $\frac{kd_p}{D_v}$  as a function of particle Reynolds number  $ud_p / D_v$ , where  $u$  is the average relative velocity between fluid and solid. The relative velocity is a function of power input and fluid viscosity.

Relative velocity can also be defined as the root mean square (RMS) velocity fluctuation which is proportional to the particle diameter. It may be assumed that the scales of turbulent fluctuations are in the so called inertial sub range of the eddy energy distribution, so that the RMS velocity fluctuation depends primarily on the mean power per unit volume and the particle size.

Applying this assumption to the particle Reynolds number yields the following equation:

$$\frac{kd_p}{D_v} N_{sc}^{-1/3} = f \left( \frac{P/Vd_p^4}{u} \right)^{1/3} \quad (3.3)$$

The third correlation method is based on the slip viscosity and terminal velocity of the particle. In a reactor in which the solid particles are fully suspended, Harriott<sup>(10)</sup> showed that the relative particle liquid velocity is always greater than the free-fall terminal velocity of the solid particles in static liquid.

### 3.2 Dimensional Analysis

Many dimensionless equations for agitation power have been derived by among of early investigators using dimensional analysis. This considered that impeller power should be a function of the geometry of the impeller and the tank, the properties of the fluid (viscosity and density), the rotational speed of the impeller, and gravitational force. The Buckingham pi theorem gives the following general dimensionless equation for the relationship of the variables:

$$f \left( \frac{D^2 N_p}{\mu}, \frac{DN^2}{g}, \frac{Pg_c}{\rho N^3 D^5}, \frac{D}{T}, \frac{D}{Z}, \frac{D}{C}, \frac{D}{p}, \frac{D}{W}, \frac{D}{l}, \frac{n_2}{n_1} \right) = 0 \quad (3.4)$$

whereas

- $D$  = impeller diameter
- $T$  = tank diameter
- $Z$  = liquid depth
- $C$  = clearance of impeller off vessel bottom



$W$	= blade width
$p$	= pitch of blade
$n$	= number of blades
$l$	= blade length
$\rho$	= density
$P$	= Power consumption
$\mu$	= Viscosity
$N$	= impeller rotational speed
$g$	= gravitational acceleration
$g_c$	= Newton's law conversion factor

Nagata used this correlation to investigate the impeller power input in slurry system and modified by the density factor, i.e.  $P_{A,Sl} = \frac{P_A \rho_{Sl}}{\rho_L}$  Total power input referred to slurry volume is given by

$$\frac{P_T}{V_{Sl}} = \frac{P_A}{V_{Sl}} \frac{\rho_{Sl}}{\rho_L} + \frac{E}{V_{Sl}} \quad (3.2)$$

whereas

$P_T$	= Total power input in aerated stirred liquid
$P_A$	= Power input in aerated stirred liquid
$V_{Sl}$	= Volume of slurry
$E$	= Energy input

### 3.2.1 Similarity

Equality of all groups in Eq. (3.1) assures similarity between systems of different size. The types of similarity of interest here are geometric, kinematic, and dynamic.

#### 3.2.1.1 Geometric similarity.

The last seven terms in Eq. (3.1) represent the condition of geometric similarity which requires that all corresponding dimensions in systems of different size bear the same ratio to each other. The reference dimension used is the impeller

diameter. The last term in Eq.(3.1) is not a linear dimension relationship but is required to account for change in number of impeller blades.

Equation (3.1) assumes a single impeller centered on the axis of a vertical cylindrical flat bottom tank. To be fully inclusive, the equation would have to be expanded to include:

- a. off-center impeller positions
- b. multiple impellers
- c. baffle width and number of baffles
- d. tank shape

### 3.2.1.2 Dynamic and kinematic similarity.

Given geometric similarity, two systems are dynamically similar when the ratios of all corresponding forces are equal, Kinematic similarity requires that velocities at corresponding points be in the same ratio. These two similarity criteria are presented together since they are interrelated in a fluid system.

Confining the discussion to geometrically similar systems, Eq. (3.1) may be stated as

$$f\left(\frac{D_i^2 N \rho}{\mu}, \frac{D_i N^2}{g}, \frac{P g_c}{D_i^5 N^3 \rho}\right) = 0 \quad (3.3)$$

Equation of the groups in this expression insures dynamic and kinematic similarity. This relationship was derived by dimensional analysis, but the same dimensionless groups may also be obtained from the Navier-Stokes equation of motion. A complete discussion of the derivation and application of the Navier-Stokes equation is given in standard references such as Bird et al. and Schlichting. It will not be presented here. For the complex, three-dimensional flow in a mixing system an analytical solution to the Navier-Stokes equations cannot be obtained. However, the equations may be stated in dimensionless form as follows:

$$f\left(\frac{\rho v L}{\mu}, \frac{v^2}{Lg}, \frac{\Delta P}{v^2 \rho}\right) = 0 \quad (3.4)$$

whereas

$v$  = velocity

$L$  = a characteristic length

$\Delta P$  = pressure difference

The groups in this equation are the time as those of Eq.(3.3), as will be demonstrated below, and a definite physical significance may be attributed to each group.

### 3.2.2 Physical significance of dimensionless groups

#### 3.2.2.1 Reynolds number, $N_{Re}$

The first group in Eq. (3.4) is the Reynolds number representing the ratio of inertial forces to viscous forces. Since this ratio determines whether the flow is laminar or turbulent, Reynolds number is a critical group in correlating of the variable in various systems. In similar systems, any convenient velocity and length may be used in the Reynolds number. For agitation, the following are the ones generally employed:

$L = D_i$  and  $v = ND_i$  substitution gives

$$N_{Re} = \frac{(\rho)(ND_i)(D_i)}{\mu} = \frac{D_i^2 N \rho}{\mu} \quad (3.5)$$

This is identical to the group derived by dimensional analysis.

#### 3.2.2.2. Froude number, $N_{Fr}$

This group is representing the ratio of inertial to gravitational forces. Substituting Eq. 3.5 into this group gives for an agitator:

$$N_{Fr} = \frac{(ND_i)^2}{(D_i g)} = \frac{D_i N^2}{g} \quad (3.6)$$

In many fluid flow problems, gravitational effects are unimportant and the Froude number is not a significant variable. The reason it is included here is that most agitation operations are carried out with a free liquid surface in the tank. The shape of surface and, therefore, the flow pattern in the vessel, are affected by gravitational field. This is particularly noticeable in unbaffed tanks where vortexing occurs; the shape of vortex represents a balancing of gravitational and inertial forces.

### 3.2.2.3 Power number, $N_p$

The pressure coefficient in Eq.(3.4) represents the ratio of differences pressure that producing flow to inertial forces. In practice, the pressure distribution is not known, but in dynamically similar systems it can be shown that  $\Delta P$  and power consumption for agitating a liquid are related by :

$$\frac{kP}{ND_i^3} = \Delta P \quad (3.6)$$

Making this substitution into the pressure coefficient together with the reference velocity  $v = ND_i$  gives

$$N_p = \frac{Pg}{(\rho N^3 D_i^5)} \quad (3.7)$$

The power number derived from dimensional analysis is the same.

An understanding of the physical significance of the power number is enhanced by considering it as a drag coefficient or friction factor. The drag coefficient of a solid body immersed in a flowing stream is usually defined as

$$C_D = \frac{F_D g_c}{\left(\frac{\rho v^2}{2}\right)A} \quad (3.8)$$

whereas

- $C_D$  = drag coefficient
- $F_D$  = drag force on the body
- $v$  = velocity of flowing stream
- $A$  = cross-sectional area of the body

For a mixing impeller,  $N_p$  can be show to be analogous to  $C_D$  from the following arguments:

$$v \propto ND_i$$

$$A \propto D_i^2 \quad (\text{geometrically similar impellers})$$

$$P \propto NF_D D_i$$

Introduction of these relationships into Eq. (3.9) gives

$$C_D \propto \frac{\left[ \left( \frac{P}{ND_i} \right) g_c \right]}{\rho (ND_i)^2 D_i^2} \quad (3.10)$$

simplifying

$$C_D \propto \frac{[Pg_c]}{\rho N^3 D_i^5} \quad (3.11)$$

or

$$C_D \propto N_p \quad (3.12)$$

The analogy of  $C_D$  to  $N_p$  is a useful for observation. The correlations of drag coefficients and power number have many relationships to each other. For pressure drop in pipes, the use of friction factor is analogous to  $N_p$  for impellers and  $C_D$  for immersed bodies.

### 3.3 Standard Tank Configuration<sup>(21)</sup>

The vessel configurations, shown in Figure 3.1 are known as the standard tank configuration. The Standard tank configuration has the following geometrical relationships:

- The agitator is a standard six-bladed turbine
- Impeller diameter,  $D_i = 1/3$  tank diameter,  $T$ .
- Impeller height from the tank bottom,  $H_i = 1.0$  impeller diameter.
- Impeller blade length,  $L = 1/4$  impeller diameter.
- Impeller blade width,  $W = 1/5$  impeller diameter.
- Liquid height,  $H_l = 1.0$  tank diameter.

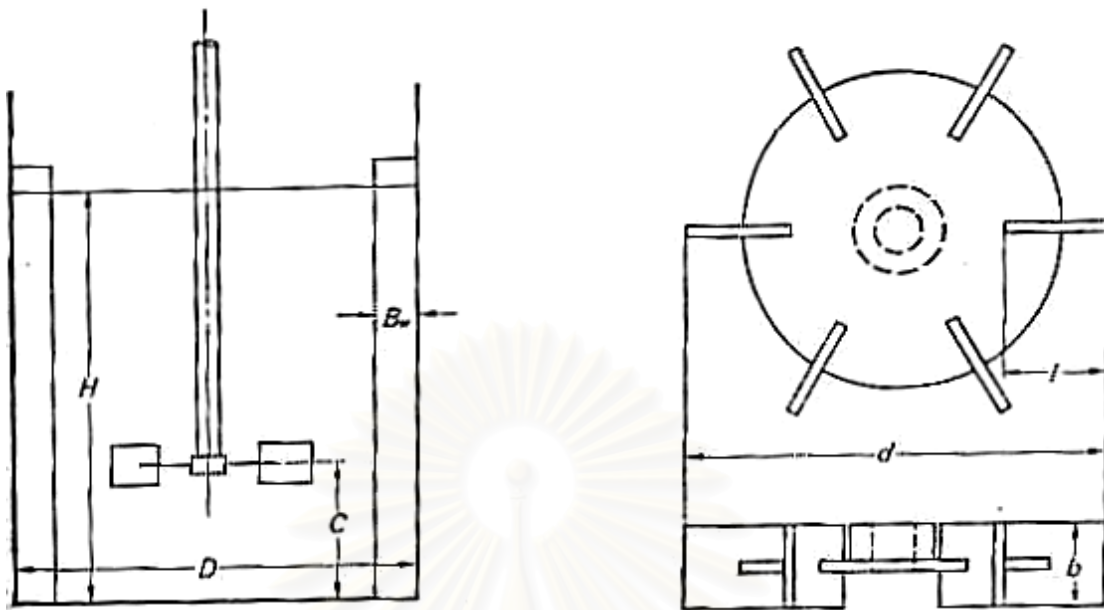


Figure 3.1 Standard Tank Configuration <sup>(18)</sup>

### 3.4 Mixing time correlation <sup>(18)</sup>

Mixing time required for completely mixed condition is one of important parameters in a mixing process. If the time of mixing employed is much longer than the required mixing time, it would waste time and energy. In term for industrial process, it means decreasing production capacity and increasing extra expenditure. In many cases excessive agitation can cause segregation of the components to be mixed on the other hand, if the time of mixing is insufficient a non uniform product will be formed.

The dimensionless mixing time,  $\tau = t_m(N)$ , is influenced by the geometry of the mixing vessel, the injection methods for the ingredients, and  $R_e$  or  $P_e$ , which controls the convection flow or diffusion.

In geometrically similar vessels, the state of liquid flow is classified roughly by Reynolds number,  $R_e = \frac{D_i^2 N}{\nu}$  as follows;

1) a low Reynolds range where the impeller speed is not large and the secondary circulation flow due to the centrifugal effect is negligible.

2) a medium Reynolds range where the secondary circulation flow is appreciable (laminar range)

3) a high Reynolds range (a complete turbulent state)

In each individual range, the liquid flow pattern is similar. In a completely turbulent flow range, the turbulent Peclet number ( $P_{et}$ ) gives a certain constant value, so that dimensionless mixing time  $\tau = t_m N$  shows a constant value and  $t_m$  is inversely proportional to  $N$ .

Blend time,  $t_m$ , can be defined as the time from the start of mixing at some unmixed condition until the vessel contents reach a predetermined value of uniformity. All the particles are suspended relatively uniformly through out the tank, with the exception of the top near the surface, depending upon the settling velocity of the particles. Frequently used criteria include the time to reach specified variations in temperature, density, component concentration, etc.

No universally accepted definition exists of what constitutes a completely blend. Some process may require as little as 95 % uniformity, while other may require in excess of 99.9 %. Methods of determining uniformity must be considered. The degree of uniformity must be established on the basis of process objectives or decided for each specific case. Since concentration is dependent on location, multiple samples at different locations are required to assure uniformity.

### 3.5 Product Data Calculations <sup>(1)</sup>

#### 3.5.1 Simplified Analysis Batch Operation

Batch mixers are normally evaluated on the basis of the mixing time required to obtain a specified mixture.

Properties of interest are examined as a function of the mixing time, and the required mixing time is the shortest time at which there is satisfactory assurance that the values of properties are within selected limits. Decisions may be based on visual examination of a plot of time of sampling (abscissa and independent variable) versus

measured values of the property (ordinate and dependent variable) for many operations. Evaluations may also be made at different operating conditions (for example, mixer speeds) and the effect of important operating variables determined by examining the required mixing time as a function of the operating variables.

AIChE Equipment Testing Procedure recommended the selection of the mixing time of the curing-rate additive as the time required to achieve concentration of mixture within acceptable limit as shown in Figure 3.2 or to reach composition equilibrium by statistical analyzing as shown in Figure 3.3

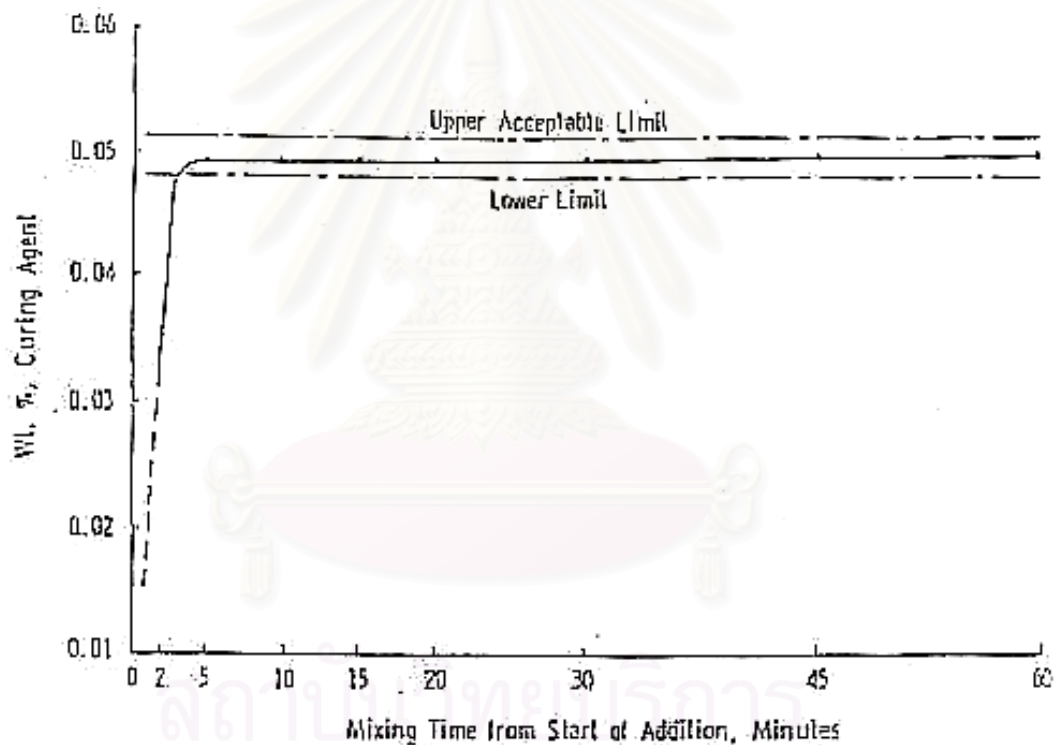


Figure 3.2 Composition & mixing time in 150 gal. Dough mixer <sup>(1)</sup>

Kramer et al. (1953) selected required mixing time as the time for concentration variations to become less than 0.1 % of the average KCl concentration as shown in Figure 3.4.



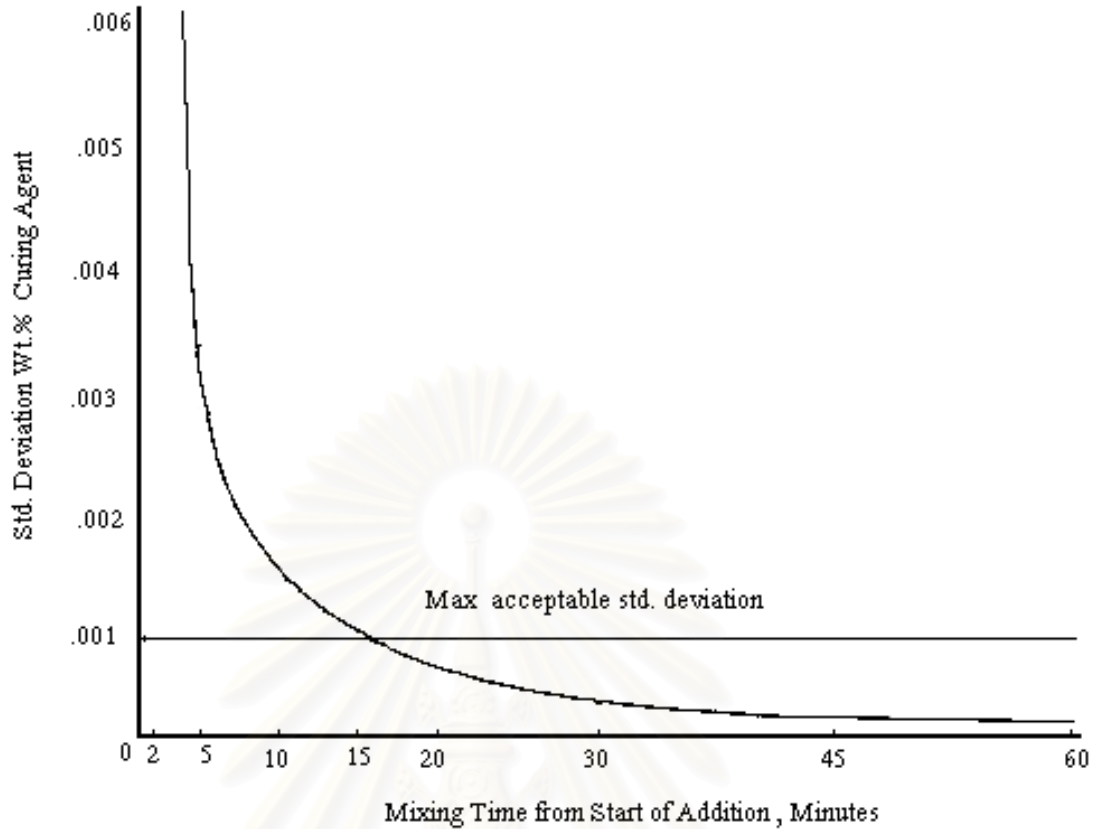


Figure 3.3 Standard variation & mixing time in 150 gal. Dough mixer <sup>(1)</sup>

Noi et al. <sup>(20)</sup> selected required mixing time as the time to achieve variation of concentration of radioactive material (count rate) at both detectors less than 1 time of standard deviation as shown in Figure 3.5 (a) and Figure 3.5(b).

Pipop T. <sup>(3)</sup> selected required mixing time as the time to achieve variation of concentration of radioactive (count rate) at both detectors less than 3 times of standard deviation as shown in Figure 3.6.

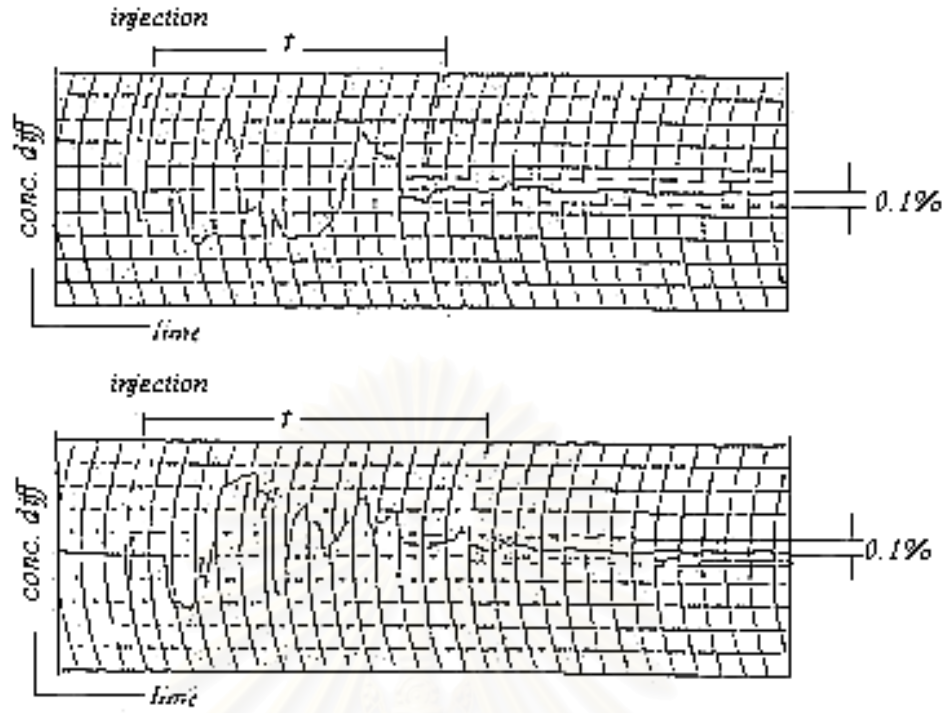


Figure 3.4 Method of determining mixing time <sup>(15)</sup>

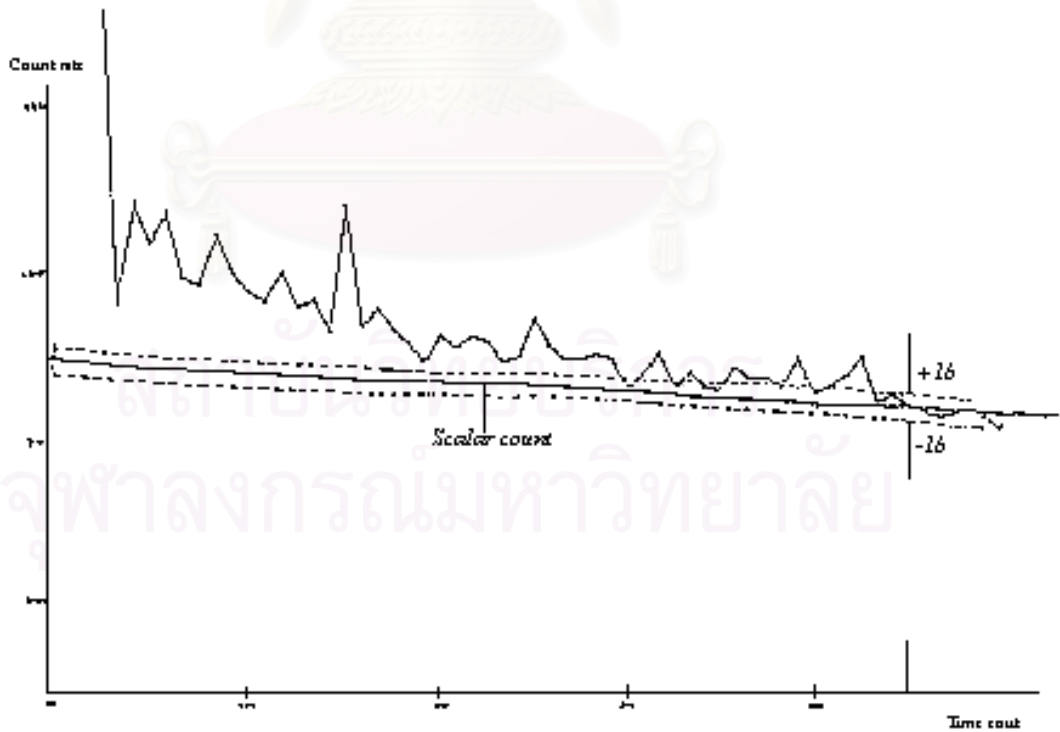


Figure 3.5 (a) Scalar counts from detector No. 1 <sup>(20)</sup>

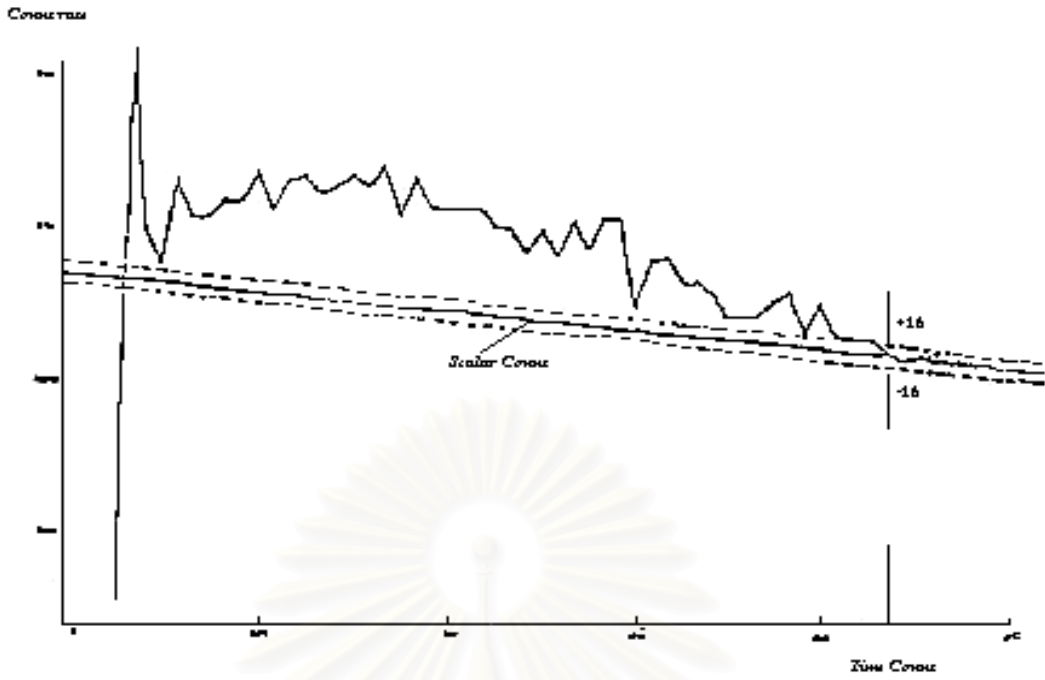


Figure 3.5 (b) Scalar counts from detector No. 2 <sup>(20)</sup>

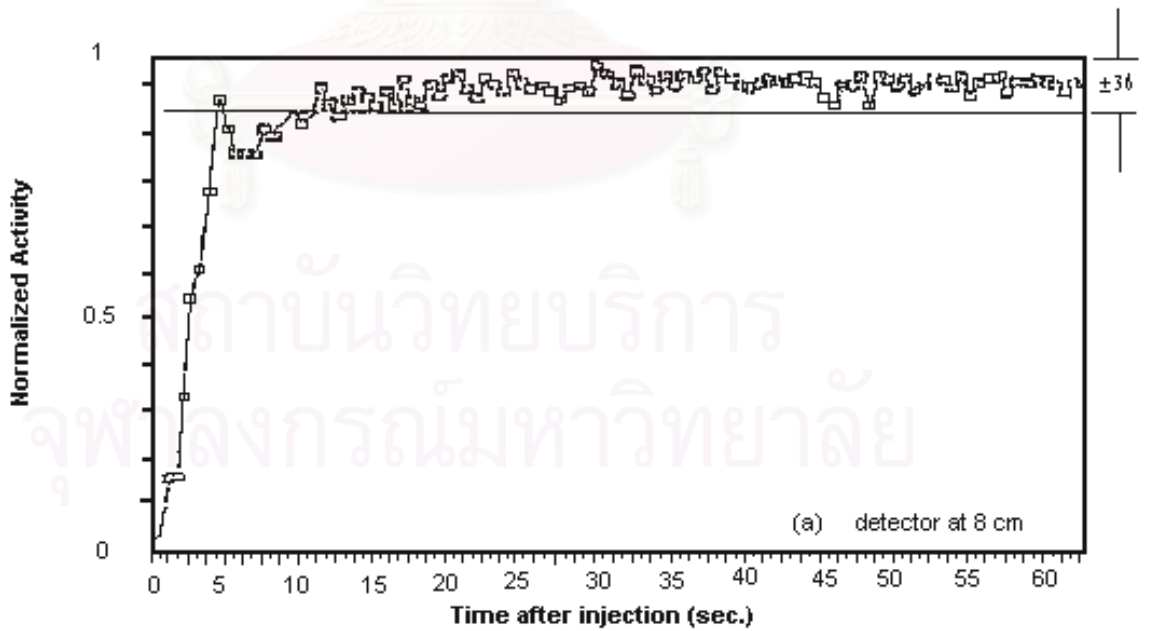


Figure 3.6 Tracer respond curve using radioactive technique. <sup>(3)</sup>

### 3.5.2 Effect of operating conditions

The effect of important operating variables determined by examining the required mixing time as a function of the operating variables speed, viscosity etc.

Kramer et al.<sup>(15)</sup> studied the mixing time of weak and concentrated KCl solutions using the propellers and turbines. Two sizes of vessels were used (10.5 in. and 21. in. diameter tank). Kramer used electrical conductivity cell for measuring fluctuations of solutions. The data which Kramer obtained on experiments shown that the mixing time,  $t_m$ , was found to be inversely proportional to the agitator rotation speed,  $N$ , as shown in Figure 3.7.

Marr<sup>(18)</sup> carried out experimental measurements of batch mixing time using a phenolphthalein indicator, NaOH and HCl solution. First, NaOH and indicator were added to the vessel with the agitator in motion. Then, HCl solution was added adjacent to the impeller and the time for the red color to disappear was measured. A 11.5 in. diameter tank with three 1.5 in. wide, vertical baffles was used. It was found that  $t_m$  was proportional to  $1/N$ .

A plot of apparent viscosity versus shear rate on log-log coordinates gives a straight line of negative slope as shown in Figure 3.9

Metzer and Otto<sup>(18)</sup> were proposing a useful procedure for prediction of power consumption in non-Newtonian fluids using fundamental viscometric data. It found that the fluid motion in the vicinity of the impeller could be characterized by relating shear rate to impeller speed as follows:-

$$\text{Shear rate} \propto N$$

This equation is a useful design equation since it can be used to calculate the average viscosity of a non-Newtonian liquid in an agitated vessel at any agitator speed.

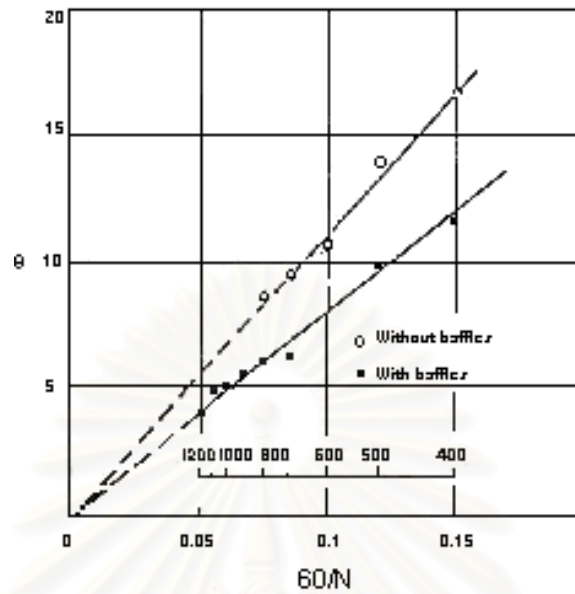


Figure 3.7 Relationship between mixing time and rotation speed <sup>(15)</sup>

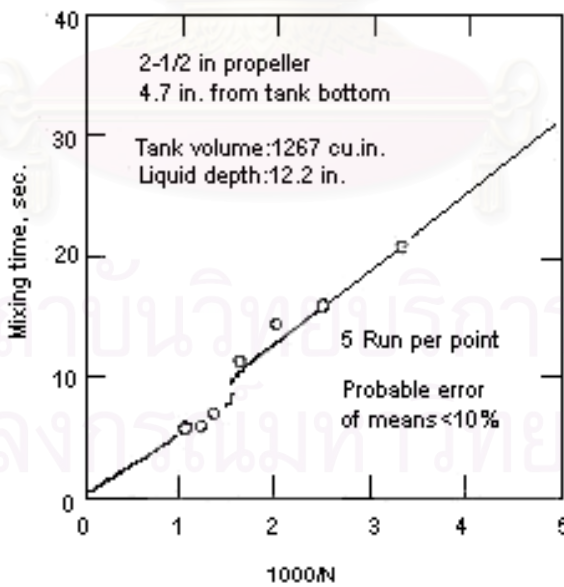


Figure 3.8 Relationship between mixing time and rotation speed <sup>(18)</sup>

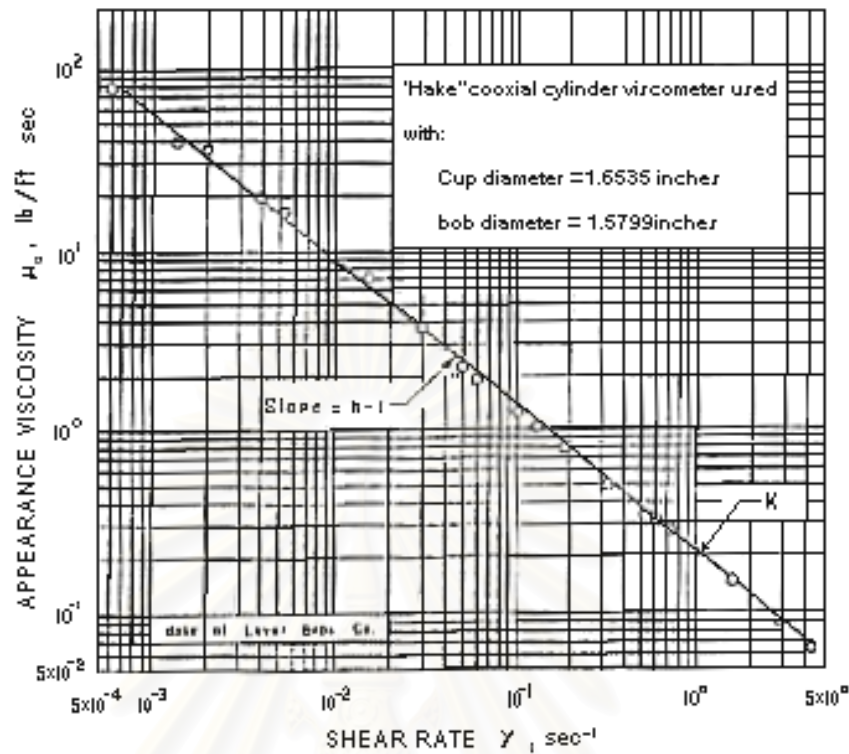


Figure 3.9 Apparent viscosities versus shear rate <sup>(25)</sup>

### 3.6 Flow Pattern in Agitated Vessel

The type of flow in an agitated vessel depends on the type of impeller, the characteristics of the fluid, and the size and proportions of the tank, baffles, and impeller. The velocity of the fluid at any point in the tank has three components, and the overall flow pattern in the tank depends on the variations in these three velocity components from point to point. The first velocity component is radial and acts in a direction perpendicular to the shaft of the impeller. The second component is longitudinal and acts in a direction parallel with the shaft. The third component is tangential, or rotational, and acts in a direction tangent to a circular path around the shaft. In the usual case of a vertical shaft, the radial and tangential components are in a horizontal plane, and the longitudinal component is vertical. The radial and longitudinal components are useful and provide the flow necessary for the mixing action. When the

shaft is vertical and centrally located in the tank, the tangential component is generally disadvantageous. The tangential flow follows a circular path around the shaft, creates a vortex at the surface of liquid, as show in Figure.3.10, and tends to perpetuate, by a laminar-flow circulation, stratification at the various levels without accomplishing longitudinal flow between levels. If solid particles are present, circulatory currents tend to throw the particles to the outside by centrifugal force, from the where they move downward and to the center of the tank at the bottom. Instead of mixing, its reverse, concentration, occurs. Since, in circulatory flow, the liquid flows with the direction of motion of the impeller blades, the relative velocity between the blades and the liquid is reduced and the power that can be absorbed by the liquids is limited. In an unbaffled vessel circulatory flow is induced by all type of impeller, whether axial flow or radial flow. In fact, if the swirling is strong, the flow pattern in the tank is virtually the same regardless of the design of the impeller. At high impeller speed the vortex may be so deep that it reaches the impeller, and gas from above the liquid is drawn down into the charge. Generally this is undesirable.

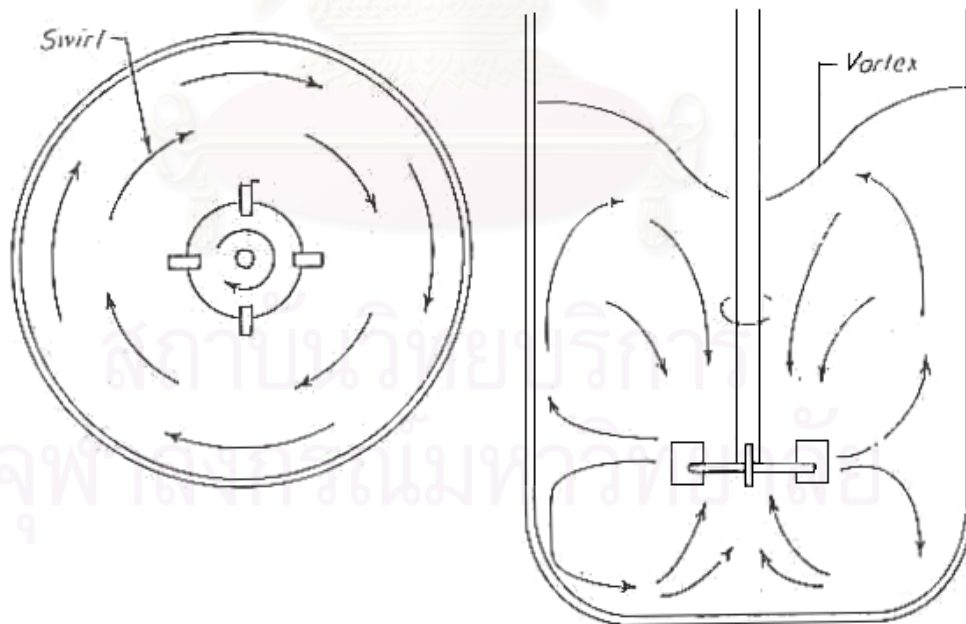


Figure 3.10 Vortex formation and circulation pattern in and agitated tank<sup>(7)</sup>

## CHAPTER IV

### EXPERIMENTAL

#### 4.1 The Apparatus

The standard mixing tank and apparatus consist of two major parts mixing system and tracer measurement system.

##### 4.1.1 Mixing system

Standard tank configuration was used in this study having diameter is 25 cm. Mild steel was selected as construction material. The impeller used is standard high speed shear impeller positioned in the vessel axis, at the 1/3 of high from bottom of the still liquid.

Agitator: A US digital variable speed motor model ML-4RT equipped with a controller unit, attached with a single-stage rotor-stator mixer, a high-speed rotor turn within a slotted stator. The mixer design as shown in Figure 4.1, 4.2 and 4.3 was used in this study and Table 4.1 summarized configurations of mixing tanks.

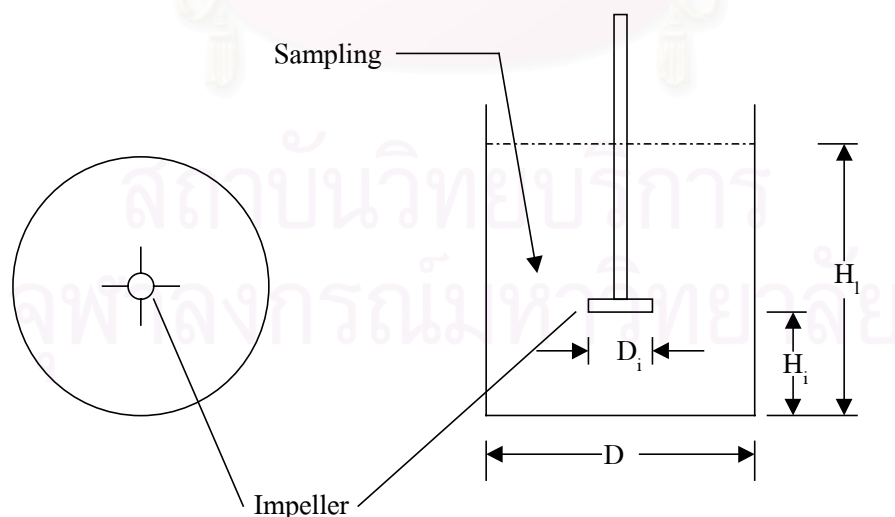


Figure 4.1 Setup of impeller, liquid level, diameter of tank and sampling point



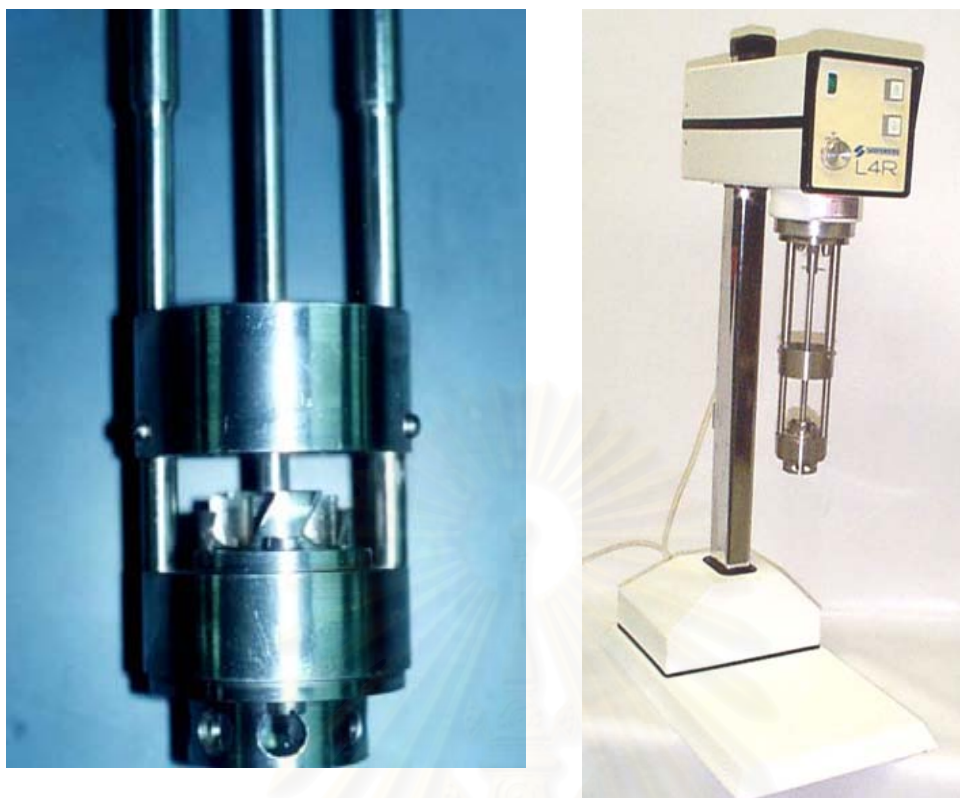


Figure 4.2 Single-stage rotor-stator shear impeller and Silverson's high shear rotor/stator batch mixer for Experiment



Figure 4.3 General purpose disintegrating head of Shear mixer and digital tachometer

Table 4.1 Configuration of mixing tanks

Conditions	Dimension (cm)
Liquid level, $H_i$	25
Impeller height from tank bottom, $H_i$	8.33
Impeller diameter, $D_i$	8.33
Impeller blade width, $W$	1.67
Impeller blade length, $L$	2.08
Number of blades	6
Angle of blade	$90^\circ$
Tank diameter	25

#### 4.1.2 Tracer measurement system

UV Spectrophotometer was used as tracer measurement to determine the required mixing time. UV Spectrophotometer consists of the following:-

Type of spectrometer: Double Beam UV/VIS Spectrophotometer, model Lambda 12

Optional Components: Automatic control system and analysis program (UV win lab)

For Figure 4.4 show Double Beam UV/VIS Spectrometer setup

#### 4.1.3 Auxiliary equipment

Tachometer : A US made Lutron, digital tachometer model DT-2232



Figure 4.3 Double Beam UV/VIS Spectrophotometer

## 4.2 Chemical

Chemicals used in this experiment were:

Basic oil: Various basic oil supplied by The Shell Company of Thailand Limited and classified by viscosity @100 °C as 4.7, 11.2 and 32.0 cSt (approximately) were used.

Additive: Powder of sulphur supplied by The Shell Company of Thailand Limited and classified by percentage of Sulphur content and dissolving in mineral oil is facilitated by the use of a small particle size.

Typical properties of base oil and Sulphur additive are shown in Appendix B, C, D and E respectively

### 4.3 Procedure

The objective of this experiment was to obtain the required mixing time by observing the dissolving of Sulphur powder in mineral oils.

The procedures for experiments were summarized as follows:-

#### 4.3.1 Experiment

The following is an outline of the procedure for each experiment. The mixing tank was mounted on a support. Half of all basic oils were charged into blending tank followed by all additives and topped up to the balance which is equal to the tank's diameter with basic oils. The agitator was mounted on a support at the desired impeller level as shown in Figure 4.1. The heater was turned on and adjusted to the desired temperature. This temperature was controlled to be  $\pm 2$  °C from the set value. Impeller drive was supplied, and adjusted to the desired speed (digital variable speed). The speed was confirmed by a digital tachometer at top drive shaft. Fluctuations of speed were no greater than 5 rpm. from any set speed. Every 10 minute, 0.5 ml. of sample was sampled at middle level of the vessel for measurement of Sulphur content by UV Spectrophotometer. The sampling process was continued every 10 minutes until the end of blending process. (At least one hour to ensure getting homogeneous lubricating oil) The drawn samples were prepared into Quart cell for measurement of Sulphur content that do not dissolve as described in the standard test method (Appendix A-3). The sampling times were plotted against the measured value, Sulphur content, to determine the required mixing time.

Samples were taken after completion of each mixing process for analysis against the quality in specifications to ensure that the experiment resulted in homogeneous lubricating oils. The tested items can be shown as follows:-

- Density (ASTM D-1298)
- Viscosity, kinematics (ASTM D-445)

The detail of each method can be shown in Appendix A.

The experiments were test on parameters studied in this work were the effect by the speed of agitator, viscosity of lubricating oils, and temperature on the required mixing time.

The ranges of parameter studied were the following:

- Diameter of tank: 25 cm. Respectively
- Type of impeller: single-stage rotor-stator mixer, a high-speed roter turn within a slotted stator.
- Speed of impeller: 300, 400, 500 rpm.
- Baffles: without baffles installation
- Viscosity of lubricating oils @100 °C as 4.7, 11.2 and 32.0 cSt
- Temperature for mixing : 80, 100, 120 °C

#### 4.3.2 Determination of Solid Dissolution Rate Coefficient <sup>(7)</sup>

The determination of dissolution rate coefficient concerns the measuring of concentrations before and after diffusion.

Rate of solid dissolution in this study is calculated by using Hixson and Crowell equation (2.2)

Shape factor is required to define the size of a particle by single dimension. The variation between these diameters increases as the particles diverge more from the spherical shape and hence shape is an important factor in the correlation of sizing analyses made by various procedures.

One of the earliest defined shape factor is the Circularity ( $\psi_c$ ) by used microscope analysis. Circularity is the ratio of the perimeter of a circle having the same area as the projected area of the particle to the actual particle perimeter. This is clearly only a two dimensional representation of a particle shape and as such can be evaluated by microscopy, preferably linked to an image analyzer. For circular images, microscope analysis prefers the definition:

$$\psi_c = \frac{S_c}{S_A} \quad (4.2)$$

whereas

$\psi_c$  = Circularity (Shape factor is the circle)

$S_c$  = The perimeter of circle having the same area as the projected area of the particle.

$S_A$  = The actual particle perimeter

For determined in this work were presented in Appendix H

#### 4.3.3 Determination of Solubility

Accurate values of saturation concentration (solubility) are essential for a proper calculation of mass diffusion. No previous work has been reported for solubility of sulphur powder in mineral oil at any other temperatures. Therefore solubility at different temperatures between 80 to 120 °C was determined in this work. The solubility data of %Sulphur dissolve in mineral oil at temperature between 80 to 120 °C were presented in Appendix C

#### 4.3.4 Dimensional Analysis of Dissolution Rate Correlation

In solid liquid agitation the entire mass transfer relation can be expressed by independent variable as

$$K = f(D_i, \rho, \mu, D_v, W)$$

In agitated solid liquid systems, the appropriate equation is

$$\frac{KT}{D_v} = f\left(\frac{D_i^2 w \rho}{\mu}, \frac{\mu}{\rho D_v}\right)$$

$$Sh_T = f(Re_a, Sc) \quad (4.3)$$

whereas  $Sh_T$  is the Sherwood number referred to agitated tank,  $\frac{KT}{D_v}$

$Re_a$  is the Reynolds number referred to agitator,  $\frac{D_i^2 w \rho}{\mu}$

$Sc$  is the Schmidt number,  $\frac{\mu}{\rho D_v}$

In this work the result will be correlated in terms of tank Sherwood number as a function of agitator Reynolds number and Schmidt number.



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## CHAPTER V

### RESULTS AND DISCUSSIONS

#### 5.1 General

The important factors for blending process which are used for the criteria of a suitable indicator that represents homogenous conditions are the total volume of samples should be minimized and performed with a minimal effect on the lubricant formulation.

Percentage of Sulphur in base oil that does not dissolve was selected as the parameter to determine the required mixing time and indicate the homogeneity of the mineral oil.

A set of 27 mixing experiment was conducted in one type of agitated batch mixer. In each batch mixer, different measurements are carried out to determine the required mixing time in agitated batch mixer for the following conditions:-

1. Speed of impeller: 300, 400 and 500 rpm.
2. Viscosity of base oils @ 100 °C: 4.7, 11.2 and 32 cSt.
4. Temperature for blending mineral oils: 80, 100 and 120 °C.

From above conditions were summarized as in Table 5.1

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Table 5.1 Experimental Conditions

Experiment No.	Viscosity of base oil@100°C, cSt. (approximate)	Temperature for Blending, °C	Speed, rpm.
1	4.7	80	300
2	4.7	80	400
3	4.7	80	500
4	4.7	100	300
5	4.7	100	400
6	4.7	100	500
7	4.7	120	300
8	4.7	120	400
9	4.7	120	500
10	11.2	80	300
11	11.2	80	400
12	11.2	80	500
13	11.2	100	300
14	11.2	100	400
15	11.2	100	500
16	11.2	120	300
17	11.2	120	400
18	11.2	120	500
19	32.0	80	300
20	32.0	80	400
21	32.0	80	500
22	32.0	100	300
23	32.0	100	400
24	32.0	100	500
25	32.0	120	300
26	32.0	120	400
27	32.0	120	500

## 5.2 Instrumental and Experimental error

### 5.2.1 Instrumental error

UV Spectrophotometer instrument is used to determine %Sulphur that do not dissolve in base oil. Accuracy of the instrument is found by analysis of same sample twice. It is found that the accuracy is within  $\pm 3$  percent, as shown in Table 5.2.

Table 5.2 Precision of concentration of Sulphur-content at various times

Sampling time	% Sulphur Content (%wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	%Error	Test-1	Test-2	Average	%Error
0	0.174	0.172	0.173 $\pm$ 0.001	1.156	0.172	0.172	0.172 $\pm$ 0.000	0.000
1	0.173	0.174	0.174 $\pm$ 0.001	0.576	0.171	0.171	0.171 $\pm$ 0.000	0.000
2	0.172	0.172	0.172 $\pm$ 0.000	0.000	0.171	0.172	0.172 $\pm$ 0.001	0.583
3	0.174	0.174	0.174 $\pm$ 0.000	0.000	0.171	0.171	0.171 $\pm$ 0.000	0.000
4	0.172	0.173	0.173 $\pm$ 0.001	0.580	0.171	0.171	0.171 $\pm$ 0.000	0.000
5	0.172	0.172	0.172 $\pm$ 0.000	0.000	0.171	0.172	0.172 $\pm$ 0.001	0.583
6	0.172	0.172	0.172 $\pm$ 0.000	0.000	0.170	0.171	0.171 $\pm$ 0.001	0.587
7	0.172	0.173	0.173 $\pm$ 0.001	0.580	0.171	0.171	0.171 $\pm$ 0.000	0.000
8	0.172	0.172	0.172 $\pm$ 0.000	0.000	0.171	0.171	0.171 $\pm$ 0.000	0.000
9	0.172	0.173	0.173 $\pm$ 0.001	0.580	0.171	0.171	0.171 $\pm$ 0.000	0.000
10	0.173	0.172	0.173 $\pm$ 0.001	0.580	0.168	0.169	0.169 $\pm$ 0.001	0.593
11	0.173	0.172	0.173 $\pm$ 0.001	0.580	0.170	0.170	0.170 $\pm$ 0.000	0.000
12	0.172	0.172	0.172 $\pm$ 0.000	0.000	0.170	0.171	0.171 $\pm$ 0.001	0.587
13	0.172	0.172	0.172 $\pm$ 0.000	0.000	0.171	0.171	0.171 $\pm$ 0.000	0.000
14	0.171	0.171	0.171 $\pm$ 0.000	0.000	0.171	0.171	0.171 $\pm$ 0.000	0.000
15	0.172	0.173	0.173 $\pm$ 0.001	0.580	0.171	0.171	0.171 $\pm$ 0.000	0.000
16	0.171	0.171	0.171 $\pm$ 0.000	0.000	0.171	0.171	0.171 $\pm$ 0.000	0.000
17	0.171	0.171	0.171 $\pm$ 0.000	0.000	0.171	0.171	0.171 $\pm$ 0.000	0.000
18	0.170	0.171	0.171 $\pm$ 0.001	0.587	0.171	0.171	0.171 $\pm$ 0.000	0.000
19	0.169	0.169	0.169 $\pm$ 0.000	0.000	0.171	0.171	0.171 $\pm$ 0.000	0.000
20	0.169	0.169	0.169 $\pm$ 0.000	0.000	0.170	0.170	0.170 $\pm$ 0.000	0.000

Table 5.2 Precision of concentration of Sulphur-content at various times(Continue)

Sampling time	% Sulphur Content (%wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	%Error	Test-1	Test-2	Average	%Error
21	0.169	0.169	0.169 ± 0.000	0.000	0.170	0.170	0.170 ± 0.000	0.000
22	0.176	0.176	0.176 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
23	0.176	0.176	0.176 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
24	0.176	0.177	0.177 ± 0.001	0.567	0.171	0.171	0.171 ± 0.000	0.000
25	0.176	0.176	0.176 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
26	0.175	0.175	0.175 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
27	0.175	0.176	0.176 ± 0.001	0.570	0.171	0.171	0.171 ± 0.000	0.000
28	0.176	0.176	0.176 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
29	0.175	0.176	0.176 ± 0.001	0.570	0.172	0.171	0.172 ± 0.001	0.583
30	0.176	0.176	0.176 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
31	0.175	0.176	0.176 ± 0.001	0.570	0.172	0.172	0.172 ± 0.000	0.000
32	0.175	0.176	0.176 ± 0.001	0.570	0.171	0.172	0.172 ± 0.001	0.583
33	0.174	0.174	0.174 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
34	0.173	0.174	0.174 ± 0.001	0.576	0.171	0.171	0.171 ± 0.000	0.000
35	0.173	0.174	0.174 ± 0.001	0.576	0.171	0.171	0.171 ± 0.000	0.000
36	0.172	0.173	0.173 ± 0.001	0.580	0.171	0.171	0.171 ± 0.000	0.000
37	0.173	0.173	0.173 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
38	0.173	0.173	0.173 ± 0.000	0.000	0.170	0.171	0.171 ± 0.001	0.587
39	0.173	0.172	0.173 ± 0.001	0.580	0.170	0.170	0.170 ± 0.000	0.000
40	0.172	0.174	0.173 ± 0.001	1.156	0.170	0.170	0.170 ± 0.000	0.000
41	0.172	0.172	0.172 ± 0.000	0.000	0.170	0.170	0.170 ± 0.000	0.000
42	0.171	0.172	0.172 ± 0.001	0.583	0.171	0.170	0.171 ± 0.001	0.587
43	0.171	0.172	0.172 ± 0.001	0.583	0.171	0.171	0.171 ± 0.000	0.000
44	0.171	0.172	0.172 ± 0.001	0.583	0.171	0.171	0.171 ± 0.000	0.000
45	0.171	0.171	0.171 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
46	0.171	0.171	0.171 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
47	0.171	0.171	0.171 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
48	0.171	0.171	0.171 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
49	0.171	0.171	0.171 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000
50	0.171	0.171	0.171 ± 0.000	0.000	0.171	0.171	0.171 ± 0.000	0.000

Table 5.2 Precision of concentration of Sulphur-content at various times(Continue)

Sampling time	% Sulphur Content (%wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	%Error	Test-1	Test-2	Average	%Error
51	0.171	0.171	0.171 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
52	0.171	0.171	0.171 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
53	0.171	0.171	0.171 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
54	0.171	0.171	0.171 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
55	0.172	0.172	0.172 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
56	0.172	0.172	0.172 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
57	0.173	0.173	0.173 ± 0.000	0.000	0.172	0.172	0.172 ± 0.000	0.000
58	0.174	0.174	0.174 ± 0.000	0.000	0.174	0.174	0.174 ± 0.000	0.000
59	0.174	0.175	0.175 ± 0.001	0.573	0.174	0.174	0.174 ± 0.000	0.000
60	0.175	0.175	0.175 ± 0.000	0.000	0.176	0.176	0.176 ± 0.000	0.000
61	0.175	0.175	0.175 ± 0.000	0.000	0.176	0.175	0.176 ± 0.001	0.570
62	0.176	0.176	0.176 ± 0.000	0.000	0.174	0.174	0.174 ± 0.000	0.000
63	0.176	0.176	0.176 ± 0.000	0.000	0.176	0.176	0.176 ± 0.000	0.000
64	0.176	0.176	0.176 ± 0.000	0.000	0.174	0.174	0.174 ± 0.000	0.000
65	0.177	0.176	0.177 ± 0.001	0.567	0.171	0.174	0.173 ± 0.002	1.739
66	0.177	0.177	0.177 ± 0.000	0.000	0.174	0.174	0.174 ± 0.000	0.000
67	0.178	0.178	0.178 ± 0.000	0.000	0.174	0.174	0.174 ± 0.000	0.000
68	0.178	0.178	0.178 ± 0.000	0.000	0.174	0.174	0.174 ± 0.000	0.000
69	0.179	0.179	0.179 ± 0.000	0.000	0.175	0.174	0.175 ± 0.001	0.573
70	0.179	0.179	0.179 ± 0.000	0.000	0.174	0.174	0.174 ± 0.000	0.000
71	0.179	0.179	0.179 ± 0.000	0.000	0.176	0.176	0.176 ± 0.000	0.000
72	0.179	0.179	0.179 ± 0.000	0.000	0.176	0.176	0.176 ± 0.000	0.000
73	0.179	0.179	0.179 ± 0.000	0.000	0.176	0.176	0.176 ± 0.000	0.000
74	0.179	0.179	0.179 ± 0.000	0.000	0.177	0.177	0.177 ± 0.000	0.000
75	0.179	0.179	0.179 ± 0.000	0.000	0.179	0.179	0.179 ± 0.000	0.000
76	0.179	0.179	0.179 ± 0.000	0.000	0.181	0.181	0.181 ± 0.000	0.000
77	0.179	0.179	0.179 ± 0.000	0.000	0.182	0.182	0.182 ± 0.000	0.000
78	0.18	0.18	0.180 ± 0.000	0.000	0.183	0.183	0.183 ± 0.000	0.000
79	0.18	0.18	0.180 ± 0.000	0.000	0.185	0.184	0.185 ± 0.001	0.542
80	0.18	0.18	0.180 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
81	0.181	0.181	0.181 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000

Table 5.2 Precision of concentration of Sulphur-content at various times(Continue)

Sampling time	% Sulphur Content (%wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	%Error	Test-1	Test-2	Average	%Error
82	0.181	0.181	0.181 ± 0.000	0.000	0.185	0.186	0.186 ± 0.001	0.539
83	0.182	0.181	0.182 ± 0.001	0.551	0.185	0.185	0.185 ± 0.000	0.000
84	0.182	0.182	0.182 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
85	0.182	0.182	0.182 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
86	0.183	0.183	0.183 ± 0.000	0.000	0.184	0.184	0.184 ± 0.000	0.000
87	0.183	0.183	0.183 ± 0.000	0.000	0.184	0.184	0.184 ± 0.000	0.000
88	0.184	0.184	0.184 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
89	0.184	0.184	0.184 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
90	0.184	0.184	0.184 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
91	0.184	0.184	0.184 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
92	0.184	0.184	0.184 ± 0.000	0.000	0.185	0.185	0.185 ± 0.000	0.000
93	0.184	0.184	0.184 ± 0.000	0.000	0.187	0.187	0.187 ± 0.000	0.000
94	0.184	0.184	0.184 ± 0.000	0.000	0.189	0.189	0.189 ± 0.000	0.000
95	0.188	0.187	0.188 ± 0.001	0.533	0.189	0.189	0.189 ± 0.000	0.000
96	0.188	0.188	0.188 ± 0.000	0.000	0.192	0.192	0.192 ± 0.000	0.000
97	0.189	0.189	0.189 ± 0.000	0.000	0.192	0.192	0.192 ± 0.000	0.000
98	0.189	0.189	0.189 ± 0.000	0.000	0.191	0.191	0.191 ± 0.000	0.000
99	0.189	0.189	0.189 ± 0.000	0.000	0.192	0.192	0.192 ± 0.000	0.000
100	0.19	0.189	0.190 ± 0.001	0.528	0.193	0.193	0.193 ± 0.000	0.000
101	0.19	0.189	0.190 ± 0.001	0.528	0.193	0.193	0.193 ± 0.000	0.000
102	0.189	0.189	0.189 ± 0.000	0.000	0.193	0.194	0.194 ± 0.001	0.517
103	0.19	0.189	0.190 ± 0.001	0.528	0.193	0.193	0.193 ± 0.000	0.000
104	0.19	0.19	0.190 ± 0.000	0.000	0.193	0.194	0.194 ± 0.001	0.517
105	0.19	0.19	0.190 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000
106	0.19	0.19	0.190 ± 0.000	0.000	0.194	0.193	0.194 ± 0.001	0.517
107	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000
108	0.19	0.19	0.190 ± 0.000	0.000	0.192	0.193	0.193 ± 0.001	0.519
109	0.19	0.19	0.190 ± 0.000	0.000	0.192	0.193	0.193 ± 0.001	0.519
110	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000

Table 5.2 Precision of concentration of Sulphur-content at various times(Continue)

Sampling time	% Sulphur Content (%wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	%Error	Test-1	Test-2	Average	%Error
111	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000
112	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000
113	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000
114	0.19	0.191	0.191 ± 0.001	0.525	0.193	0.193	0.193 ± 0.000	0.000
115	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.194	0.194 ± 0.001	0.517
116	0.191	0.191	0.191 ± 0.000	0.000	0.192	0.193	0.193 ± 0.001	0.519
117	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.194	0.194 ± 0.001	0.517
118	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000
119	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000
120	0.191	0.191	0.191 ± 0.000	0.000	0.193	0.193	0.193 ± 0.000	0.000

### 5.2.2 Experimental error

Accuracy experimental method is also found by conducting the experiment at same conditions for three times. It is found that the accuracy is within  $\pm 10$  percent, as shown in Table 5.3.

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Table 5.3 Variation of concentration of Sulphur-content at various times

Sampling time	% Sulphur Content (%wt)									
	Experimental error (1)					Experimental error (2)				
	Batch 1	Batch 2	Batch 3	Average	%Error	Batch 1	Batch 2	Batch 3	Average	%Error
0	0.174	0.172	0.176	0.174 ± 0.002	2.299	0.210	0.208	0.206	0.208 ± 0.002	1.923
1	0.174	0.172	0.176	0.174 ± 0.002	2.299	0.210	0.207	0.207	0.208 ± 0.002	1.442
2	0.174	0.170	0.176	0.173 ± 0.003	3.462	0.219	0.210	0.207	0.212 ± 0.006	5.660
3	0.174	0.170	0.175	0.173 ± 0.003	2.890	0.217	0.217	0.209	0.214 ± 0.005	3.733
4	0.174	0.169	0.172	0.172 ± 0.003	2.913	0.220	0.22	0.211	0.217 ± 0.005	4.147
5	0.174	0.169	0.171	0.171 ± 0.003	2.918	0.221	0.219	0.212	0.217 ± 0.005	4.141
6	0.174	0.167	0.172	0.171 ± 0.004	4.094	0.224	0.22	0.213	0.219 ± 0.006	5.023
7	0.174	0.167	0.171	0.171 ± 0.004	4.102	0.225	0.221	0.214	0.22 ± 0.006	5.000
8	0.173	0.168	0.171	0.171 ± 0.003	2.930	0.226	0.223	0.213	0.221 ± 0.007	5.891
9	0.174	0.167	0.171	0.171 ± 0.004	4.102	0.227	0.222	0.214	0.221 ± 0.007	5.882
10	0.173	0.168	0.171	0.171 ± 0.003	2.930	0.226	0.222	0.215	0.221 ± 0.006	4.977
11	0.173	0.169	0.171	0.171 ± 0.002	2.339	0.226	0.223	0.217	0.222 ± 0.005	4.054
12	0.173	0.169	0.172	0.171 ± 0.002	2.335	0.225	0.223	0.217	0.222 ± 0.004	3.609
13	0.172	0.168	0.17	0.170 ± 0.002	2.353	0.226	0.222	0.216	0.221 ± 0.005	4.518
14	0.171	0.168	0.169	0.169 ± 0.002	1.772	0.227	0.223	0.217	0.222 ± 0.005	4.498
15	0.170	0.169	0.168	0.169 ± 0.001	1.183	0.226	0.223	0.217	0.222 ± 0.005	4.054
16	0.169	0.169	0.169	0.169 ± 0.000	0.000	0.226	0.223	0.218	0.222 ± 0.004	3.598
17	0.171	0.171	0.167	0.170 ± 0.002	2.358	0.226	0.223	0.218	0.222 ± 0.004	3.598
18	0.170	0.171	0.167	0.169 ± 0.002	2.362	0.226	0.223	0.218	0.222 ± 0.004	3.598
19	0.170	0.170	0.167	0.169 ± 0.002	1.775	0.226	0.224	0.218	0.223 ± 0.004	3.593
20	0.169	0.170	0.167	0.169 ± 0.002	1.779	0.226	0.224	0.218	0.223 ± 0.004	3.593
21	0.171	0.170	0.168	0.170 ± 0.002	1.768	0.227	0.224	0.220	0.224 ± 0.004	3.130
22	0.176	0.170	0.168	0.171 ± 0.004	4.669	0.226	0.225	0.220	0.224 ± 0.003	2.683
23	0.172	0.171	0.168	0.170 ± 0.002	2.348	0.227	0.224	0.220	0.224 ± 0.004	3.130
24	0.172	0.171	0.167	0.170 ± 0.003	2.941	0.227	0.224	0.220	0.224 ± 0.004	3.130
25	0.171	0.171	0.168	0.170 ± 0.002	1.765	0.227	0.225	0.220	0.224 ± 0.004	3.125
26	0.171	0.172	0.168	0.170 ± 0.002	2.348	0.227	0.225	0.221	0.224 ± 0.003	2.675
27	0.174	0.171	0.168	0.171 ± 0.003	3.509	0.228	0.225	0.221	0.225 ± 0.004	3.116
28	0.174	0.171	0.168	0.171 ± 0.003	3.509	0.228	0.225	0.221	0.225 ± 0.004	3.116
29	0.174	0.171	0.169	0.171 ± 0.003	2.918	0.229	0.225	0.220	0.225 ± 0.005	4.006

Table 5.3 Variation of concentration of Sulphur-content at various time(continue)

Sampling time	% Sulphur Content (%wt)									
	Experimental error (1)					Experimental error (2)				
	Batch 1	Batch 2	Batch 3	Average	%Error	Batch 1	Batch 2	Batch 3	Average	%Error
30	0.176	0.172	0.169	0.172 ± 0.004	4.062	0.231	0.225	0.220	0.225 ± 0.006	4.882
31	0.173	0.171	0.168	0.171 ± 0.003	2.930	0.232	0.225	0.222	0.226 ± 0.005	4.418
32	0.174	0.169	0.169	0.171 ± 0.003	2.930	0.232	0.226	0.222	0.227 ± 0.005	4.412
33	0.175	0.170	0.168	0.171 ± 0.004	4.094	0.232	0.227	0.222	0.227 ± 0.005	4.405
34	0.174	0.169	0.168	0.170 ± 0.003	3.523	0.233	0.228	0.222	0.228 ± 0.006	4.832
35	0.174	0.169	0.170	0.171 ± 0.003	2.924	0.232	0.227	0.223	0.227 ± 0.005	3.959
36	0.173	0.169	0.171	0.171 ± 0.002	2.339	0.233	0.229	0.223	0.228 ± 0.005	4.380
37	0.173	0.170	0.171	0.171 ± 0.002	1.751	0.233	0.230	0.224	0.229 ± 0.005	3.930
38	0.172	0.170	0.171	0.171 ± 0.001	1.170	0.235	0.229	0.224	0.229 ± 0.006	4.797
39	0.173	0.170	0.171	0.171 ± 0.002	1.751	0.235	0.230	0.225	0.23 ± 0.005	4.348
40	0.172	0.170	0.172	0.171 ± 0.001	1.167	0.236	0.229	0.232	0.232 ± 0.004	3.013
41	0.172	0.171	0.174	0.172 ± 0.002	1.741	0.235	0.232	0.228	0.232 ± 0.004	3.022
42	0.172	0.171	0.173	0.172 ± 0.001	1.163	0.237	0.230	0.228	0.232 ± 0.005	3.885
43	0.174	0.171	0.173	0.173 ± 0.002	1.737	0.238	0.231	0.229	0.233 ± 0.005	3.868
44	0.172	0.170	0.172	0.171 ± 0.001	1.167	0.238	0.232	0.230	0.233 ± 0.004	3.429
45	0.172	0.170	0.173	0.172 ± 0.002	1.748	0.241	0.232	0.230	0.234 ± 0.006	4.694
46	0.171	0.171	0.171	0.171 ± 0.000	0.000	0.241	0.232	0.230	0.234 ± 0.006	4.694
47	0.171	0.171	0.171	0.171 ± 0.000	0.000	0.243	0.233	0.231	0.236 ± 0.006	5.092
48	0.171	0.171	0.171	0.171 ± 0.000	0.000	0.242	0.233	0.231	0.235 ± 0.006	4.674
49	0.171	0.171	0.172	0.171 ± 0.001	0.584	0.242	0.233	0.232	0.236 ± 0.006	4.243
50	0.171	0.171	0.173	0.172 ± 0.001	1.165	0.243	0.233	0.232	0.236 ± 0.006	4.661
51	0.170	0.172	0.174	0.172 ± 0.002	2.326	0.243	0.235	0.232	0.237 ± 0.006	4.648
52	0.171	0.172	0.172	0.172 ± 0.001	0.583	0.244	0.235	0.233	0.237 ± 0.006	4.635
53	0.171	0.171	0.173	0.172 ± 0.001	1.165	0.244	0.237	0.233	0.238 ± 0.006	4.622
54	0.171	0.174	0.173	0.173 ± 0.002	1.737	0.245	0.238	0.235	0.239 ± 0.005	4.178
55	0.171	0.174	0.173	0.173 ± 0.002	1.737	0.245	0.241	0.235	0.24 ± 0.005	4.161
56	0.174	0.171	0.173	0.173 ± 0.002	1.737	0.245	0.243	0.237	0.242 ± 0.004	3.310
57	0.174	0.174	0.174	0.174 ± 0.000	0.000	0.247	0.245	0.237	0.243 ± 0.005	4.115
58	0.177	0.176	0.174	0.176 ± 0.002	1.708	0.246	0.245	0.238	0.243 ± 0.004	3.292
59	0.177	0.176	0.175	0.176 ± 0.001	1.136	0.246	0.244	0.238	0.243 ± 0.004	3.297



Table 5.3 Variation of concentration of Sulphur-content at various time(continue)

Sampling time	% Sulphur Content (%wt)									
	Experimental error (1)					Experimental error (2)				
	Batch 1	Batch 2	Batch 3	Average	%Error	Batch 1	Batch 2	Batch 3	Average	%Error
60	0.175	0.176	0.173	0.175 ± 0.002	1.718	0.247	0.245	0.239	0.244 ± 0.004	3.283
61	0.176	0.176	0.174	0.175 ± 0.001	1.141	0.246	0.245	0.240	0.244 ± 0.003	2.462
62	0.176	0.174	0.174	0.175 ± 0.001	1.145	0.248	0.245	0.240	0.244 ± 0.004	3.274
63	0.177	0.175	0.173	0.175 ± 0.002	2.286	0.249	0.245	0.241	0.245 ± 0.004	3.265
64	0.177	0.175	0.173	0.175 ± 0.002	2.286	0.250	0.245	0.243	0.246 ± 0.004	2.846
65	0.177	0.175	0.173	0.175 ± 0.002	2.286	0.251	0.246	0.243	0.247 ± 0.004	3.243
66	0.177	0.173	0.174	0.175 ± 0.002	2.290	0.252	0.246	0.245	0.248 ± 0.004	2.826
67	0.181	0.174	0.174	0.176 ± 0.004	3.970	0.252	0.248	0.245	0.248 ± 0.004	2.819
68	0.179	0.174	0.174	0.176 ± 0.003	2.846	0.251	0.248	0.246	0.248 ± 0.003	2.013
69	0.179	0.174	0.174	0.176 ± 0.003	2.846	0.251	0.248	0.246	0.248 ± 0.003	2.013
70	0.179	0.174	0.175	0.176 ± 0.003	2.841	0.251	0.249	0.246	0.249 ± 0.003	2.011
71	0.179	0.175	0.175	0.176 ± 0.002	2.268	0.251	0.250	0.246	0.249 ± 0.003	2.008
72	0.178	0.176	0.174	0.176 ± 0.002	2.273	0.252	0.250	0.248	0.25 ± 0.002	1.600
73	0.178	0.178	0.176	0.177 ± 0.001	1.128	0.254	0.251	0.248	0.251 ± 0.003	2.390
74	0.180	0.181	0.176	0.179 ± 0.003	2.793	0.254	0.253	0.248	0.252 ± 0.003	2.384
75	0.180	0.180	0.176	0.179 ± 0.002	2.239	0.255	0.251	0.250	0.252 ± 0.003	1.984
76	0.182	0.181	0.176	0.180 ± 0.003	3.340	0.256	0.251	0.251	0.253 ± 0.003	1.979
77	0.179	0.187	0.177	0.181 ± 0.005	5.525	0.256	0.254	0.251	0.254 ± 0.003	1.971
78	0.179	0.185	0.181	0.182 ± 0.003	3.303	0.256	0.255	0.252	0.254 ± 0.002	1.573
79	0.180	0.184	0.180	0.181 ± 0.002	2.206	0.257	0.255	0.253	0.255 ± 0.002	1.569
80	0.180	0.185	0.182	0.182 ± 0.003	2.742	0.257	0.256	0.252	0.255 ± 0.003	1.961
81	0.181	0.185	0.183	0.183 ± 0.002	2.186	0.256	0.257	0.253	0.255 ± 0.002	1.567
82	0.181	0.184	0.185	0.183 ± 0.002	2.182	0.258	0.258	0.254	0.257 ± 0.002	1.558
83	0.181	0.184	0.187	0.184 ± 0.003	3.261	0.258	0.258	0.256	0.257 ± 0.001	0.777
84	0.181	0.185	0.189	0.185 ± 0.004	4.324	0.260	0.258	0.256	0.258 ± 0.002	1.550
85	0.181	0.185	0.189	0.185 ± 0.004	4.324	0.260	0.259	0.257	0.259 ± 0.002	1.160
86	0.182	0.185	0.189	0.185 ± 0.004	3.777	0.261	0.259	0.258	0.259 ± 0.002	1.157
87	0.182	0.185	0.189	0.185 ± 0.004	3.777	0.261	0.258	0.258	0.259 ± 0.002	1.158
88	0.183	0.184	0.188	0.185 ± 0.003	2.703	0.262	0.260	0.258	0.26 ± 0.002	1.538
89	0.183	0.184	0.188	0.185 ± 0.003	2.703	0.262	0.260	0.257	0.26 ± 0.003	1.926

Table 5.3 Variation of concentration of Sulphur-content at various time(continue)

Sampling time	% Sulphur Content (%wt)									
	Experimental error (1)					Experimental error (2)				
	Batch 1	Batch 2	Batch 3	Average	%Error	Batch 1	Batch 2	Batch 3	Average	%Error
90	0.184	0.185	0.189	0.186 ± 0.003	2.688	0.262	0.260	0.257	0.26 ± 0.003	1.926
91	0.183	0.183	0.190	0.185 ± 0.004	3.777	0.263	0.260	0.258	0.26 ± 0.003	1.921
92	0.184	0.187	0.189	0.187 ± 0.003	2.679	0.263	0.260	0.259	0.261 ± 0.002	1.535
93	0.183	0.187	0.189	0.186 ± 0.003	3.220	0.263	0.260	0.260	0.261 ± 0.002	1.149
94	0.185	0.189	0.190	0.188 ± 0.003	2.660	0.264	0.260	0.261	0.262 ± 0.002	1.529
95	0.187	0.190	0.191	0.189 ± 0.002	2.113	0.264	0.260	0.260	0.261 ± 0.002	1.531
96	0.189	0.189	0.190	0.189 ± 0.001	0.528	0.263	0.260	0.260	0.261 ± 0.002	1.149
97	0.189	0.190	0.191	0.190 ± 0.001	1.053	0.264	0.260	0.261	0.262 ± 0.002	1.529
98	0.190	0.192	0.191	0.191 ± 0.001	1.047	0.264	0.260	0.261	0.262 ± 0.002	1.529
99	0.189	0.193	0.192	0.191 ± 0.002	2.091	0.263	0.260	0.262	0.262 ± 0.002	1.146
100	0.190	0.193	0.191	0.191 ± 0.002	1.568	0.263	0.260	0.263	0.262 ± 0.002	1.145
101	0.190	0.192	0.194	0.192 ± 0.002	2.083	0.263	0.260	0.262	0.262 ± 0.002	1.146
102	0.189	0.193	0.194	0.192 ± 0.003	2.604	0.263	0.260	0.262	0.262 ± 0.002	1.146
103	0.189	0.193	0.192	0.191 ± 0.002	2.091	0.263	0.260	0.263	0.262 ± 0.002	1.145
104	0.189	0.193	0.192	0.191 ± 0.002	2.091	0.263	0.260	0.263	0.262 ± 0.002	1.145
105	0.189	0.193	0.190	0.191 ± 0.002	2.098	0.263	0.260	0.266	0.263 ± 0.003	2.281
106	0.190	0.192	0.192	0.191 ± 0.001	1.045	0.264	0.260	0.266	0.263 ± 0.003	2.278
107	0.192	0.193	0.194	0.193 ± 0.001	1.036	0.263	0.260	0.267	0.263 ± 0.004	2.658
108	0.192	0.193	0.194	0.193 ± 0.001	1.036	0.263	0.260	0.266	0.263 ± 0.003	2.281
109	0.192	0.193	0.194	0.193 ± 0.001	1.036	0.263	0.260	0.265	0.263 ± 0.003	1.904
110	0.191	0.193	0.192	0.192 ± 0.001	1.042	0.263	0.260	0.265	0.263 ± 0.003	1.904
111	0.192	0.193	0.193	0.193 ± 0.001	0.519	0.263	0.260	0.266	0.263 ± 0.003	2.281
112	0.192	0.193	0.193	0.193 ± 0.001	0.519	0.263	0.260	0.265	0.263 ± 0.003	1.904
113	0.192	0.192	0.193	0.192 ± 0.001	0.520	0.263	0.260	0.267	0.263 ± 0.004	2.658
114	0.191	0.193	0.193	0.192 ± 0.001	1.040	0.263	0.260	0.265	0.263 ± 0.003	1.904
115	0.191	0.193	0.192	0.192 ± 0.001	1.042	0.263	0.260	0.265	0.263 ± 0.003	1.904
116	0.191	0.192	0.192	0.192 ± 0.001	0.522	0.263	0.260	0.265	0.263 ± 0.003	1.904
117	0.190	0.193	0.193	0.192 ± 0.002	1.563	0.263	0.260	0.265	0.263 ± 0.003	1.904
118	0.190	0.193	0.193	0.192 ± 0.002	1.563	0.263	0.260	0.265	0.263 ± 0.003	1.904
119	0.190	0.193	0.193	0.192 ± 0.002	1.563	0.263	0.260	0.265	0.263 ± 0.003	1.904
120	0.191	0.193	0.193	0.192 ± 0.001	1.040	0.263	0.260	0.265	0.263 ± 0.003	1.904

### 5.3 Determination of required mixing time

The mixing time was measured by examining the time history of concentration fluctuation following the release of percent Sulphur that do not dissolve. In these experiments the mixing time,  $t_m$  was defined as the time required reaching a specified level of uniformity in a mixing system.

In order to obtain the percent of Sulphur content from the experiment data show the result are presented as a plot of time of sampling versus the percentage Sulphur content as shown in Figure 5.1

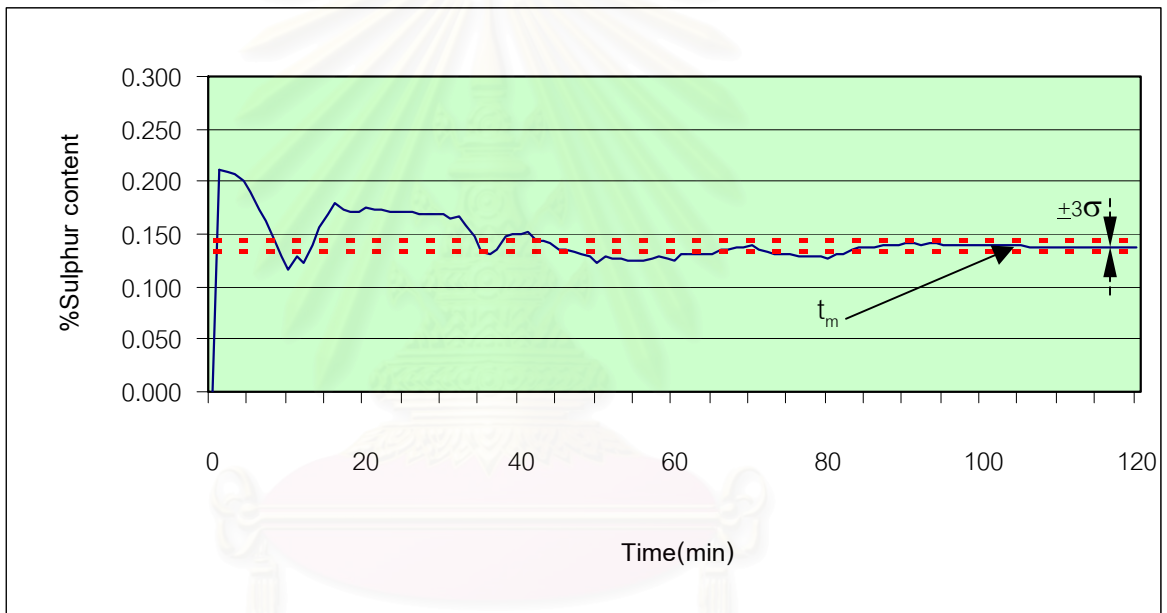


Figure 5.1 Mixing curve of %Sulphur content versus time of sampling

From this figure, only one data was obtained at each measuring interval and this data was the required mixing time. From visual examination, it was observed that the homogeneous solution was obtain after the Sulphur content reached the steady mean volume

AIChE Equipment Testing Procedure<sup>(1)</sup> (Dry Solid, Paste and Dough Mixing Equipment, 1979) recommended the selection of the mixing time of the curing-

rate additive as the time required to achieve concentration of mixture within acceptable limit or to reach composition equilibrium by statistical analyzing.

Pipop T. and Tawatchai S. studied the mixing by used tracer technique. The time of mixing,  $t_m$ , was selected as the time to achieve variation of concentration of radioactive material to become less than  $\pm 3$  time of standard deviation as shown in Figure 3.6.

In this study, it was found that the distribution data of Sulphur content gave the result in the manner similar to tracer response curve by Pipop T. The time to achieve variation of concentration of mixture from their mean within  $\pm 3$  time of their standard deviation was the proper condition to determine the required mixing time.

For sample calculation, from Figure 5.1 as mixing time curve of a experiment (Base oil type 3, viscosity of mineral oil @100°C  $\approx$  32.0 cSt, temperature of base oil at 100°C, 400 rpm of impeller speed), by statistical analysis as shown in Appendix F. The mean and Standard deviation of equilibrium were 0.166 and 0.0006 respectively. The upper and lower limits were 0.168 and 0.164 respectively. The required mixing time as defined in chapter 3.4 was determined by the shortest time in which the variation of Sulphur content are within those limits. The shortest time in this case 103 min. The other result was calculated with the same method as summarized in Table 5.4 and the other graphs are shown in Appendix G.

The variation of homogeneous solution may be caused by two main factors. Firstly, the nature absorption UV of Sulphur was randomly absorption UV so the detectors will variation value. Secondly, the limitation of sensitivity of measuring instrument must be considered.

From Table 5.4, the results were plotted between rotational speed of impeller and mixing time as shown in Figure 5.2

Table 5.4 Mixing Time from experiments

Viscosity base oil @100°C (cSt)	Temperature (deg C)	Speed (rpm)	Mixing time, $t_m$ (min)
Base oil Type 1 (4.7)	80	300	120
		400	112
		500	111
	100	300	115
		400	105
		500	92
	120	300	14
		400	13
		500	11
Base oil type 2 (11.2)	80	300	116
		400	103
		500	102
	100	300	99
		400	96
		500	92
	120	300	46
		400	37
		500	31
Base oil type 3 (32.0)	80	300	107
		400	103
		500	95
	100	300	92
		400	89
		500	75
	120	300	81
		400	78
		500	72

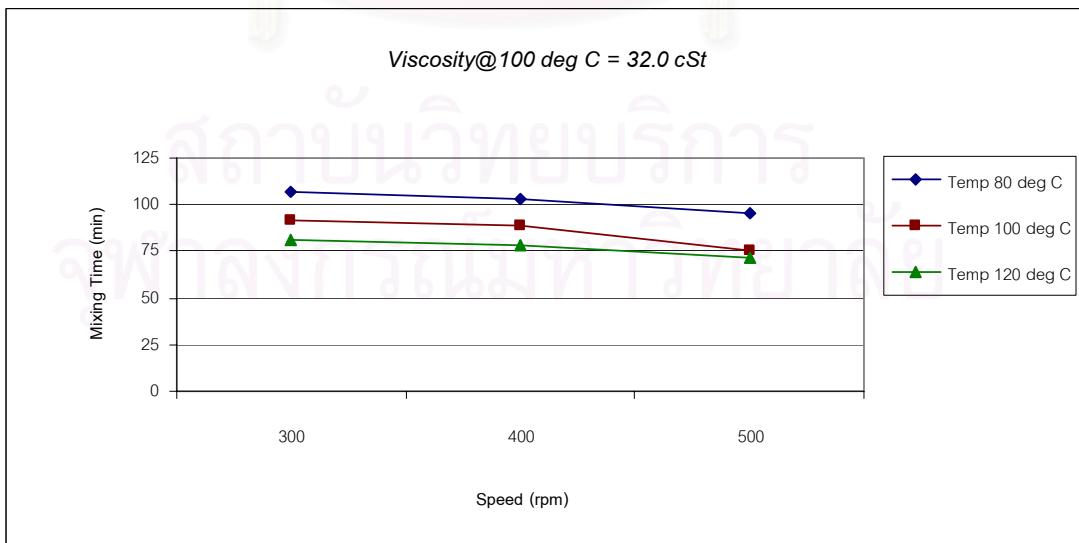
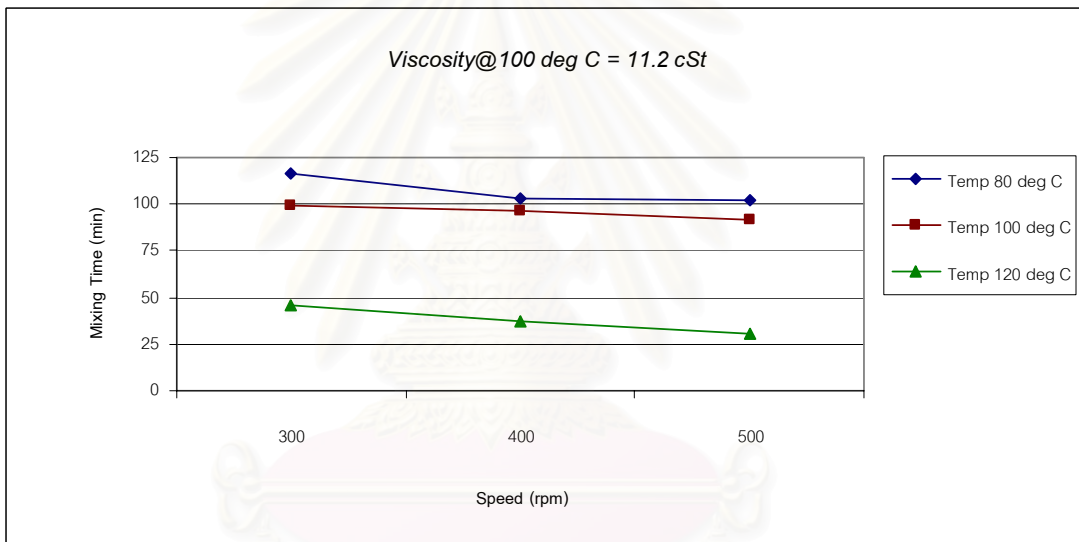
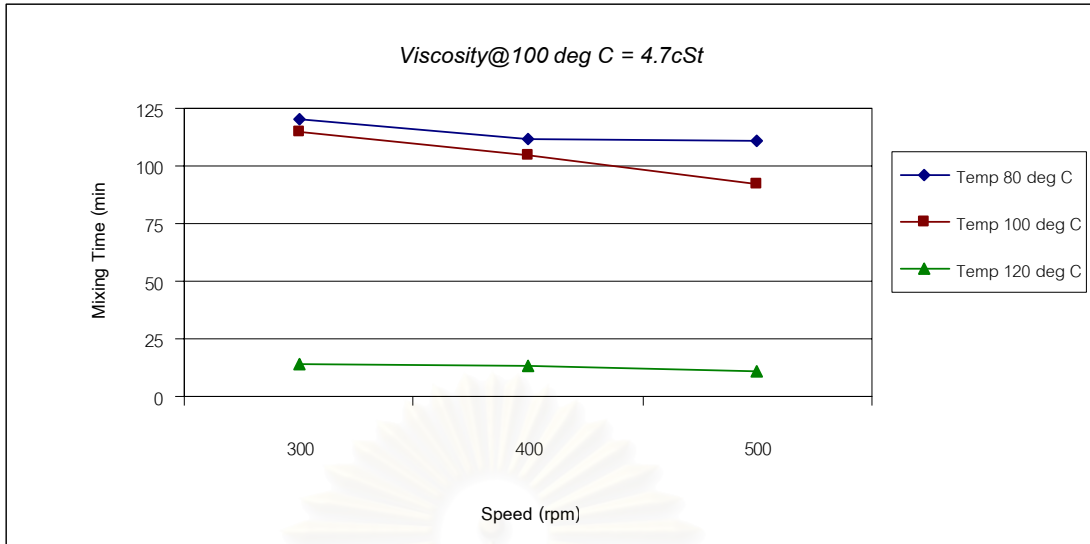


Figure 5.2 Relationship between mixing time and rotation speed

### 5.3.1 Effect of rotational speed of impeller

Plotted of mixing time,  $t_m$ , versus inversely rotational speed ( $N$ ) was prepared on normal co-ordinates. All correlation line with a slope was obtained as Figure 5.2. The curve of decrease of mixing time accompanied by an increase in agitator speed has two distinct points. When liquid have lower viscosity show agitator speed have small effect than higher viscosity. The result can explain by relatively of stirrer speeds, since the particles are not suspended at low speed and are easily broken at high speed.

### 5.3.2 Effect of viscosity

A plot of shear stress against shear rate was shown in Figure 3.9, for high viscosity liquids, viscosities decrease with increasing shear rate. The power relation as described in Chapter 3.2 was changed to:

$$P \propto (N^2 D_i^3 \mu) \quad (5.4)$$

In general, for constant configuration and speed

$$P \propto \mu \quad (5.5)$$

Figure 3.9, plot of apparent viscosity versus shear rate will be a straight line. For the above explanation, it can be confirmed with Metzner's and Otto's experiments.

Metzner and Otto<sup>(18)</sup> were the first investigators to propose a useful procedure for prediction of power consumption in non-Newtonian fluids using fundamental viscometer data. It was found that the fluid motion in the vicinity of the impeller could be characterized by relating shear rate to impeller speed as follow:

$$\text{Shear rate} \propto N \quad (5.6)$$

From figure 3.14 and the above equation, it can be concluded that viscosity can be expressed a function of speed.

The data of experiments plotted in Figure 5.3 showed that a longer mixing time obtained at temperature 120 °C by 32 cSt. viscosity of mineral oil than by 4.7 cSt. viscosity of mineral oil. This is caused from the higher difference in the viscosity and lower shear rate of mineral oil. For the lower temperature viscosity have lower effect to mixing time.

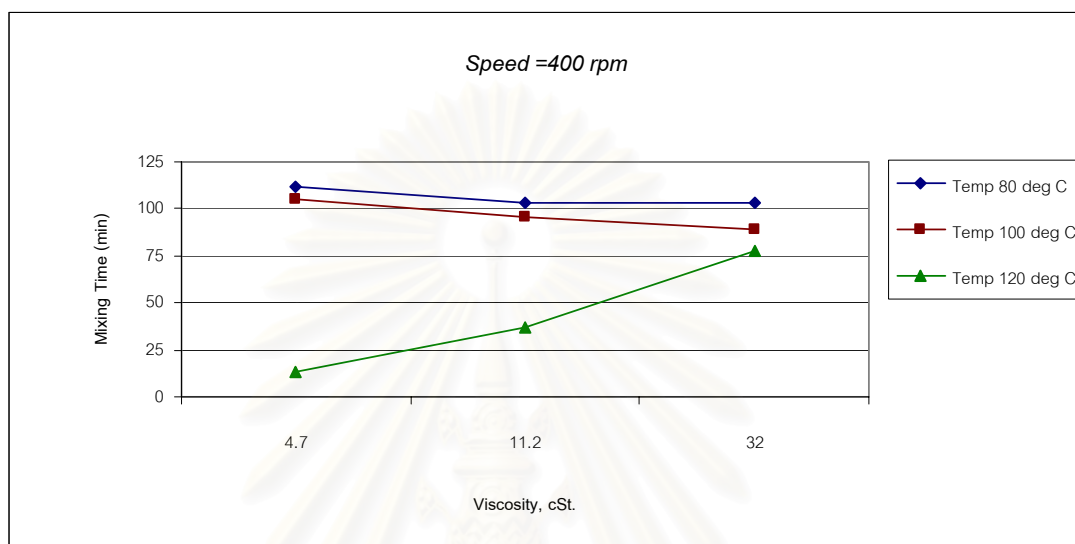


Figure 5.3 Relationship between mixing time and viscosity

The difference in viscosities of the components and final product provide the “driving force”, the greater the differences in viscosities of the components, the more horsepower is required. Also, the higher the final product viscosity, the more power required.

### 5.3.3 Effect of temperature

Dissolution is accompanied by a temperature change proportional to heat of solution of the respective substance and this phenomena can utilized by determine the mixing time. Plotted of mixing time,  $t_m$ , versus rotational speed in Figure 5.2 showed that longer mixing time can obtain at lower temperature. For temperature 80 to 100 °C show the mixing time very difference from temperature at 120 °C. This is caused from Sulphur can transform from Rhombic Sulphur to Monoclinic Sulphur at transition point (95.6 °C) and the process is slow, so that it is possible rapid heating of



Sulphur to attain the melting point at 119 °C. From the reason Sulphur can melting at high temperature so liquid Sulphur are easily to dissolve in mineral oil.

## 5.4 Dimensional analysis

### 5.4.1 Mixing time required under each temperature

Since it is neither convenient nor economical to experiment with industrial size units to get optimal process conditions, it is desirable to have a scale up technique to predict the mixing time of industrial scale. From this experiment, it can be observed that the mixing time may be related with operating conditions, such as: rotational speed, vessel geometry and any physical properties of mineral oil. So, the many variables of any physical properties and operating conditions can be conveniently related by using suitable dimensionless parameters and combined them to arrive at two parameters that related all the variables involved in the mixing process.

For geometrically similar tanks; such as: constant impeller, tank geometric ratios, location of addition, the dimensionless mixing time should depend on:

$$Nt_m = f \left\{ \frac{\rho N D_i^2}{\mu}, \frac{\Delta \rho g H_l}{\rho N^2 D_i^2}, \frac{\mu_2}{\mu_1}, \frac{H_l}{D_i} \right\} \quad (5.7)$$

Where the second dimensionless group on the right-hand side is Richardson number,  $R_r$ , and  $H_l$  is depth of liquid in tank.

This general dependence can be simplified to:

$$\tau = Nt_m = f \{ R_e, R_i \} \quad (5.8)$$

Richardson number is related to the density differences in two-component or two stratified layers. For this study, the experiments were conducted in multi-component system. So Reynolds number is a more proper dimensionless for mixing time to use.

The Reynolds number,  $Re$ , defined as  $\frac{\rho D_i^2 N}{\mu}$ , which represents the ratio of inertial forces to viscous forces. The dimensionless of mixing time,  $\tau$ , defined as  $Nt_m$ , represents the ratio of blending time to a the period of agitator revolution.

With the logarithmic plot of  $\tau$  against  $Re$ , it can be observed that a straight line correlation fits the data points. A linear correlation analysis provides the following equation,

For temperature 80°C

- Base oil type 1  $\tau = 9.401 Re + (6.433 \times 10^3)$  (5.9)

- Base oil type 2  $\tau = 2.891 Re + (9.933 \times 10^3)$  (5.10)

- Base oil type 3  $\tau = 1.721 Re + (9.467 \times 10^3)$  (5.11)

For temperature 100°C

- Base oil type 1  $\tau = 9.298 Re + (1.783 \times 10^4)$  (5.12)

- Base oil type 2  $\tau = 7.905 Re + (5.433 \times 10^3)$  (5.13)

- Base oil type 3  $\tau = 2.987 Re + (1.376 \times 10^4)$  (5.14)

For temperature 120°C

- Base oil type 1  $\tau = 4.192 Re + (2.367 \times 10^3)$  (5.15)

- Base oil type 2  $\tau = 2.760 Re + (1.130 \times 10^4)$  (5.16)

- Base oil type 3  $\tau = 15.113 Re + (7.097 \times 10^3)$  (5.17)

Information on the mixing time from this experiment and from this correlation should be compared as shown in Appendix J, showing relative errors between the mixing time from experimental and calculated mixing time from the correlation. The maximum relative error is  $\pm 3\%$ .

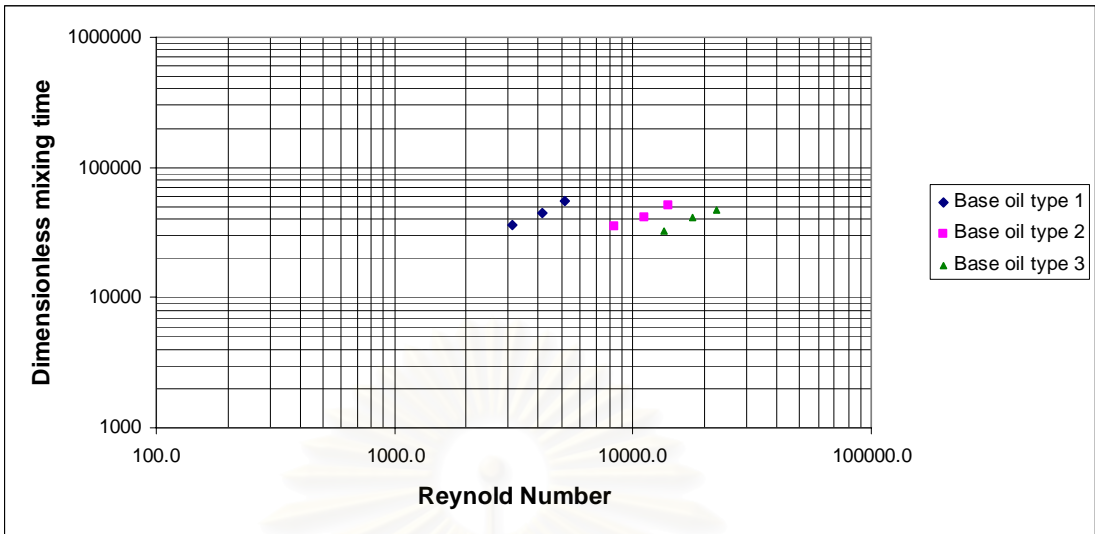


Figure 5.4 Dimensionless mixing time plotted linearly against Reynolds number of Sulphur dissolution in mineral oil at temperature 80 °C

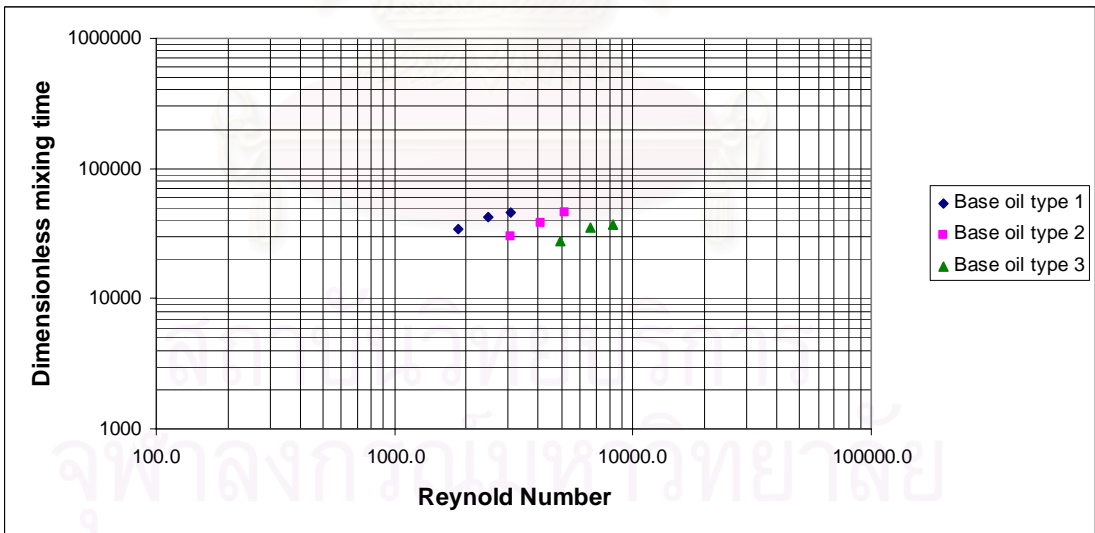


Figure 5.5 Dimensionless mixing time plotted linearly against Reynolds number of Sulphur dissolution in mineral oil at temperature 100 °C

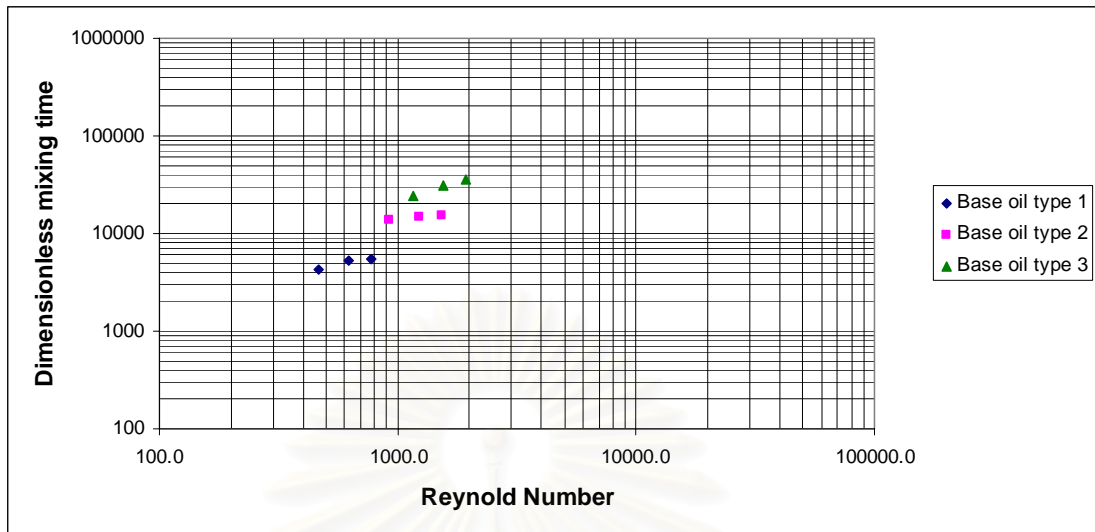


Figure 5.6 Dimensionless mixing time plotted linearly against Reynolds number of Sulphur dissolution in mineral oil at temperature 120 °C

#### 5.4.2 The Solid-Liquid Dissolution Rate Correlation

Dissolution rate coefficient of the solid-liquid system is expressed by the equation

$$Sh_T = r Re_a^p Sc^q$$

Where a dimensionless group,  $Sh_T$  is a function of other dimensionless group,  $Re_a, Sc$ . In this paper, the solid-liquid dissolution rate correlations for high speed shear mixer are obtained as show in Table 5.5. By analytical calculation technique (Show in Appendix K, L and M), constant  $r$  for each system is obtained as in Table 5.6

Table 5.5 Sulphur powder Dissolution Rate Correlation for High Speed Shear Mixer

Experimental Systems	Correlation
System I	$Sh = r Re_a^{0.378} Sc^{0.454}$ $0.465 \times 10^3 < Re_a < 5.185 \times 10^3$ $8.217 \times 10^{11} < Sc < 374.155 \times 10^{11}$
System II	$Sh = r Re_a^{0.378} Sc^{0.454}$ $0.924 \times 10^3 < Re_a < 14.007 \times 10^3$ $2.879 \times 10^{11} < Sc < 163.819 \times 10^{11}$
System III	$Sh = r Re_a^{0.378} Sc^{0.454}$ $1.161 \times 10^3 < Re_a < 22.371 \times 10^3$ $1.711 \times 10^{11} < Sc < 95.897 \times 10^{11}$

Where: System I is 0.3 wt % of Sulphur in Mineral oil at 80°C  
System II is 0.3 wt % of Sulphur in Mineral oil at 100°C  
System III is 0.3 wt % of Sulphur in Mineral oil at 120°C

Table 5.6 Values of r for this experimental system

Experimental Systems	$r$	Range of $Re_a \times 10^3$	Range of $Sc \times 10^{11}$
System I	$5.407 \times 10^4$	0.465-5.185	8.217-374.155
System II	$7.359 \times 10^4$	0.924-14.007	2.879-163.819
System III	$1.101 \times 10^5$	1.161-22.371	1.711-95.897

Where: System I is 0.3 wt % of Sulphur in Mineral oil at 80°C  
System II is 0.3 wt % of Sulphur in Mineral oil at 100°C  
System III is 0.3 wt % of Sulphur in Mineral oil at 120°C

For calculation correlation as in Table 5.5 and 5.6, the solutions show in Appendix L and Appendix M

## 5.5 Comparison with Correlation with Other Investigation

The exponents of Reynolds number and of Schmidt number of the dissolution rate correlation can reported by various investigators are summarized in Table 5.7.

In table 5.7, the exponents obtained for various variables of the other researches are compared to this work. It is seen that the range of exponent value for High Speed Shear Mixer

Table 5.7 Comparison of the exponents obtained variables for various

Author	Exponent of $Re$	Exponent of $Sc$	System of agitator utilize
Barker and Trey bal. Humphrey	0.833	0.5	Standard 6 blade turbine
Van Ness	0.87	0.5	Standard 6 blade turbine
Johnson and Chen- Jung Huang	0.71	0.5	Standard 6 blade turbine
Hixson and Baum	1.40	0.5	Standard 6 blade turbine
Askew	0.55	0.3	Standard 6 blade turbine
Beckmann	0.667	0.3	Marine
Keey and Glen	0.8	0.5	Paddle
Saetun	0.8	0.197	Paddle
This work	0.378	0.454	High Speed Shear Mixer

Exponent value of  $Re$  from this work is different from other investigators because of this work used different type of agitator. The exponent of  $Re$  in this study is smaller than other researches can cause by the increase in diffusion resistance from high viscosity of base oil. The exponent of  $Re$  varies widely by the difference in agitation condition and species of solid. The rotor-stator mixer in this work has characteristics of a high speed, high shear mixing head. It is used for application for fast disintegration, dispersion, solubilization and homogenization. These characteristic were different from propellers that are employed with low shear and no particle disintegration. For the

pattern flow of high speed shear mixer agitator as show in Figure 5.7. These figure show parts of solid particle are piled on the vessel bottom. Particle in the upper layer of the pile contact well with the liquid, but the particles inside the pile are in rest, and the turbulence of the liquid does not reach the surface of the inner particles. By increase agitator speed, the number of the piled particles diminishes and the effective solid-liquid contact area increases until all the solid particles are finally fluidized.



Figure 5.7 The formation and circulation pattern of High speed shear mixer agitator

#### 5.5.1 The influence of Reynolds number

Under isothermal condition for any particular solid-liquid system,  $\mu$ ,  $\rho$  and  $D_v$  are constant. The value of Reynolds number depends on the rotation speeds of the agitator,  $N$

The number and parameter of the dimensionless group proposed by various, researches and different therefore, it is difficult to carry out complete comparisons. However, there are three important variables; the Reynolds number, Schmidt number, and type of impeller which are in common.

To determine the influence of the High speed shear mixer type at various temperatures, the data from table 5.8-5.10 were plotted as shown in Figures 5.8-5.10. Each line present at constant temperature and having average slope at 0.378, which is the exponent of the Reynolds number in the correlation.

Table 5.8  $Re_a$  vs.  $Sh$  of 0.3% Sulphur powder in base oil at 80 °C

Base oil	Speed	$Re_a \times 10^3$	$Sh \times 10^{10}$
Type 1 (4.7 cSt)	300	3.111	0.576
	400	4.148	0.587
	500	5.185	0.619
Type 2 (11.2 cSt)	300	1.855	1.282
	400	2.474	1.304
	500	3.092	1.390
Type 3 (32.0 cSt)	300	0.465	4.002
	400	0.620	4.383
	500	0.775	4.499

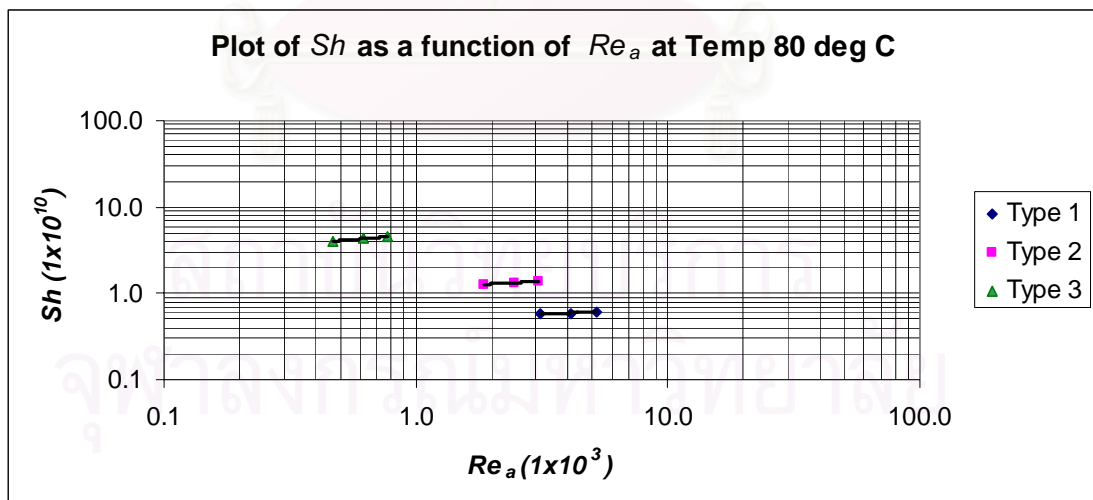
Table 5.9  $Re_a$  vs.  $Sh$  of 0.3% Sulphur powder in base oil at 100 °C

Base oil	Speed	$Re_a \times 10^3$	$Sh \times 10^{10}$
Type 1 (4.7 cSt)	300	8.404	0.601
	400	11.205	0.633
	500	14.007	0.719
Type 2 (11.2 cSt)	300	3.093	1.408
	400	4.124	1.493
	500	5.155	1.543
Type 3 (32.0 cSt)	300	0.924	4.565
	400	1.232	4.794
	500	1.540	5.232



Table 5.10  $Re_a$  vs.  $Sh$  of 0.3% Sulphur powder in base oil at 120 °C

Base oil	Speed	$Re_a \times 10^3$	$Sh \times 10^{10}$
Type 1 (4.7 cSt)	300	13.422	0.539
	400	17.897	0.580
	500	22.371	0.601
Type 2 (11.2 cSt)	300	4.972	1.287
	400	6.630	1.366
	500	8.287	1.490
Type 3 (32.0 cSt)	300	1.161	4.368
	400	1.548	4.617
	500	1.935	4.718

Figure 5.8 Plot of  $Sh$  as function of  $Re_a$  at 80 °C

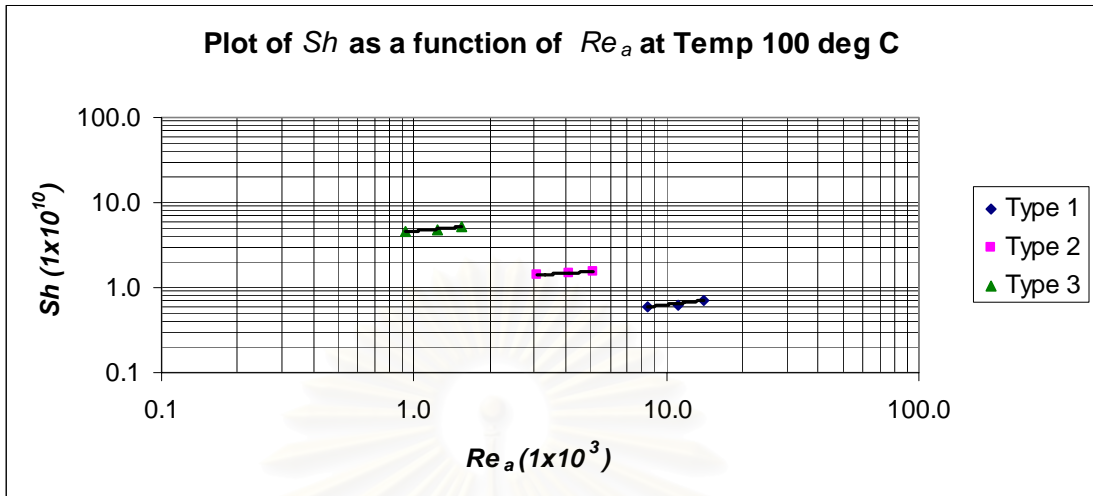


Figure 5.9 Plot of  $Sh$  as function of  $Re_a$  at 100°C

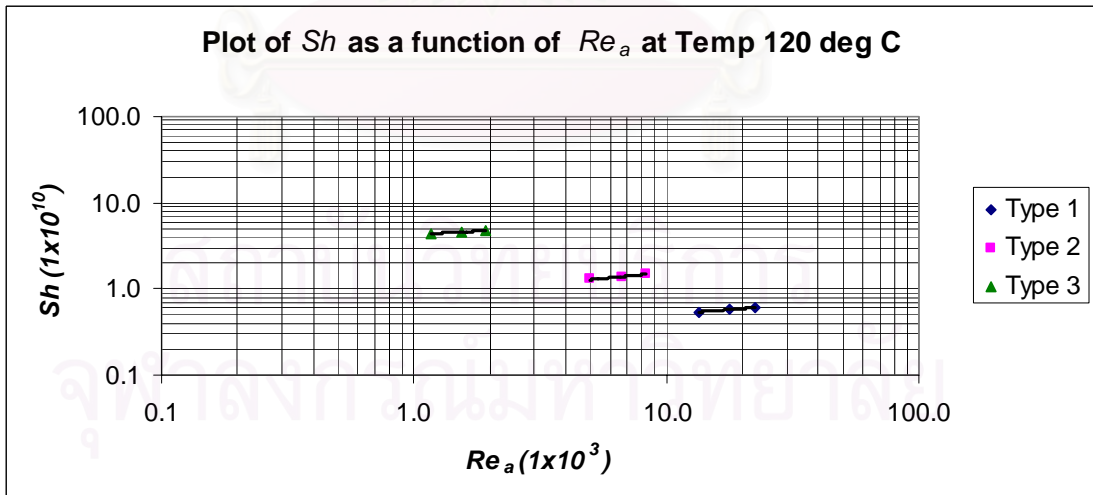


Figure 5.10 Plot of  $Sh$  as function of  $Re_a$  at 120°C

### 5.5.2 Influence of Schmidt Number

To determine the influence of the Schmidt number on solid dissolution the temperature were varied which the Reynolds number was kept constant. Data from the table 5.11- 5.13 are plotted as shown in Figures 5.11-5.13. From these Figure found that lines are parallel, having average slope of 0.454 which is the exponent of Schmidt number of correlation in this experiment

Table 5.11  $Sc$  vs.  $Sh$  of 0.3% Sulphur powder in base oil at 300 rpm

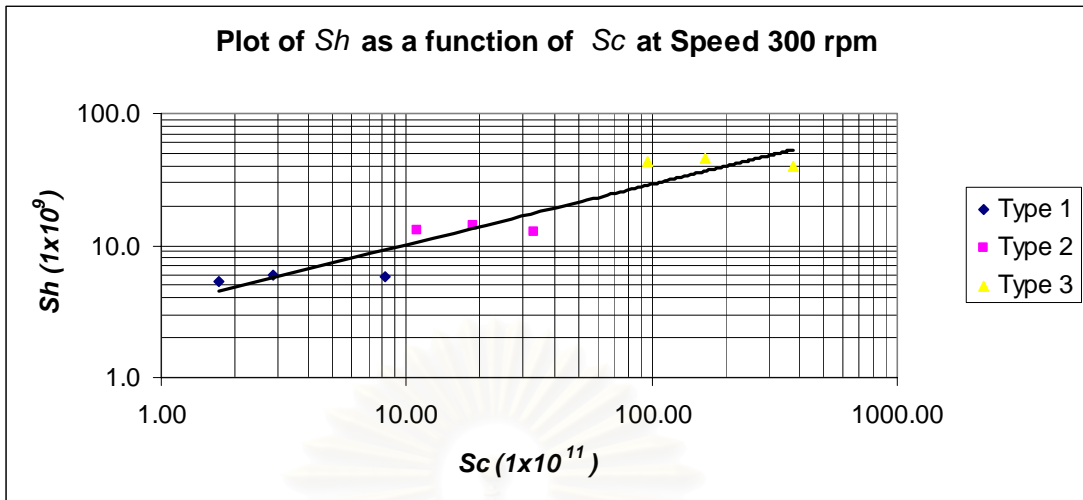
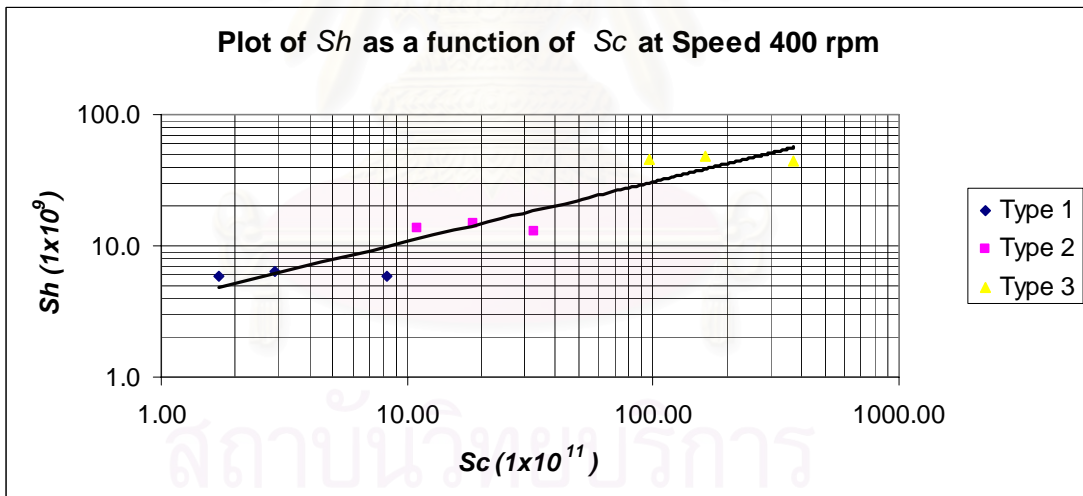
Base oil	Temperature ( $^{\circ}C$ )	$Sc \times 10^{11}$	$Sh \times 10^9$
Type 1 (4.7 cSt)	80	8.217	41.459
	100	2.879	34.668
	120	1.711	3.524
Type 2 (11.2 cSt)	80	32.837	99.565
	100	18.640	74.133
	120	11.005	34.964
Type 3 (32.0 cSt)	80	374.155	316.790
	100	163.819	226.764
	120	95.897	166.678

Table 5.12  $Sc$  vs.  $Sh$  of 0.3% Sulphur powder in base oil at 400 rpm

Base oil	Temperature ( $^{\circ}C$ )	$Sc \times 10^{11}$	$Sh \times 10^9$
Type 1 (4.7 cSt)	80	8.217	5.872
	100	2.879	6.328
	120	1.711	5.804
Type 2 (11.2 cSt)	80	32.837	13.043
	100	18.640	14.925
	120	11.005	13.663
Type 3 (32.0 cSt)	80	374.155	43.833
	100	163.819	47.943
	120	95.897	46.169

Table 5.13  $Sc$  vs.  $Sh$  of 0.3% Sulphur powder in base oil at 500 rpm

Base oil	Temperature ( $^{\circ}C$ )	$Sc \times 10^{11}$	$Sh \times 10^9$
Type 1 (4.7 cSt)	80	8.217	6.186
	100	2.879	7.192
	120	1.711	6.005
Type 2 (11.2 cSt)	80	32.837	13.897
	100	18.640	15.433
	120	11.005	14.896
Type 3 (32.0 cSt)	80	374.155	44.988
	100	163.819	52.321
	120	95.897	47.183

Figure 5.11 Plot of  $Sh$  as function of  $Sc$  at speed 300 rpmFigure 5.12 Plot of  $Sh$  as function of  $Sc$  at speed 400 rpm

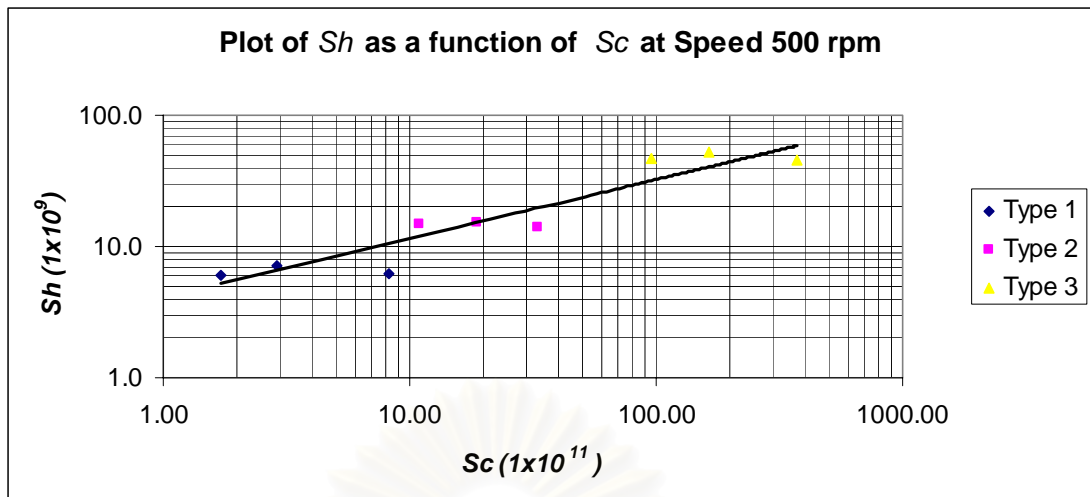


Figure 5.13 Plot of  $Sh$  as function of  $Sc$  at speed 500 rpm

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## CHAPTER VI

### CONCLUSION

#### 6.1 CONCLUSION

An investigation for mixing time required for complete dissolution of sulphur in lubricating oil by measurement of sulphur content in agitated batch mixer using UV Spectroscopy gave the results which could be conclude about the mixing system as follow:

1. The required mixing time was found to be inversely to the rotational speed of impeller.

2. The required mixing time of sulphur in lubricating oil depends on the phase of sulphur and viscosity of lubricating oils. At high rotational speed, turbulent diffusion will have increasing influence on movement of lubricating oil. Temperature exhibited effect significant on structure of sulphur by changing it's to liquid phase. At high temperature (higher than 100 °C) it was found that required mixing time became shorter than that of lower temperature.

3. Two dimensionless numbers, mixing time,  $\tau$ , as defined  $Nt_m$ , and Reynolds number,  $Re$  defined as  $Re = \frac{D_i^2 \rho N}{\mu}$ , including vessel geometry relative to impeller and physical properties of base oils, have a linear correlation for each temperature, which can be expressed as

For temperature 80°C

- Base oil type 1       $\tau = 9.401 Re + (6.433 \times 10^3)$

- Base oil type 2       $\tau = 2.891 Re + (9.933 \times 10^3)$

- Base oil type 3       $\tau = 1.721 Re + (9.467 \times 10^3)$

For temperature 100°C

- Base oil type 1  $\tau = 9.298 \text{ Re} + (1.783 \times 10^4)$

- Base oil type 2  $\tau = 7.905 \text{ Re} + (5.433 \times 10^3)$

- Base oil type 3  $\tau = 2.987 \text{ Re} + (1.376 \times 10^4)$

For temperature 120°C

- Base oil type 1  $\tau = 4.192 \text{ Re} + (2.367 \times 10^3)$

- Base oil type 2  $\tau = 2.760 \text{ Re} + (1.130 \times 10^4)$

- Base oil type 3  $\tau = 15.113 \text{ Re} + (7.097 \times 10^3)$

4. This investigation showed that the dissolution rate coefficient of sulphur powder in lubricating oil by used High speed shear mixer could be correlated by the following dimensionless equation.

$$Sh = r Re_a^{0.378} Sc^{0.454}$$

when  $0.465 \times 10^3 < Re_a < 22.371 \times 10^3$

$$1.711 \times 10^{11} < Sc < 374.155 \times 10^{11}$$

The constant  $r$  depends on solid liquid system. Although  $r$  in each system is different, but the exponent of the Reynolds number and Schmidt number are the same for every system for similar impeller type. The constant  $r$  in the dimensionless equation depends on the solid liquid system, temperature, mixing condition and the characteristics of liquid solution.

5. The results of this work are difference from other investigators because the agitating system, the solid liquid system used and the experimental condition are not the same. Our results did not differ much from the others, and showed the same trend of dependency on the speed of agitation and the temperature of the system. Therefore, it can be concluded that there is no general correlation for the solid liquid dissolution.



## 6.2 RECOMMENDATIONS

From model development in the present experiments, the limitation of performance equation 5.9-5.11 as follows:

1. It was only applied in lubricating oil as additive element is sulphur powder. For other additives that have same particle size can not adjust in a similar manner from this study because of specific physical property of sulphur were different from other additives.

2. It can be applied in the range of viscosities@100°C: 4.7 - 32.0 cSt. (approximately) and range of temperature 80,100 and 120 °C

For the correlation that develop by used dimensionless, the correlation varies from case to case and there is no general correlation for the dissolution rate coefficient could have resulted not only from the Reynolds number, Schmidt number and temperature of liquid system but also from other factors such as solid liquid system, diameter of solid particles, level of agitation, shape of particles, density difference between solid and liquid system, diffusivity, viscosity etc. The result above suggests that to arrive at a more generalized equation of solid liquid dissolution; further studies should be carried out, such as determination of the influence of other factors mentioned and the power consumption.

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APPENDIES

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APPENDIX A  
STANDARD TEST METHOD

A-1

Standard Test Method for  
Kinematics Viscosity of Transparent and Opaque Liquid  
ASTM D 445

1. Scope

1.1 This test method covers the determination of the kinematics viscosity of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematics viscosity by the density of liquid.

1.2 This test method intended primarily for application to liquids for which the shear stress and shear rates are proportional.

1.3 This test method depends on the behavior of the sample, and ideally the coefficient of viscosity should be independent of the rate of shear (this is commonly called Newtonian flow behavior). If, however, the coefficient of viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters.

2. Apparatus

2.1 *Viscometers* of the glass capillary type, calibrated and capable of measuring kinematics viscosity within the limit of precision. Viscometer list in table A-1.1 meet these requirements.

2.2 *Viscometer Holders* to enable the viscometer to be suspended in a similar position as when calibrated. The proper alignment of vertical parts may be confirmed by using a plumb line.

2.3 *Viscometer Thermostat and Bath* - Any transparent liquid or vapor bath may be used, provided that it is of sufficient depth that at no time during the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

2.3.1 The temperature control must be such that for the range from 15 -100 °C ( 60-212 °F) the temperature of the bath medium does not vary by more than 0.01 °C ( 0.02 °F) over the range of the viscometers, or between the position of each viscometer, or at the location of the thermometer. For temperature outside this range, the variation must not exceed 0.03 °C (0.05 °F)

2.4 *Temperature-Measuring Device* - Standardized liquid in glass temperatures (Table A-1.2) of an accuracy after correction of 0.02 °C ( 0.04 °F) can be used, or any other thermometric device of equal or better accuracy. If standardized liquid in glass thermometers are used, it is recommended (but not required) that two thermometers be used, They must agree within 0.04 °C ( 0.07 °F)

Table A-1.1 Viscometer Type

Viscometer Identification	Range, cSt (mm <sup>2</sup> /s) <sup>A</sup>
1. Oswald type for Transparent liquids :	
1. Cannon Fenske routine <sup>C</sup>	0.5 <sup>B</sup> - 20000
2. Zeitfuchs <sup>C</sup>	0.6 - 3000
3. SIL <sup>C</sup>	0.6 - 10000
4. Cannon-manning Semi-micro <sup>C</sup>	0.4 - 20000
5. BS/IP U-Tube <sup>D</sup>	0.9 <sup>B</sup> - 10000
6. BS/IP U-tube Miniature <sup>D</sup>	0.2 - 100
7. Pinkevitch	0.6 <sup>B</sup> - 17000
2. Suspended-Level Types for Transparent Liquids :	
1. Ubbelohde <sup>C</sup>	0.3 <sup>B</sup> - 100000
2. FitzSimons <sup>C</sup>	0.6 - 1200
3. Atlantic <sup>C</sup>	0.75 <sup>B</sup> - 5000
4. Cannon-Ubbelohde, Cannon-Ubbelohde dilution <sup>C</sup>	0.5 <sup>B</sup> - 100000
5. Cannon-Ubbelohde semi-micro <sup>C</sup>	0.4 - 20000
6. BS/IP- Suspended Level <sup>D</sup>	3.5 <sup>B</sup> - 100000
7. BS/IP- Suspended Level, Shortened Form <sup>D</sup>	1.05 <sup>B</sup> - 10000
8. BS/IP Miniature Suspended Level <sup>D</sup>	0.6 - 3000
3. Reverse-flow Types for Transparent and Opaque Liquids:	
1. Zeitfuchs Cross-Arm <sup>C</sup>	0.6 - 100000
2. Cannon-Fenske Opaque <sup>C</sup>	0.4 - 20000
3. Lantz-Zeitfuchs <sup>C</sup>	60 - 100000
4. BS/IP U-tube Reverse flow <sup>D</sup>	0.6 - 300000

<sup>A</sup> Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these

viscometers are designed for a flow time in excess of 200 sec. except where noted in Table 3.

<sup>B</sup> In each of these series, the minimum flow time for the viscometers with the lowest constant exceeds 200 sec.

<sup>C</sup> Specifications and operating instructions for these viscometers have been assembled in Specifications and Operating instructions D 446.

<sup>D</sup> Specifications for these are given in appendixes to IP 71.

Table A-1.2 Kinematics Viscosity Test Thermometer<sup>A</sup>

Test Temperature <sup>B</sup>		Scale Error <sup>B</sup>		Thermometer	Number
°F	°C			ASTM <sup>C</sup>	IP <sup>D</sup>
-65	-53.9			74F, C+	69F, C
-60 to -35	-51 to -35			43F	65F, C
-40	-40			73F, C	68F, C
-15	-26.1			126F, C	71F, C
	-20			127C	99C
0	-17.8			72F, C+	67F, C
32	0			128F, C	33F, C
68 and 70	20 and 21.1			44F, C	29F, C
77	25			45F, C	30F, C
86	30			118F, C	
100	37.8			28F, C	31F, C
	40			120C	92C
122	50			46F, C	66F, C
130	54.4			29F, C+	34F, C
140	60			47F, C	35F, C
	80				100C
180	82.2			48F, C	90F, C
200	93.3			129F, C	36F, C
210 and 212	98.9 and 100			30F	32F, C
	100			121C	
275	135			110F, C	

<sup>A</sup> The smallest graduation of the Fahrenheit thermometers is 0.1 °F and for the Celsius thermometers is 0.05 °C

except for ASTM 43F and 65F for which it is 0.2 °F.

<sup>B</sup> Scale error for the Fahrenheit thermometers is not to exceed + 0.2 °F (except for ASTM 110F which is + 0.3 °F)

<sup>C</sup> Complete construction detail is given in Specification E 1.

<sup>D</sup> Complete construction detail is given in Part 1 of IP Standards for Petroleum and its Products.

+ Editorially corrected.



2.5 *Timing Device*- Any timing device may be used provided that the readings can be taken with a discrimination of 0.2 sec. or better, and that it has an accuracy within + 0.07 % when tested over intervals of 15 min.

2.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

### 3. General procedure for Kinematics Viscosity

3.1 The specific details of operation vary for the different types of viscometers list in Table A-1.1. The operating in conditions for the different types of viscometers are given in Specification D 446.

3.2 Maintain the bath at the test temperature within limits given in 2.3.1 taking account of the precaution of the correction supplied on certifications of calibration.

3.2.1 In order to obtain the most reliable temperature measurement, it is recommended that two thermometer with valid calibration certificates be used. The thermometer should be held in an upright position under the condition of immersion as when calibrated. They should be used with a lens assembly giving about five times magnification and with should be arranged eliminate paralaxation.

3.3 Select a clean dry, calibrated viscometer having a covering the estimated kinematics viscosity (that is, a capillary for a very viscous liquid and a narrower for a more fluid liquid). The flow time should not less than 200 sec., or as noted in Table A-1.3.

Table A-1.3. Minimum flow times

Note- All sizes of all viscometers listed in Specification D 446 are designed for a flow time in excess off 200 sec., except as listed below. The minimum flow times for the six "BS/IP" viscometers listed in Table 1 are given in the appendices to IP 71.

Viscometer Identification	ASTM Size	Minimum flow time, sec.
Cannon-Fenske routine	25	250
Ubbelohde	0	300
Atlantic	0C	250
Cannon-Ubbelohde, Cannon-Ubbelohde dilution	25	250

3.3.1 When the test temperature is below the dew point, is loosely packed drying tubes to the open ends of the viscometer. The drying tubes must fit the design of the viscometer and not restrict the flow of the sample by ensures created in the instrument. Carefully flush the moisten near from the viscometer by applying vacuum to one of drying tubes. Finally, before placing the viscometer in the draw up the sample into the working capillary and drying bulb and allow to drain back as an additional against moisture condensing or freezing on the tubes.

3.3.2 Viscometers used for silicone fluids, fluoro-carbons, the other liquids which are difficult to remove by the use of cleaning agent, should be reserved for the exclusive use of the fluids except when calibrating. Such viscometers should be subjected to calibration checks at frequent inter. The solvent washings from these viscometers should not used for the cleaning of other viscometers.

#### 4. Cleaning of Viscometer

4.1 Between successive determinations, clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing the slow stream of filtered dry air through the viscometer for 2 min. or until the last trace of solvent is removed.

4.2 Periodically clean the viscometer with chromic acid cleaning solution for at least twelve hours to remove residual traces of organic deposits, nonchromium-containing, strongly oxidizing acid cleaning solutions may be substituted so as to avoid disposal problems of chromium-containing solutions. Rinse thoroughly with distilled water followed by acetone, and dry with clean, dry air. Inorganic deposits may be removed by hydrochloric acid treatment before use of cleaning acid, particularly if barium salts are suspected. The use of alkaline cleaning solutions is not recommended as this can enlarge the working capillary and necessitate recalibration.



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A-2  
Standard Test method for  
Density, Relative Density (Specific Gravity), or API Gravity  
of Crude Petroleum and Liquid Petroleum Products by Hydrometer method  
ASTM D 1298

## 1. Scope

This practice covers the laboratory determination, using a glass hydrometer, of the density, relative density (specific gravity), or API gravity of crude petroleum, petroleum products, or mixtures of petroleum and nonpetroleum products normally handled as liquids, and having a Reid vapor pressure of (179 kPa) 26 lb or less. Values are measured on a hydrometer at convenient temperatures, readings of density being reduced to 15 °C, and readings of relative density and API gravity to 60 °F, by means of international standard tables. By means of these same tables, values determined in any one of the three systems of measurement are convertible to equivalent values in either of the other two so that measurements may be made in the units of local convenience.

## 2. Apparatus

2.1 *Hydrometers*, glass, graduated in units of density, relative density, or API gravity as required, determining to ASTM specification or specification of IP standards

2.2 *Thermometers*, having ranges shown in table 2 and determining to specifications of the American Society for Testing and Material or the Institute of Petroleum.

2.3 *Hydrometer cylinder*, clear glass, plastic, or other for convenience in pouring, the cylinder may have a rim. The inside diameter of the cylinder shall be at 25

mm. greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such appropriate hydrometer floats in the sample with at 25 mm. clearance between the bottom of the hydrometer and the bottom of the cylinder.

2.4 *Constant-temperature bath*, for use when the nature of the sample requires a test temperature much above or below room temperature.

### 3. Procedure

3.1 Adjust the temperature of sample, bring the hydrometer cylinder and thermometer to approximately the same temperature as the sample to be tested.

3.2 Transfer the sample to a clean hydrometer cylinder without spashing, to avoid the formation of air bubbles, and to reduce to a minimum evaporation of the lower boiling constituents of more volatile samples. Transfer highly volatile samples to the cylinder by water displacement. Remove any air bubbles formed, after they have collected on the surface of sample, by touching them with a piece of clean filter paper before inserting the hydrometer.

3.3 Place the cylinder containing the sample in a vertical position in a location free from air currents. Ensure that the temperature of the sample does not change appreciably during the time necessary to complete the test; during this period, the temperature of the surrounding medium should not change more than 2°C (5°F). When testing at temperatures much above or below room temperature, a constant temperature bath may be necessary to avoid excessive temperature changes.

3.4 Lower the hydrometer gently into the sample. Take care to avoid wetting the stem above the level to which it will be immersed in the liquid. Continuously stir the sample with the thermometer, Taking care that the mercury thread is kept fully immersed and that the stem of the hydrometer is not wetted above the immersion level. As soon as a steady reading is obtained, record the temperature of the sample to nearest 0.25°C (0.5°F) and then remove the temperature.

3.5 Depress the hydrometer about two scale divisions into the liquid, and then release it. The remainder of the stem of the hydrometer, which is above the level of the liquid, must be kept dry since unnecessary liquid on stem affects the reading obtained. With samples of low viscosity, impart a slight spin to the hydrometer on releasing to assist in bringing it to rest, floating freely away from the walls of the cylinder. Allow sufficient time for the hydrometer to come to rest, and for all air bubbles to come to the surface. This is particularly necessary in the case of more viscous samples.

3.6 When the hydrometer has come to rest, floating freely away from the walls of the cylinder, estimate the hydrometer scale reading to the nearest 0.0001 relative density (specific gravity) or density or 0.05°API. The correction hydrometer reading is that point on the hydrometer scale which the principle surface of the liquid cuts the scale. Determine this point by placing the eye slightly below as seen as a distorted ellipse, appears to become a straight cutting the hydrometer scale. (see figure A-2.1.)

3.7 With an opaque liquid take a reading by observed with the eye slightly above the plane of the surface of liquid, the point on the hydrometer scale to which sample rises. This reading, at the top of the minimize requires correction since hydrometers are calibrated to read at the principal surface of the liquid. The correction of the particular hydrometer in use may be determined observing the maximum height above the principal surface of the liquid to which oil rises on the hydrometer scale. The hydrometer in question is immersed in a transparent having a surface tension similar to that of the sample to test. (see figure A-2.2)

3.8 Immediately after observing the hydrometer value, again cautiously stir the sample with the thermometer keeping the mercury thread fully immersed. Recorded temperature of the sample to the nearest 0.2°C (0.5°F). Should this temperature differ from the previous reading by more than 0.5°C (1°F), repeat the hydrometer test then thermometer observations until the temperature comes stable within 0.5°C (1°F).

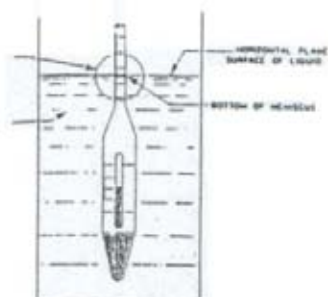


Figure A-2.1 Hydrometer scale reading for Transparent liquids

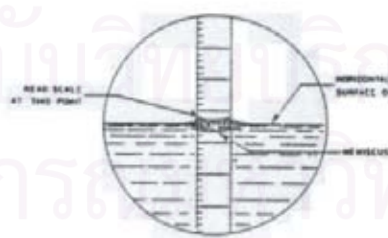
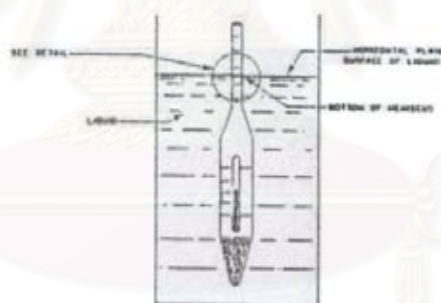


Figure A-2.2 Hydrometer scale reading for Opaque fluids.

## APPENDIX B

### BASIC OIL SPECIFICATION

Product : BASIC OIL-1

Table B-1 Specification of BASIC OIL-1

Properties	Unit	Method	Results
Appearance	-	Visual	Cl. & Br.
Density @ 15 Deg C	kg/l.	ASTM D 1298	0.86-0.88
Color, ASTM	-	ASTM D 1500	1.5 Max
Flash point, PMcc	Deg. C	ASTM D 93	200 Min
Viscosity @ 40 Deg C	cSt.	ASTM D 445	22-27
Viscosity @ 100 Deg C	cSt.	ASTM D 445	4.4-4.9
Viscosity index	-	ASTM D 2270	95 Min
Crackle test	-	-	pass
Pour point	Deg. C	ASTM D 97	-15 Max

BASIC OIL-1 is a paraffinic base oil refined from crude oil for use as a blending component of lubricating and greases.



Product : BASIC OIL-2

Table B-2 Specification of BASIC OIL-2

Properties	Unit	Method	Results
Appearance	-	Visual	Cl. & Br.
Density @ 15 Deg C	kg/l.	ASTM D 1298	0.885-0.895
Color, ASTM	-	ASTM D 1500	3.5 Max
Flash point, PMcc	Deg. C	ASTM D 93	228 Min
Viscosity @ 40 Deg C	cSt.	ASTM D 445	90-110
Viscosity @ 100 Deg C	cSt.	ASTM D 445	10.7-11.8
Viscosity index	-	ASTM D 2270	95 Min
Crackle test	-	-	pass
Pour point	Deg. C	ASTM D 97	-9 Max

BASIC OIL-2 is a paraffinic base oil refined from crude oil for use as a blending component of lubricating and greases.

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Product : BASIC OIL-3

Table B-3 Specification of BASIC OIL-3

Properties	Unit	Method	Results
Appearance	-	Visual	Cl. & Br.
Density @ 15 Deg C	kg/l.	ASTM D 1298	0.896-0.920
Color, ASTM	-	ASTM D 1500	5.5 Max
Flash point, PMcc	Deg. C	ASTM D 93	267 Min
Viscosity @ 40 Deg C	cSt.	ASTM D 445	440-550
Viscosity @ 100 Deg C	cSt.	ASTM D 445	30.5-33.5
Viscosity index	-	ASTM D 2270	95 Min
Crackle test	-	-	pass
Pour point	Deg. C	ASTM D 97	-9 Max

BASIC OIL-3 is a paraffinic base oil refined from crude oil for use as a blending component of lubricating and greases.

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## APPENDIX C

### ADDITIVE SPECIFICATION

**Table C-1** Specification of Sulphur

Properties	Unit	Method	Result
Appearance @ 20 Deg C	-	Visual	Yellow powder or crystals
Sulphur content	% w	AAS	99.5 –100.0
Particle size	% wt passing 80 mesh	Sieve Test	99.6
	% wt passing 100 mesh		99.4
	% wt passing 200 mesh		99.2
	% wt passing 325 mesh		99.5

Application : Lubricant Additive

Function : E P agent

Description : Sulphur powder

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## APPENDIX D

### PARTICLER SIZE DISTRIBUTION OF SULPHUR POWDER

#### D.1 Scope

This test describe the determination of the particle size distribution of Sulphur powder. The sample is placed on the uppermost of a stack of metal wire cloth, after which the sieve is mechanically shaken. The amount of sample retained on each sieve and that collected in the pan is weighed, from wich particle size distribution is calculated.

#### D.2 Apparatus

2.1 Wire Cloth Sieves, diameter 203 mm, according to ASTM E11, wich lid and pan. Apertures as required according to the particle size distribution of the product tested.

2.2 Mechanical Sieve Shaker, designed to accommodate 203 mm (or 200 mm) sieves, simulating a rotating and tapping motion as used in manual sieving.

2.3 Brush, soft nylon or sable, width 12 mm.

2.4 Sample Scoop, cylindrical.

2.5 Microscope, magnification 10 to 50 times.

#### D.3 Sampling

Correct sampling is of the highest importance and is the basic requirement for reliable sieve analysis. Great care must be exercised to obtain samples which are truly representative of the batch of lot being tested. The biggest cause of inconsistencies in test results is improper sampling, thereby obtaining a sample which

does not truly represent the material under test. Therefore, once a sampling procedure is established, this same procedure should always be followed.

#### D.4 Cleaning Sieves

Clean the wire cloth sieves by gently brushing the underside with the nylon or sable brush and blowing gently with a stream of clean dry air.

#### D.5 Procedure

5.1 Select four or more sieves of the series which will cover the expected particle size range. Weigh each of the sieves and the pan to the nearest 0.01 g and stack the sieves on the pan in order of increasing apertures with the coarsest sieve on top. Weigh approx. 50 g of sample, to the nearest 0.01 g, and transfer to the uppermost sieve. Cover with the lid and clamp the stack of sieves to the shaker. Connect an earth wire to the stack of sieves.

5.2 Start the shaker and allow to vibrate until sieving is complete. If necessary, check the completeness of the sieving as described.

5.3 Reweigh each sieve and the pan, to the nearest 0.01 g

Note 1. Relatively small fractions may be weighted more accurately after transferring them to dishes or weighing paper.

Note 2. Vibration time depends on the number of sieves used, their sizes and the flow properties of the material under test. For an assembly of five to seven sieves, the finest of which has an aperture of 45 micron, a vibration time of about 10 minutes (apertures wider than 1 mm) a two minute vibration period will be sufficient.

## 6. Calculation

6.1 Calculate the sieve fractions retained relation on the each sieve and that collected in the pan, by means of one of the following equations.

$$\text{Sieve Fraction, \% (m/m)} = \frac{m_i}{m_s} \times 100$$

where:

$m_i$  = mass of fraction retained on the given sieve or that collected in pan, g,

$m_s$  = mass of sample taken, g.

Calculate the cumulative sieve fraction retained on the sieves, to the nearest 0.1%(m/m), starting with the fraction retained on the sieve with the largest aperture.

6.2 Plot the cumulative sieve fractions retained on the sieves in %(m/m) against the aperture size, in micron, on logarithmic probability paper, as shown in Fig D-1

## 7. Reporting

The particle size distribution of Sulphur powder relating to the data show in Table D-1, D-2 tabulating the size fractions retained on each sieve to the nearest 0.1%(m/m), against the limiting sieve apertures.

Table D-1 Size of particle data

Particle size range, micron	Sieve Fraction %(m/m)
177 to 210	0.4
149 to 177	0.2
74 to 149	0.2
44 to 74	3.7
37 to 44	95.5

Table D-2 Relative percentage frequency distribution: tabular calculation of mean

Particle size range (micron)	Interval $Dx$	Average size $x$	Percentage in range $d\phi$	Percentage per micron $d\phi/dx$	$xd\phi$
177 to 210	33	193.5	0.4	0.012	77.4
149 to 177	28	163.0	0.2	0.007	32.6
74 to 149	75	111.5	0.2	0.003	22.3
44 to 74	30	59.0	3.7	0.123	218.3
37 to 44	7	40.5	95.5	13.643	3867.75
				$\sum xd\phi$	4218.35
Mean size				$= \frac{\sum xd\phi}{\sum d\phi}$	42.18

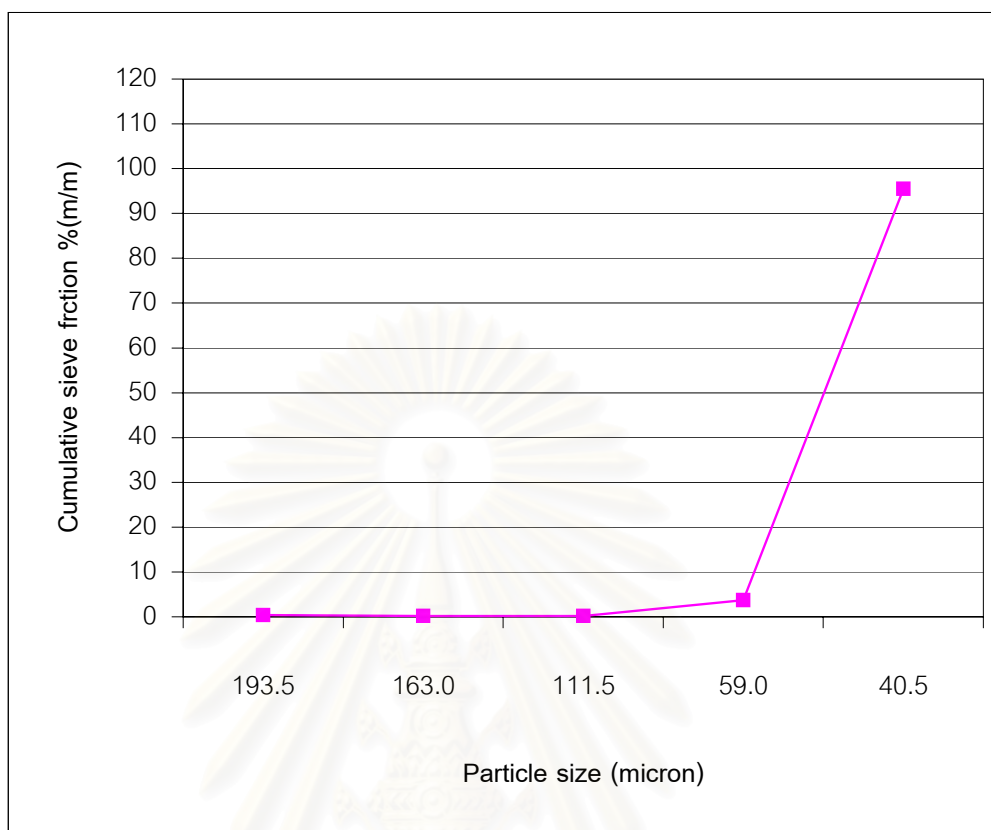


Figure D-1. Typical cumulative particle size distribution curve of Sulphur powder

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## APPENDIX E

### VISCOSITY AND DENSITY OF LUBRICATING OILS

Table E-1 Viscosity of lubricating oil at various temperature

Temperature (°C)	Viscosity of Lubricating Oils , Poise		
	@ 100 °C = 4.7 cSt	@ 100 °C = 11.2 cSt	@ 100 °C = 32.0 cSt
40	24.257	84.520	448.280
50	17.198	53.118	289.379
60	14.493	42.423	174.282
70	11.558	24.101	117.992
80	9.614	16.059	62.879
90	7.531	12.066	42.180
100	3.501	9.476	31.125
110	2.650	7.276	29.077
120	2.156	5.769	24.331

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Table E-2 Density of lubricating oil at various temperature

Temperature (°C)	Density of Lubricating Oils , g / cm <sup>3</sup>		
	@ 100 °C = 4.7 cSt	@ 100 °C = 11.2 cSt	@ 100 °C = 32.0 cSt
40	0.8903	0.8861	0.8718
50	0.8832	0.8793	0.8647
60	0.8762	0.8724	0.8575
70	0.8692	0.8655	0.8503
80	0.8621	0.8587	0.8431
90	0.8551	0.8517	0.8359
100	0.8480	0.8448	0.8288
110	0.8411	0.8378	0.8216
120	0.8340	0.8268	0.8144

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## APPENDIX F

### CALCULATION SHAPE FACTOR OF SULPHUR POWDER

The particle shapes of sulphur in this experiment were determined by the defined shape factor is the circularity. To find the circularity of this experiment was collected data by random sampling and measurement by microscopy. These measurements were read and counted area by using a scale of graph. All data of calculation can be shown as in table F-1.

Table F-1 Circularity of Sulphur particles

Projected area of particle (Unit)	Actual particle perimeter (Unit)	Perimeter of a particle having the same area as the projected area of particle (Unit)	Circularity
44	23.0	23.5	1.02
53	24.0	25.8	1.08
60	28.0	27.5	0.98
68	28.0	29.2	1.04
90	32.0	33.6	1.05
90	34.0	33.6	0.99
99	39.0	35.3	0.90
106	36.0	36.5	1.01
114	38.0	37.9	1.00
118	39.0	38.5	0.99
120	39.0	38.8	1.00
120	39.0	38.8	1.00
121	37.0	39.0	1.05

Projected area of particle (Unit)	Actual particle perimeter (Unit)	Perimeter of a particle having the same area as the projected area of particle (Unit)	Circularity
122	36.0	39.2	1.09
124	42.0	39.5	0.94
124	42.0	39.5	0.94
132	40.0	40.7	1.02
134	40.0	41.0	1.03
136	46.0	41.3	0.90
137	46.0	41.5	0.90
143	43.0	42.4	0.99
148	49.0	43.1	0.88
156	52.0	44.3	0.85
168	44.0	46.0	1.04
176	44.0	47.0	1.07
176	45.0	47.0	1.05
177	51.0	47.2	0.92
177	44.0	47.2	1.07
177	51.0	47.2	0.92
184	52.0	48.1	0.92
189	51.0	48.7	0.96
189	59.0	48.7	0.83
189	51.0	48.7	0.96
194	59.0	49.4	0.84
195	59.0	49.5	0.84
198	59.0	49.9	0.85
201	51.0	50.3	0.99

Projected area of particle (Unit)	Actual particle perimeter (Unit)	Perimeter of a particle having the same area as the projected area of particle (Unit)	Circularity
201	62.0	50.3	0.81
205	66.0	50.8	0.77
205	55.0	50.8	0.92
209	55.0	51.3	0.93
210	55.0	51.4	0.93
211	55.0	51.5	0.94
211	53.0	51.5	0.97
212	53.0	51.6	0.97
214	54.0	51.9	0.96
215	56.0	52.0	0.93
215	56.0	52.0	0.93
216	54.0	52.1	0.96
218	61.0	52.4	0.86
218	60.0	52.4	0.87
218	59.0	52.4	0.89
218	51.0	52.4	1.03
218	51.0	52.4	1.03
222	54.0	52.8	0.98
224	52.0	53.1	1.02
224	53.0	53.1	1.00
224	54.0	53.1	0.98
224	55.0	53.1	0.96
224	59.0	53.1	0.90
229	57.0	53.7	0.94

Projected area of particle (Unit)	Actual particle perimeter (Unit)	Perimeter of a particle having the same area as the projected area of particle (Unit)	Circularity
232	55.0	54.0	0.98
235	56.0	54.4	0.97
237	54.0	54.6	1.01
237	62.0	54.6	0.88
237	61.0	54.6	0.89
241	66.0	55.0	0.83
247	61.0	55.7	0.91
247	62.0	55.7	0.90
249	52.0	55.9	1.08
249	62.0	55.9	0.90
249	65.0	55.9	0.86
249	66.0	55.9	0.85
253	59.0	56.4	0.96
253	55.0	56.4	1.03
253	60.0	56.4	0.94
254	62.0	56.5	0.91
259	61.0	57.1	0.94
261	54.0	57.3	1.06
261	61.0	57.3	0.94
267	59.0	57.9	0.98
283	58.0	59.6	1.03
283	58.0	59.6	1.03
286	65.0	60.0	0.92
289	72.0	60.3	0.84

Projected area of particle (Unit)	Actual particle perimeter (Unit)	Perimeter of a particle having the same area as the projected area of particle (Unit)	Circularity
302	66.0	61.6	0.93
302	66.0	61.6	0.93
302	59.0	61.6	1.04
302	58.0	61.6	1.06
315	58.0	62.9	1.08
315	58.0	62.9	1.08
330	66.0	64.4	0.98
330	66.0	64.4	0.98
332	59.0	64.6	1.09
332	71.0	64.6	0.91
336	71.0	65.0	0.92
336	74.0	65.0	0.88
357	69.0	67.0	0.97
357	62.0	67.0	1.08
361	72.0	67.4	0.94
361	71.0	67.4	0.95

Average Circularity 0.96

Standard Deviation 0.07

Max 1.09

Min 0.77

## APPENDIX G

### DATA FROM EXPERIMENTS

Data were obtained by using UV spectrophotometer for measurement Sulphur content in lubricating oils. Data are shown the value between sampling time and concentration of Sulphur and plotted sampling times versus concentration of Sulphur.

All of data are shown as follows:

Table G-1 (a) Data from experiments of Base oil 1 viscosity @ 100 °C 4.7 cSt

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1	0.295	0.225	0.159	0.179	0.195	0.156	0.291	0.292	0.290
2	0.294	0.221	0.154	0.177	0.196	0.156	0.290	0.290	0.282
3	0.294	0.218	0.149	0.175	0.195	0.156	0.290	0.288	0.274
4	0.294	0.213	0.141	0.171	0.195	0.155	0.289	0.286	0.273
5	0.293	0.205	0.138	0.171	0.194	0.155	0.289	0.284	0.273
6	0.292	0.201	0.131	0.170	0.195	0.155	0.288	0.281	0.274
7	0.293	0.170	0.129	0.169	0.195	0.154	0.287	0.280	0.275
8	0.291	0.141	0.125	0.165	0.195	0.155	0.287	0.279	0.275
9	0.291	0.113	0.123	0.168	0.195	0.154	0.287	0.279	0.274
10	0.200	0.104	0.127	0.169	0.195	0.154	0.287	0.279	0.274
11	0.190	0.180	0.121	0.166	0.195	0.157	0.287	0.279	0.275
12	0.190	0.111	0.117	0.164	0.197	0.158	0.287	0.279	0.275
13	0.186	0.128	0.114	0.163	0.197	0.159	0.287	0.276	0.276
14	0.181	0.136	0.105	0.163	0.197	0.161	0.286	0.275	0.276
15	0.181	0.159	0.101	0.161	0.197	0.163	0.284	0.275	0.276
16	0.180	0.182	0.098	0.160	0.197	0.169	0.284	0.274	0.276
17	0.180	0.193	0.093	0.159	0.198	0.172	0.284	0.275	0.276
18	0.179	0.200	0.090	0.158	0.198	0.176	0.285	0.275	0.276
19	0.180	0.204	0.090	0.159	0.199	0.180	0.284	0.275	0.276



Table G-1 (a) Data from experiments of Base oil viscosity @ 100 °C 4.7 cSt (continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
20	0.179	0.206	0.089	0.160	0.200	0.182	0.284	0.275	0.276
21	0.179	0.210	0.092	0.161	0.200	0.182	0.284	0.274	0.276
22	0.181	0.199	0.099	0.162	0.201	0.182	0.284	0.274	0.276
23	0.186	0.182	0.102	0.161	0.201	0.181	0.284	0.273	0.276
24	0.190	0.174	0.105	0.163	0.201	0.180	0.284	0.272	0.276
25	0.190	0.166	0.109	0.165	0.202	0.180	0.284	0.272	0.276
26	0.188	0.155	0.111	0.166	0.202	0.180	0.284	0.271	0.276
27	0.187	0.153	0.113	0.167	0.202	0.180	0.284	0.271	0.276
28	0.190	0.147	0.114	0.168	0.203	0.179	0.284	0.270	0.276
29	0.200	0.131	0.117	0.167	0.203	0.179	0.284	0.270	0.276
30	0.201	0.122	0.119	0.167	0.204	0.179	0.284	0.270	0.276
31	0.200	0.119	0.113	0.167	0.201	0.180	0.284	0.270	0.276
32	0.195	0.111	0.112	0.168	0.198	0.180	0.284	0.270	0.276
33	0.178	0.125	0.109	0.170	0.191	0.181	0.284	0.271	0.276
34	0.169	0.124	0.105	0.170	0.186	0.181	0.284	0.271	0.276
35	0.146	0.127	0.101	0.167	0.179	0.180	0.284	0.272	0.276
36	0.133	0.135	0.099	0.169	0.174	0.181	0.284	0.273	0.276
37	0.130	0.141	0.096	0.170	0.173	0.180	0.284	0.274	0.277
38	0.123	0.148	0.094	0.170	0.171	0.180	0.284	0.274	0.277
39	0.121	0.150	0.091	0.169	0.169	0.180	0.284	0.275	0.277
40	0.119	0.151	0.089	0.169	0.169	0.181	0.284	0.275	0.277
41	0.144	0.162	0.087	0.167	0.167	0.179	0.284	0.275	0.277
42	0.155	0.165	0.089	0.170	0.166	0.180	0.284	0.275	0.277
43	0.190	0.171	0.100	0.169	0.165	0.180	0.284	0.275	0.277
44	0.201	0.182	0.101	0.168	0.164	0.179	0.284	0.275	0.277
45	0.213	0.195	0.110	0.168	0.161	0.178	0.284	0.275	0.277
46	0.209	0.201	0.119	0.166	0.164	0.178	0.284	0.275	0.277
47	0.220	0.205	0.124	0.166	0.170	0.177	0.284	0.275	0.277
48	0.223	0.209	0.133	0.165	0.173	0.177	0.284	0.275	0.277
49	0.225	0.212	0.141	0.166	0.176	0.175	0.285	0.275	0.277

Table G-1 (a) Data from experiments of Base oil viscosity @ 100 °C 4.7 cSt (continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
50	0.221	0.214	0.148	0.166	0.177	0.174	0.284	0.275	0.277
51	0.218	0.218	0.151	0.165	0.181	0.173	0.284	0.275	0.277
52	0.209	0.221	0.160	0.164	0.190	0.174	0.284	0.275	0.277
53	0.201	0.222	0.168	0.164	0.201	0.175	0.284	0.275	0.277
54	0.200	0.219	0.173	0.162	0.202	0.176	0.284	0.275	0.277
55	0.197	0.218	0.174	0.161	0.205	0.178	0.284	0.275	0.277
56	0.196	0.219	0.178	0.160	0.208	0.177	0.284	0.275	0.277
57	0.191	0.219	0.180	0.159	0.211	0.178	0.284	0.274	0.277
58	0.192	0.221	0.182	0.158	0.213	0.179	0.284	0.274	0.277
59	0.195	0.222	0.188	0.154	0.213	0.178	0.284	0.274	0.277
60	0.193	0.222	0.189	0.152	0.214	0.178	0.283	0.274	0.277
61	0.195	0.221	0.182	0.151	0.211	0.178	0.283	0.274	0.277
62	0.197	0.218	0.174	0.151	0.209	0.176	0.283	0.274	0.276
63	0.199	0.209	0.166	0.150	0.205	0.176	0.283	0.274	0.276
64	0.201	0.198	0.154	0.150	0.203	0.174	0.283	0.274	0.276
65	0.202	0.186	0.151	0.149	0.199	0.173	0.283	0.274	0.276
66	0.201	0.171	0.148	0.148	0.192	0.172	0.283	0.274	0.276
67	0.203	0.166	0.141	0.147	0.186	0.171	0.283	0.274	0.276
68	0.206	0.159	0.132	0.148	0.181	0.170	0.282	0.274	0.276
69	0.207	0.155	0.113	0.147	0.180	0.169	0.282	0.274	0.276
70	0.209	0.152	0.104	0.147	0.177	0.169	0.282	0.274	0.276
71	0.211	0.148	0.101	0.142	0.179	0.170	0.282	0.274	0.276
72	0.216	0.157	0.113	0.148	0.182	0.171	0.283	0.274	0.276
73	0.219	0.160	0.119	0.147	0.186	0.172	0.283	0.274	0.276
74	0.220	0.162	0.122	0.151	0.190	0.172	0.283	0.274	0.276
75	0.225	0.168	0.125	0.159	0.191	0.173	0.283	0.274	0.276
76	0.231	0.162	0.129	0.161	0.192	0.174	0.283	0.274	0.276
77	0.238	0.159	0.131	0.163	0.192	0.176	0.283	0.274	0.276
78	0.241	0.157	0.132	0.164	0.193	0.175	0.283	0.274	0.276
79	0.246	0.154	0.133	0.166	0.194	0.175	0.283	0.274	0.276

Table G-1 (a) Data from experiments of Base oil viscosity @ 100 °C 4.7 cSt (continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
80	0.244	0.152	0.134	0.169	0.194	0.175	0.283	0.274	0.276
81	0.241	0.161	0.135	0.168	0.195	0.179	0.283	0.274	0.276
82	0.238	0.175	0.131	0.165	0.196	0.181	0.283	0.274	0.276
83	0.226	0.181	0.130	0.164	0.196	0.182	0.282	0.274	0.276
84	0.208	0.184	0.128	0.163	0.198	0.183	0.282	0.274	0.276
85	0.199	0.200	0.127	0.166	0.199	0.184	0.282	0.274	0.276
86	0.187	0.205	0.126	0.165	0.200	0.185	0.282	0.274	0.276
87	0.171	0.209	0.129	0.165	0.201	0.186	0.282	0.274	0.276
88	0.165	0.211	0.127	0.166	0.200	0.187	0.282	0.274	0.276
89	0.160	0.213	0.126	0.164	0.201	0.188	0.282	0.274	0.277
90	0.162	0.215	0.126	0.164	0.201	0.188	0.282	0.274	0.277
91	0.179	0.218	0.131	0.163	0.203	0.187	0.282	0.274	0.277
92	0.206	0.219	0.138	0.168	0.205	0.185	0.282	0.274	0.277
93	0.212	0.211	0.141	0.169	0.206	0.184	0.282	0.274	0.277
94	0.228	0.207	0.149	0.169	0.207	0.184	0.282	0.274	0.277
95	0.236	0.200	0.152	0.170	0.209	0.183	0.282	0.274	0.277
96	0.244	0.192	0.153	0.169	0.210	0.181	0.282	0.274	0.277
97	0.249	0.188	0.154	0.169	0.211	0.181	0.282	0.274	0.277
98	0.255	0.182	0.155	0.169	0.213	0.181	0.282	0.274	0.277
99	0.253	0.171	0.157	0.168	0.214	0.180	0.282	0.274	0.277
100	0.252	0.160	0.156	0.168	0.217	0.181	0.282	0.274	0.277
101	0.248	0.164	0.151	0.169	0.219	0.181	0.282	0.274	0.277
102	0.241	0.168	0.149	0.169	0.221	0.181	0.282	0.274	0.277
103	0.235	0.171	0.147	0.168	0.221	0.181	0.282	0.274	0.277
104	0.231	0.181	0.144	0.167	0.221	0.181	0.282	0.274	0.277
105	0.225	0.180	0.141	0.166	0.222	0.180	0.282	0.274	0.277
106	0.221	0.175	0.139	0.164	0.222	0.181	0.282	0.274	0.277
107	0.220	0.175	0.133	0.165	0.224	0.181	0.282	0.274	0.277
108	0.213	0.174	0.132	0.163	0.225	0.181	0.282	0.274	0.276
109	0.211	0.177	0.131	0.162	0.225	0.181	0.282	0.274	0.276

Table G-1 (a) Data from experiments of Base oil viscosity @ 100 °C 4.7 cSt (continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
110	0.210	0.178	0.129	0.161	0.224	0.181	0.282	0.274	0.276
111	0.215	0.176	0.119	0.162	0.224	0.181	0.282	0.274	0.276
112	0.219	0.176	0.119	0.163	0.224	0.181	0.282	0.274	0.276
113	0.221	0.176	0.120	0.164	0.224	0.181	0.282	0.274	0.276
114	0.225	0.171	0.120	0.165	0.225	0.182	0.282	0.274	0.276
115	0.231	0.172	0.117	0.166	0.224	0.183	0.282	0.274	0.276
116	0.229	0.172	0.119	0.167	0.224	0.183	0.282	0.274	0.276
117	0.228	0.172	0.118	0.168	0.223	0.183	0.282	0.274	0.276
118	0.221	0.175	0.117	0.167	0.223	0.183	0.281	0.274	0.276
119	0.218	0.172	0.117	0.167	0.223	0.183	0.281	0.274	0.276
120	0.223	0.172	0.117	0.167	0.223	0.183	0.281	0.274	0.276

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Table G-1 (b) Data from experiments of Base oil viscosity @ 100 °C 11.2 cSt

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1	0.190	0.221	0.224	0.291	0.174	0.208	0.286	0.299	0.298
2	0.190	0.220	0.221	0.291	0.173	0.212	0.286	0.299	0.298
3	0.188	0.218	0.220	0.290	0.173	0.214	0.288	0.299	0.298
4	0.187	0.219	0.218	0.289	0.172	0.217	0.288	0.299	0.298
5	0.187	0.209	0.217	0.291	0.171	0.217	0.290	0.298	0.298
6	0.188	0.201	0.214	0.291	0.171	0.219	0.290	0.299	0.299
7	0.188	0.195	0.211	0.291	0.171	0.220	0.291	0.299	0.298
8	0.187	0.192	0.211	0.291	0.171	0.221	0.291	0.299	0.299
9	0.188	0.187	0.210	0.292	0.171	0.221	0.292	0.299	0.299
10	0.189	0.185	0.210	0.292	0.171	0.221	0.293	0.299	0.299
11	0.185	0.183	0.209	0.292	0.171	0.222	0.293	0.299	0.299
12	0.179	0.183	0.207	0.292	0.171	0.222	0.293	0.299	0.299
13	0.179	0.182	0.207	0.292	0.170	0.221	0.293	0.298	0.299
14	0.175	0.186	0.208	0.292	0.170	0.222	0.293	0.298	0.299
15	0.174	0.189	0.207	0.292	0.169	0.222	0.293	0.297	0.299
16	0.173	0.191	0.207	0.292	0.169	0.222	0.293	0.297	0.299
17	0.173	0.191	0.207	0.292	0.170	0.222	0.293	0.297	0.298
18	0.172	0.196	0.207	0.292	0.169	0.223	0.293	0.297	0.298
19	0.171	0.197	0.208	0.292	0.169	0.223	0.293	0.297	0.298
20	0.170	0.197	0.207	0.292	0.169	0.223	0.293	0.296	0.297
21	0.170	0.199	0.206	0.292	0.170	0.223	0.293	0.295	0.297
22	0.170	0.197	0.208	0.292	0.171	0.223	0.293	0.295	0.297
23	0.171	0.195	0.204	0.292	0.170	0.224	0.293	0.295	0.297
24	0.172	0.194	0.204	0.291	0.170	0.224	0.293	0.295	0.298
25	0.173	0.191	0.204	0.291	0.170	0.224	0.293	0.295	0.298
26	0.173	0.190	0.203	0.291	0.170	0.224	0.293	0.295	0.297
27	0.173	0.189	0.199	0.291	0.171	0.225	0.292	0.295	0.296
28	0.173	0.188	0.192	0.290	0.171	0.225	0.293	0.294	0.296
29	0.174	0.187	0.188	0.290	0.171	0.225	0.293	0.294	0.296
30	0.175	0.188	0.185	0.290	0.172	0.225	0.292	0.294	0.295

Table G-1 (b) Data from experiments of Base oil viscosity @ 100 °C 11.2 cSt  
(continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
31	0.178	0.187	0.181	0.290	0.171	0.226	0.292	0.294	0.295
32	0.179	0.186	0.181	0.290	0.171	0.227	0.292	0.294	0.295
33	0.175	0.183	0.178	0.288	0.171	0.227	0.292	0.294	0.295
34	0.171	0.181	0.177	0.288	0.171	0.227	0.293	0.294	0.294
35	0.169	0.181	0.172	0.288	0.171	0.227	0.293	0.294	0.294
36	0.165	0.180	0.174	0.287	0.171	0.228	0.293	0.295	0.294
37	0.162	0.179	0.173	0.287	0.171	0.229	0.293	0.294	0.295
38	0.160	0.178	0.171	0.287	0.171	0.229	0.293	0.294	0.294
39	0.159	0.177	0.171	0.287	0.171	0.230	0.293	0.294	0.294
40	0.159	0.176	0.170	0.287	0.172	0.232	0.293	0.294	0.293
41	0.158	0.175	0.169	0.287	0.172	0.231	0.293	0.294	0.294
42	0.158	0.176	0.164	0.287	0.172	0.232	0.293	0.294	0.294
43	0.157	0.176	0.168	0.286	0.172	0.233	0.292	0.294	0.293
44	0.157	0.176	0.168	0.286	0.172	0.233	0.292	0.294	0.294
45	0.155	0.176	0.166	0.286	0.172	0.234	0.292	0.294	0.294
46	0.155	0.176	0.166	0.286	0.171	0.235	0.292	0.294	0.294
47	0.151	0.175	0.168	0.285	0.171	0.236	0.292	0.294	0.294
48	0.151	0.176	0.168	0.285	0.171	0.235	0.292	0.294	0.294
49	0.150	0.176	0.168	0.285	0.171	0.235	0.292	0.294	0.294
50	0.149	0.176	0.168	0.284	0.172	0.236	0.292	0.294	0.294
51	0.148	0.175	0.166	0.285	0.172	0.237	0.291	0.293	0.294
52	0.148	0.173	0.166	0.285	0.172	0.237	0.291	0.293	0.293
53	0.148	0.171	0.165	0.285	0.172	0.238	0.291	0.292	0.293
54	0.148	0.170	0.164	0.283	0.173	0.239	0.291	0.291	0.293
55	0.146	0.169	0.164	0.284	0.173	0.241	0.290	0.291	0.293
56	0.144	0.161	0.164	0.284	0.172	0.242	0.290	0.291	0.293
57	0.144	0.159	0.159	0.284	0.174	0.243	0.289	0.291	0.293
58	0.145	0.155	0.159	0.285	0.175	0.243	0.289	0.291	0.293
59	0.146	0.154	0.158	0.285	0.176	0.243	0.289	0.291	0.293
60	0.144	0.151	0.158	0.285	0.175	0.244	0.289	0.291	0.293

Table G-1 (b) Data from experiments of Base oil viscosity @ 100 °C 11.2 cSt

(continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
61	0.143	0.152	0.159	0.285	0.175	0.244	0.289	0.291	0.293
62	0.143	0.151	0.161	0.285	0.175	0.244	0.289	0.291	0.293
63	0.142	0.151	0.161	0.285	0.175	0.245	0.288	0.291	0.293
64	0.141	0.158	0.162	0.285	0.175	0.246	0.288	0.291	0.293
65	0.141	0.157	0.162	0.285	0.175	0.247	0.288	0.291	0.293
66	0.140	0.157	0.162	0.286	0.175	0.248	0.288	0.291	0.294
67	0.140	0.157	0.163	0.286	0.176	0.248	0.288	0.291	0.293
68	0.136	0.156	0.163	0.286	0.175	0.248	0.288	0.291	0.293
69	0.135	0.157	0.164	0.286	0.175	0.248	0.288	0.291	0.293
70	0.130	0.158	0.163	0.286	0.176	0.249	0.288	0.291	0.293
71	0.129	0.153	0.161	0.286	0.176	0.249	0.288	0.291	0.293
72	0.128	0.151	0.158	0.285	0.176	0.250	0.287	0.291	0.293
73	0.128	0.149	0.155	0.285	0.177	0.251	0.288	0.291	0.293
74	0.128	0.148	0.151	0.284	0.179	0.252	0.288	0.291	0.293
75	0.126	0.141	0.147	0.284	0.178	0.252	0.288	0.291	0.293
76	0.126	0.140	0.144	0.283	0.179	0.253	0.287	0.291	0.293
77	0.123	0.135	0.136	0.282	0.181	0.254	0.287	0.291	0.293
78	0.121	0.129	0.132	0.282	0.182	0.254	0.287	0.291	0.293
79	0.120	0.123	0.129	0.282	0.181	0.255	0.287	0.291	0.293
80	0.119	0.120	0.122	0.281	0.182	0.255	0.287	0.291	0.293
81	0.119	0.111	0.128	0.282	0.183	0.256	0.287	0.291	0.293
82	0.118	0.119	0.127	0.284	0.183	0.257	0.287	0.291	0.293
83	0.118	0.120	0.128	0.284	0.184	0.257	0.287	0.291	0.293
84	0.118	0.131	0.126	0.284	0.185	0.258	0.287	0.291	0.293
85	0.118	0.141	0.125	0.284	0.185	0.259	0.287	0.291	0.293
86	0.118	0.149	0.124	0.283	0.185	0.259	0.288	0.291	0.293
87	0.116	0.158	0.127	0.284	0.185	0.259	0.287	0.291	0.293
88	0.117	0.157	0.127	0.284	0.185	0.260	0.287	0.291	0.293
89	0.117	0.154	0.126	0.284	0.185	0.260	0.287	0.291	0.293
90	0.119	0.152	0.126	0.284	0.186	0.260	0.287	0.291	0.292

Table G-1 (b) Data from experiments of Base oil viscosity @ 100 °C 11.2 cSt

(continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
91	0.119	0.152	0.127	0.284	0.185	0.260	0.287	0.291	0.293
92	0.118	0.153	0.127	0.283	0.186	0.261	0.287	0.291	0.292
93	0.118	0.151	0.127	0.284	0.186	0.261	0.287	0.291	0.292
94	0.118	0.151	0.128	0.284	0.188	0.261	0.287	0.290	0.292
95	0.118	0.151	0.127	0.285	0.189	0.261	0.288	0.290	0.292
96	0.118	0.151	0.127	0.285	0.189	0.261	0.287	0.290	0.292
97	0.119	0.152	0.127	0.285	0.190	0.262	0.287	0.290	0.293
98	0.119	0.152	0.129	0.286	0.191	0.261	0.287	0.290	0.292
99	0.119	0.153	0.129	0.286	0.191	0.262	0.287	0.290	0.292
100	0.120	0.152	0.129	0.286	0.191	0.262	0.287	0.290	0.292
101	0.119	0.152	0.132	0.286	0.192	0.262	0.287	0.290	0.292
102	0.119	0.152	0.135	0.286	0.192	0.262	0.287	0.289	0.292
103	0.119	0.158	0.135	0.286	0.191	0.262	0.287	0.289	0.292
104	0.120	0.158	0.134	0.286	0.191	0.262	0.287	0.289	0.293
105	0.120	0.160	0.134	0.286	0.191	0.263	0.287	0.289	0.293
106	0.121	0.161	0.134	0.287	0.191	0.263	0.287	0.289	0.293
107	0.121	0.161	0.134	0.287	0.193	0.263	0.287	0.289	0.293
108	0.121	0.162	0.135	0.287	0.193	0.263	0.287	0.288	0.293
109	0.121	0.162	0.136	0.287	0.193	0.263	0.287	0.288	0.293
110	0.122	0.162	0.136	0.287	0.192	0.263	0.287	0.288	0.293
111	0.122	0.161	0.136	0.287	0.193	0.263	0.287	0.288	0.293
112	0.122	0.160	0.136	0.287	0.193	0.263	0.287	0.288	0.293
113	0.122	0.160	0.135	0.287	0.192	0.263	0.287	0.288	0.293
114	0.122	0.160	0.134	0.287	0.192	0.263	0.287	0.288	0.293
115	0.122	0.159	0.134	0.287	0.192	0.263	0.287	0.288	0.293
116	0.122	0.159	0.133	0.287	0.192	0.263	0.287	0.288	0.293
117	0.122	0.159	0.133	0.287	0.192	0.263	0.287	0.288	0.293
118	0.122	0.159	0.134	0.287	0.192	0.263	0.287	0.288	0.293
119	0.122	0.159	0.134	0.288	0.192	0.263	0.287	0.288	0.293
120	0.122	0.159	0.134	0.288	0.192	0.263	0.287	0.288	0.293



Table G-1 (c) Data from experiments of Base oil viscosity @ 100 °C 32.0 cSt

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1	0.149	0.211	0.139	0.224	0.160	0.224	0.278	0.224	0.299
2	0.148	0.209	0.124	0.221	0.145	0.221	0.279	0.225	0.299
3	0.151	0.208	0.119	0.218	0.142	0.222	0.279	0.225	0.299
4	0.160	0.201	0.101	0.216	0.133	0.222	0.276	0.226	0.299
5	0.163	0.191	0.094	0.214	0.132	0.231	0.277	0.231	0.299
6	0.165	0.173	0.088	0.217	0.140	0.233	0.277	0.232	0.299
7	0.171	0.163	0.071	0.218	0.142	0.238	0.279	0.235	0.299
8	0.170	0.144	0.060	0.219	0.149	0.241	0.279	0.240	0.299
9	0.171	0.129	0.054	0.221	0.150	0.242	0.279	0.242	0.299
10	0.172	0.116	0.046	0.224	0.155	0.245	0.280	0.245	0.299
11	0.173	0.129	0.044	0.222	0.163	0.245	0.279	0.246	0.298
12	0.174	0.123	0.041	0.222	0.172	0.245	0.279	0.248	0.298
13	0.175	0.140	0.037	0.222	0.166	0.246	0.280	0.247	0.298
14	0.174	0.156	0.036	0.218	0.165	0.246	0.281	0.248	0.298
15	0.175	0.168	0.031	0.217	0.158	0.248	0.281	0.250	0.298
16	0.176	0.179	0.032	0.214	0.153	0.249	0.281	0.250	0.298
17	0.177	0.174	0.030	0.219	0.141	0.251	0.280	0.251	0.298
18	0.178	0.172	0.027	0.218	0.150	0.250	0.281	0.250	0.298
19	0.179	0.171	0.028	0.219	0.153	0.250	0.281	0.250	0.298
20	0.180	0.175	0.028	0.221	0.154	0.249	0.281	0.249	0.298
21	0.179	0.173	0.029	0.218	0.152	0.251	0.278	0.252	0.299
22	0.179	0.173	0.030	0.216	0.150	0.251	0.276	0.253	0.297
23	0.175	0.171	0.032	0.214	0.146	0.247	0.274	0.252	0.297
24	0.171	0.172	0.033	0.212	0.139	0.247	0.274	0.252	0.297
25	0.171	0.171	0.036	0.210	0.143	0.244	0.275	0.253	0.297
26	0.169	0.171	0.038	0.207	0.141	0.249	0.279	0.254	0.298
27	0.168	0.170	0.037	0.209	0.143	0.251	0.279	0.252	0.298
28	0.166	0.170	0.035	0.210	0.146	0.252	0.278	0.252	0.298
29	0.168	0.170	0.036	0.211	0.143	0.252	0.279	0.251	0.298
30	0.167	0.170	0.037	0.213	0.151	0.251	0.280	0.251	0.297

Table G-1 (c) Data from experiments of Base oil viscosity @ 100 °C 32.0 cSt

(continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
31	0.163	0.165	0.034	0.214	0.153	0.253	0.279	0.248	0.297
32	0.161	0.166	0.031	0.216	0.154	0.254	0.279	0.248	0.297
33	0.155	0.159	0.027	0.217	0.156	0.254	0.279	0.250	0.297
34	0.159	0.147	0.019	0.217	0.158	0.252	0.278	0.251	0.297
35	0.154	0.133	0.017	0.214	0.159	0.252	0.275	0.252	0.297
36	0.152	0.131	0.014	0.212	0.163	0.253	0.275	0.252	0.296
37	0.151	0.136	0.009	0.211	0.168	0.253	0.271	0.253	0.296
38	0.149	0.147	0.006	0.209	0.169	0.253	0.276	0.253	0.297
39	0.145	0.149	0.003	0.208	0.161	0.251	0.276	0.253	0.297
40	0.141	0.150	0.000	0.208	0.154	0.251	0.278	0.254	0.297
41	0.139	0.152	0.000	0.207	0.151	0.249	0.279	0.257	0.297
42	0.138	0.143	0.001	0.205	0.150	0.249	0.277	0.257	0.297
43	0.139	0.144	0.002	0.203	0.149	0.248	0.277	0.257	0.296
44	0.141	0.141	0.008	0.198	0.148	0.247	0.275	0.257	0.296
45	0.141	0.135	0.003	0.192	0.147	0.248	0.276	0.258	0.296
46	0.140	0.135	0.009	0.191	0.146	0.246	0.277	0.259	0.295
47	0.139	0.134	0.010	0.186	0.148	0.247	0.277	0.260	0.295
48	0.140	0.130	0.013	0.190	0.149	0.248	0.277	0.259	0.294
49	0.142	0.129	0.014	0.195	0.154	0.252	0.278	0.258	0.295
50	0.144	0.123	0.013	0.197	0.154	0.258	0.278	0.258	0.295
51	0.143	0.128	0.014	0.198	0.158	0.253	0.273	0.259	0.295
52	0.140	0.127	0.011	0.201	0.159	0.254	0.273	0.257	0.295
53	0.139	0.126	0.000	0.197	0.159	0.255	0.275	0.257	0.295
54	0.135	0.125	0.003	0.194	0.157	0.255	0.275	0.255	0.295
55	0.133	0.124	0.021	0.192	0.156	0.255	0.276	0.257	0.295
56	0.131	0.124	0.030	0.191	0.155	0.259	0.275	0.256	0.295
57	0.130	0.126	0.028	0.190	0.155	0.259	0.272	0.258	0.294
58	0.129	0.128	0.031	0.188	0.154	0.259	0.277	0.258	0.294
59	0.129	0.127	0.038	0.187	0.156	0.259	0.277	0.258	0.294
60	0.127	0.126	0.043	0.185	0.156	0.259	0.277	0.259	0.294

Table G-1 (c) Data from experiments of Base oil viscosity @ 100 °C 32.0 cSt

(continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
61	0.125	0.130	0.049	0.184	0.153	0.258	0.276	0.260	0.294
62	0.124	0.130	0.058	0.183	0.153	0.259	0.271	0.260	0.293
63	0.128	0.131	0.061	0.183	0.153	0.261	0.270	0.260	0.293
64	0.127	0.131	0.069	0.183	0.151	0.262	0.269	0.260	0.293
65	0.124	0.132	0.073	0.182	0.151	0.262	0.270	0.260	0.293
66	0.124	0.136	0.078	0.181	0.150	0.261	0.272	0.258	0.292
67	0.123	0.135	0.081	0.180	0.149	0.259	0.273	0.259	0.292
68	0.122	0.137	0.082	0.179	0.151	0.258	0.274	0.260	0.292
69	0.125	0.138	0.082	0.175	0.152	0.259	0.273	0.260	0.292
70	0.123	0.139	0.081	0.174	0.154	0.261	0.273	0.261	0.292
71	0.122	0.136	0.082	0.171	0.154	0.262	0.272	0.262	0.292
72	0.121	0.133	0.083	0.170	0.153	0.262	0.272	0.262	0.292
73	0.120	0.132	0.083	0.165	0.154	0.263	0.272	0.263	0.292
74	0.120	0.131	0.084	0.161	0.155	0.263	0.271	0.263	0.292
75	0.119	0.130	0.084	0.160	0.156	0.263	0.272	0.264	0.292
76	0.117	0.129	0.084	0.157	0.157	0.265	0.275	0.264	0.293
77	0.117	0.129	0.085	0.157	0.158	0.269	0.273	0.266	0.292
78	0.117	0.128	0.084	0.155	0.159	0.270	0.274	0.268	0.292
79	0.115	0.129	0.084	0.154	0.161	0.271	0.274	0.268	0.292
80	0.114	0.126	0.084	0.156	0.164	0.270	0.272	0.270	0.292
81	0.113	0.132	0.082	0.155	0.164	0.270	0.273	0.270	0.292
82	0.119	0.132	0.081	0.153	0.164	0.269	0.271	0.269	0.292
83	0.118	0.135	0.072	0.151	0.163	0.269	0.272	0.270	0.292
84	0.118	0.137	0.071	0.151	0.163	0.268	0.271	0.270	0.292
85	0.120	0.137	0.068	0.149	0.163	0.268	0.269	0.270	0.292
86	0.121	0.138	0.064	0.147	0.163	0.268	0.269	0.270	0.292
87	0.121	0.139	0.063	0.141	0.163	0.269	0.269	0.270	0.292
88	0.121	0.139	0.063	0.139	0.163	0.269	0.270	0.270	0.292
89	0.124	0.140	0.063	0.139	0.164	0.270	0.269	0.270	0.292
90	0.125	0.141	0.063	0.139	0.164	0.270	0.270	0.270	0.292

Table G-1 (c) Data from experiments of Base oil viscosity @ 100 °C 32.0 cSt

(continued)

Time	% Sulphur content								
	80 °C			100 °C			120 °C		
	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm	300rpm	400rpm	500rpm
91	0.126	0.141	0.063	0.136	0.164	0.270	0.271	0.270	0.292
92	0.125	0.140	0.063	0.135	0.164	0.270	0.271	0.270	0.292
93	0.126	0.141	0.063	0.132	0.164	0.270	0.272	0.270	0.292
94	0.128	0.141	0.069	0.135	0.166	0.270	0.272	0.271	0.292
95	0.130	0.140	0.069	0.133	0.166	0.270	0.271	0.271	0.292
96	0.130	0.140	0.069	0.133	0.166	0.270	0.271	0.271	0.292
97	0.129	0.140	0.070	0.131	0.167	0.270	0.272	0.272	0.292
98	0.129	0.140	0.071	0.132	0.167	0.270	0.272	0.272	0.292
99	0.129	0.140	0.072	0.131	0.166	0.272	0.271	0.271	0.292
100	0.129	0.140	0.073	0.132	0.166	0.271	0.271	0.271	0.292
101	0.129	0.140	0.071	0.132	0.166	0.272	0.273	0.272	0.292
102	0.130	0.139	0.069	0.133	0.165	0.271	0.272	0.272	0.292
103	0.130	0.139	0.069	0.134	0.166	0.271	0.271	0.273	0.292
104	0.130	0.139	0.069	0.134	0.166	0.271	0.271	0.274	0.292
105	0.130	0.139	0.069	0.135	0.166	0.271	0.271	0.274	0.292
106	0.132	0.138	0.069	0.135	0.165	0.272	0.271	0.274	0.292
107	0.135	0.138	0.069	0.135	0.166	0.272	0.271	0.275	0.292
108	0.135	0.138	0.069	0.135	0.166	0.272	0.272	0.274	0.292
109	0.134	0.138	0.069	0.136	0.166	0.272	0.271	0.273	0.292
110	0.135	0.138	0.069	0.136	0.166	0.273	0.269	0.273	0.292
111	0.135	0.138	0.069	0.135	0.166	0.273	0.271	0.273	0.292
112	0.135	0.138	0.071	0.135	0.166	0.273	0.271	0.273	0.292
113	0.135	0.138	0.071	0.135	0.165	0.273	0.271	0.273	0.292
114	0.135	0.138	0.071	0.135	0.166	0.273	0.271	0.273	0.292
115	0.135	0.138	0.071	0.135	0.166	0.273	0.271	0.273	0.292
116	0.135	0.138	0.072	0.135	0.166	0.273	0.271	0.273	0.292
117	0.135	0.138	0.072	0.135	0.164	0.273	0.271	0.273	0.292
118	0.135	0.138	0.072	0.134	0.166	0.273	0.271	0.273	0.292
119	0.135	0.138	0.072	0.134	0.165	0.273	0.271	0.273	0.292
120	0.135	0.138	0.072	0.134	0.166	0.273	0.271	0.273	0.292

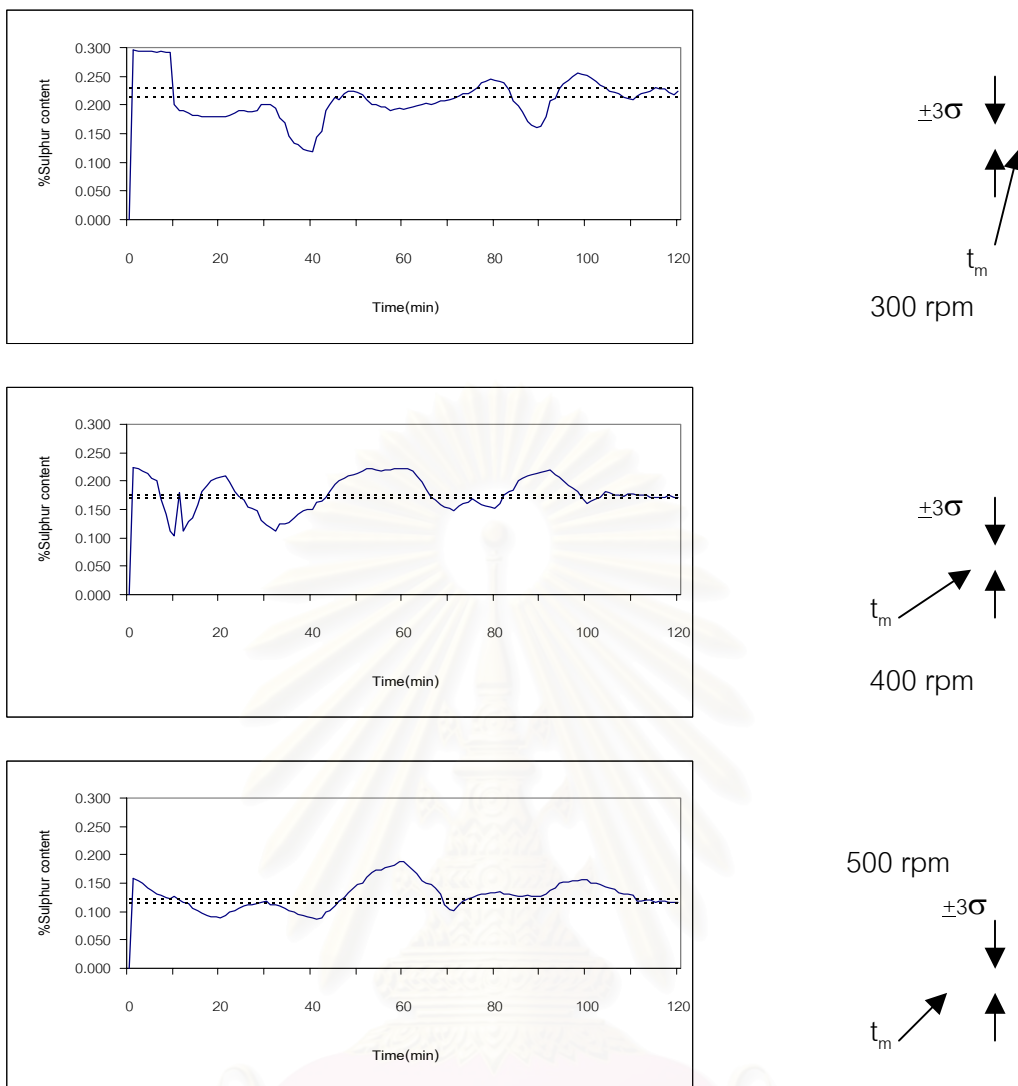
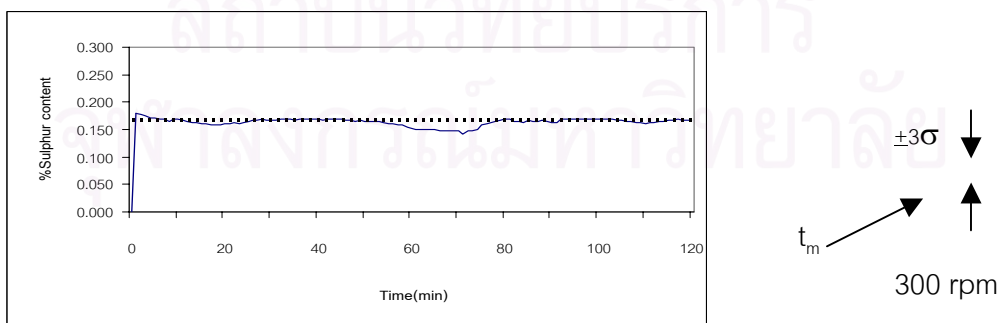


Figure G-1 Mixing Time curve, Temperature 80 °C Viscosity@100 °C=4.7 cSt

N=300,400,500 rpm



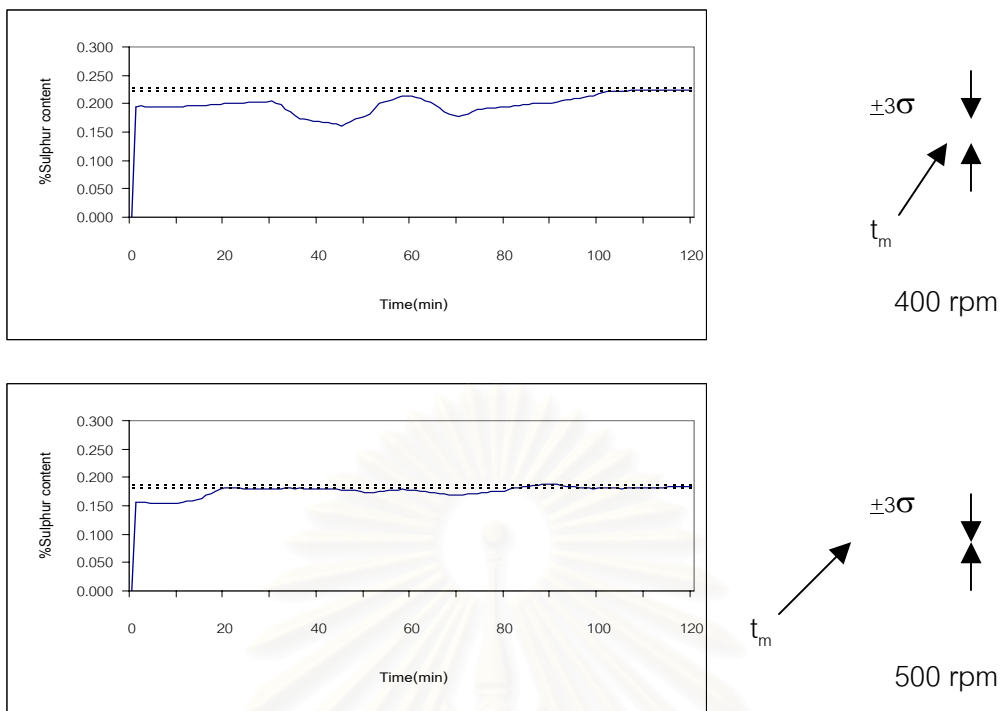
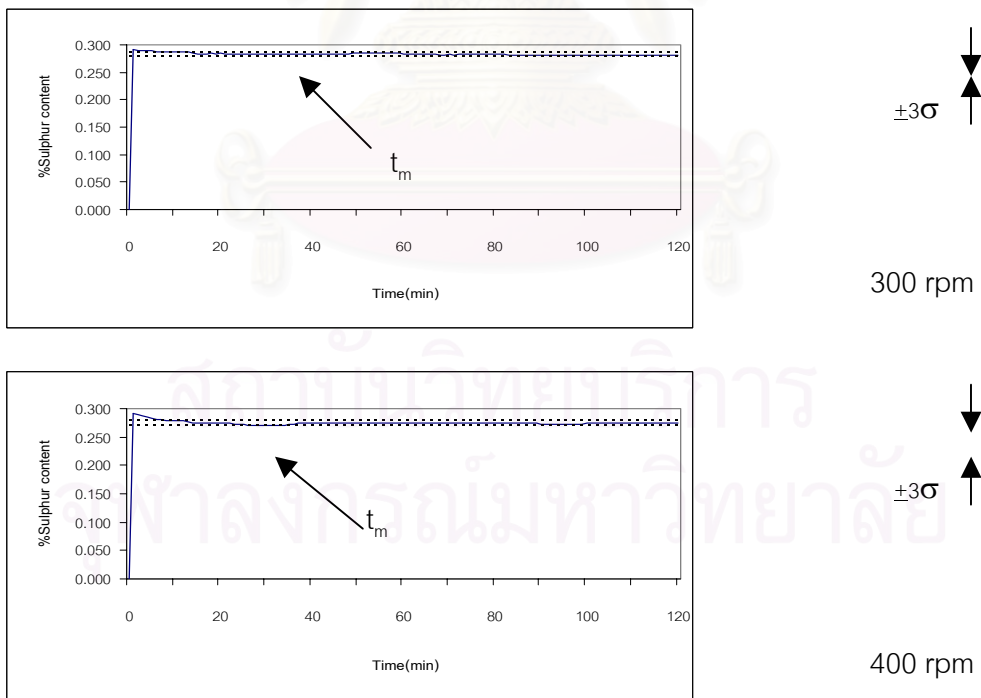


Figure G-2 Mixing Time curve, Temperature 100 °C Viscosity@100 °C=4.7 cSt

N=300,400,500 rpm



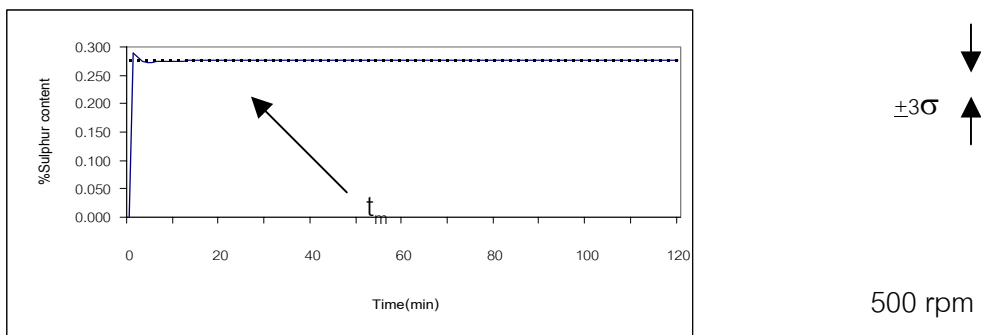


Figure G-3 Mixing Time curve, Temperature 120 °C Viscosity@100 °C=4.7 cSt

N=300,400,500 rpm

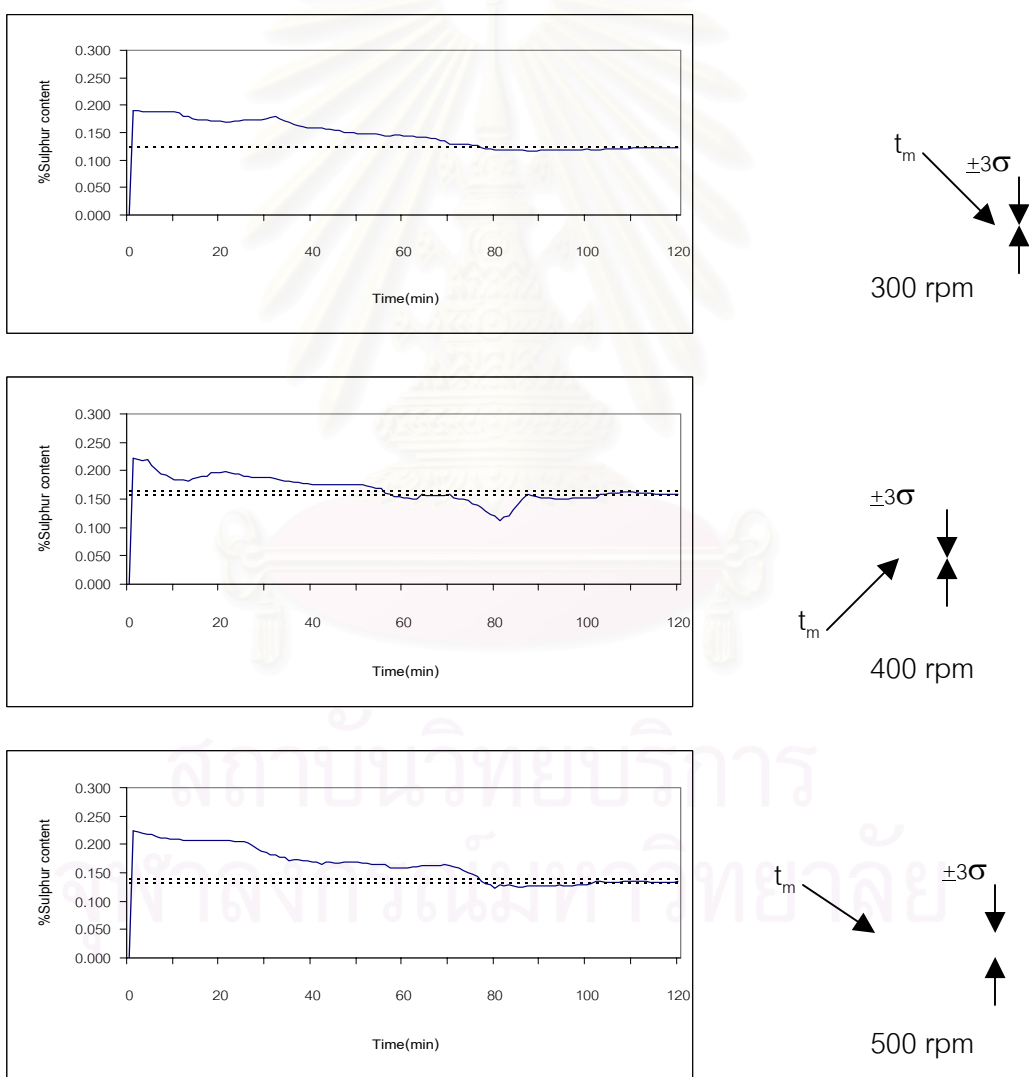


Figure G-4 Mixing Time curve, Temperature 80 °C Viscosity@100 °C=11.2 cSt

N=300,400,500 rpm

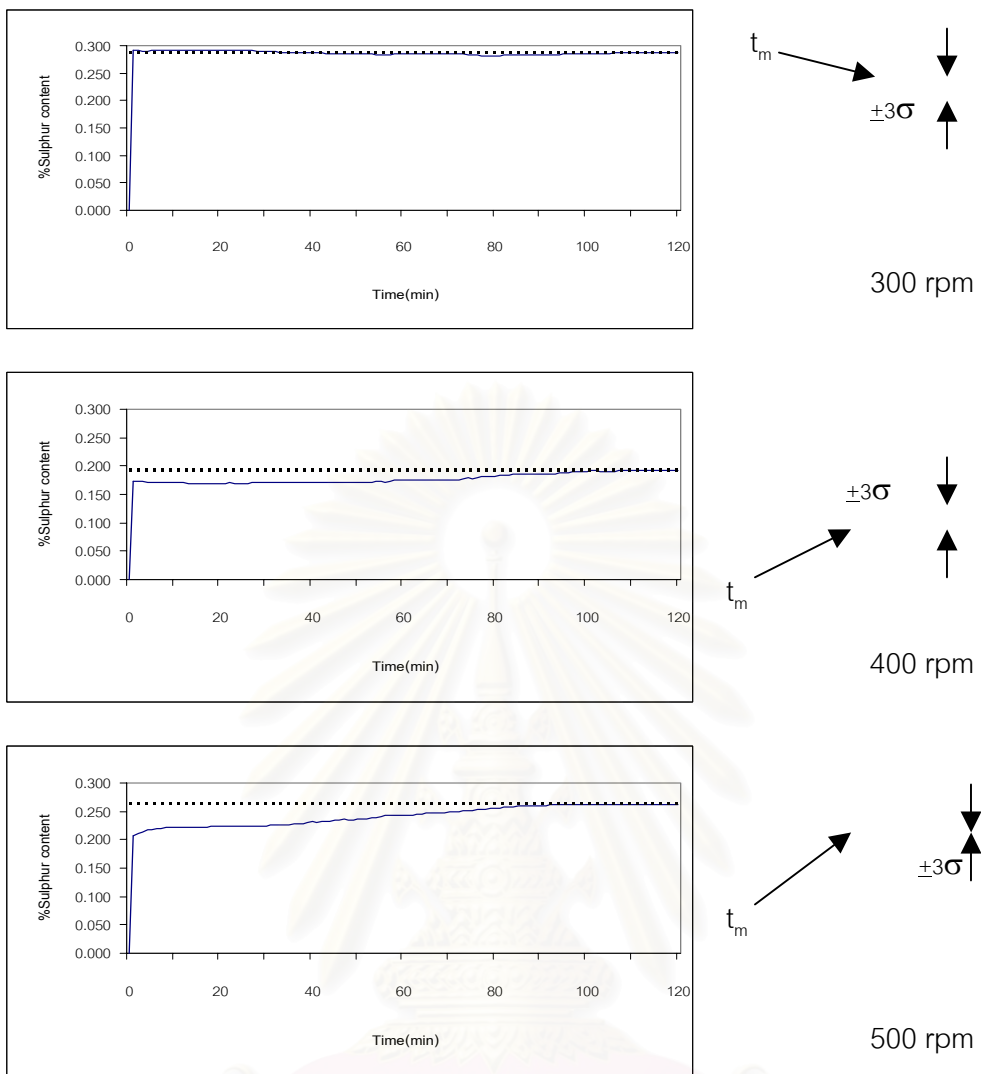
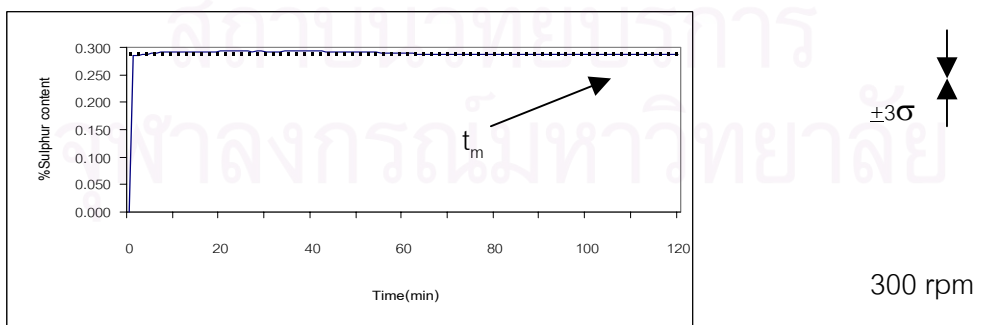


Figure G-5 Mixing Time curve, Temperature 100 °C Viscosity@100 °C=11.2 cSt  
N=300,400,500 rpm





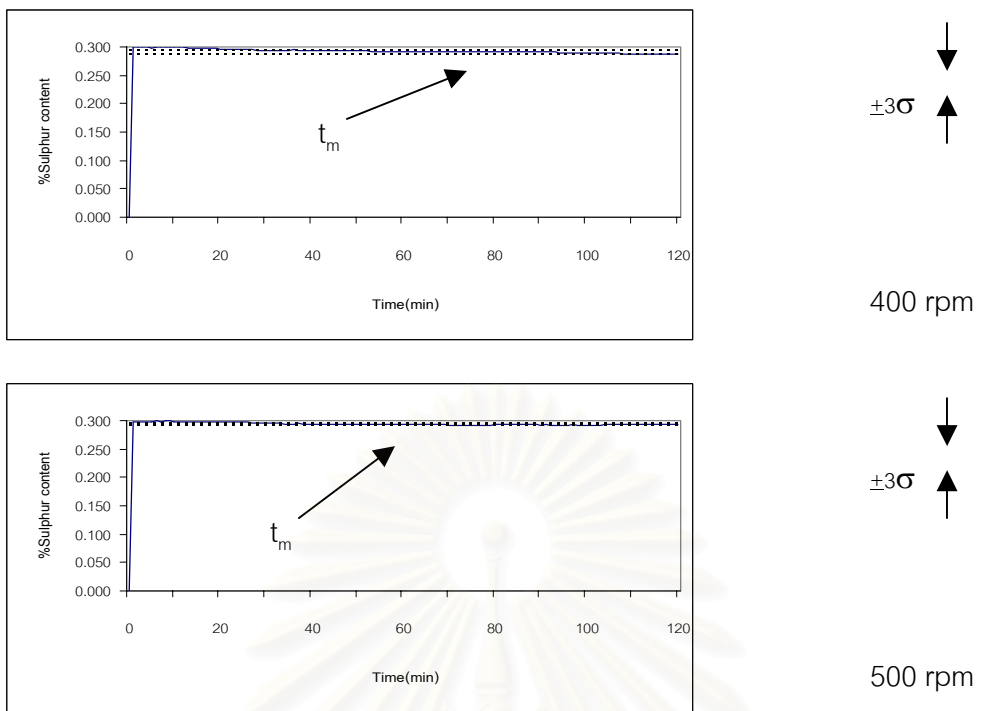
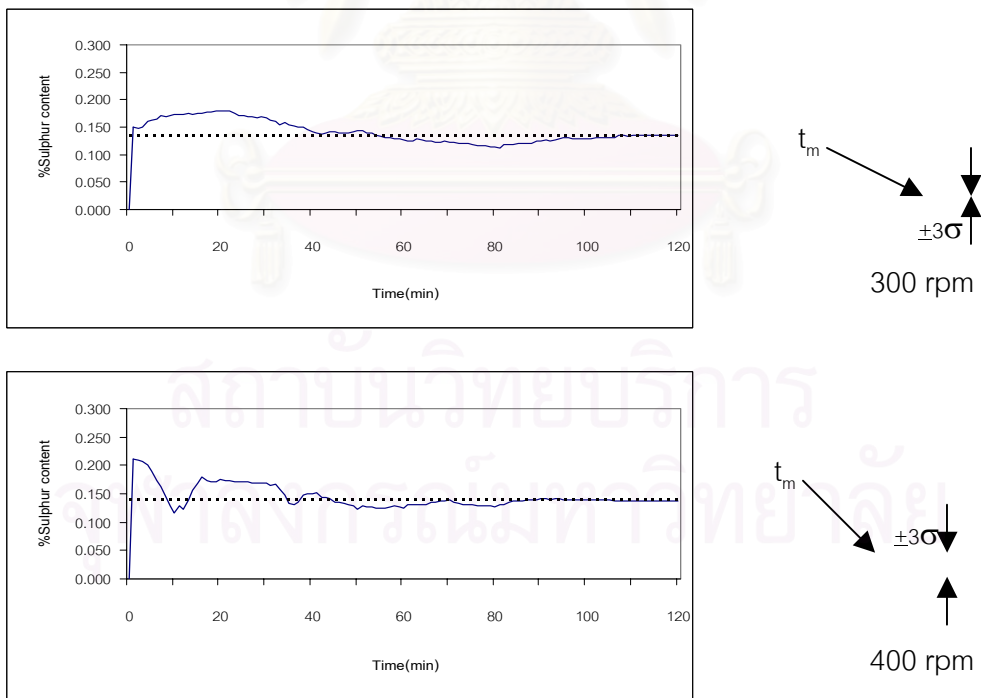


Figure G-6 Mixing Time curve, Temperature 120 °C Viscosity@100 °C=11.2 cSt

N=300,400,500 rpm



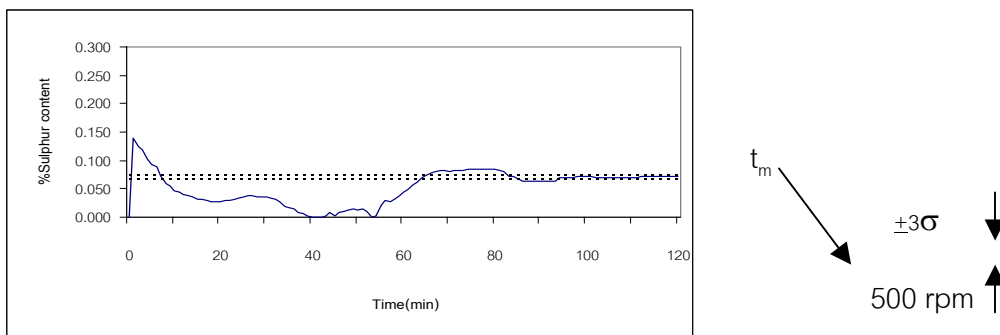


Figure G-7 Mixing Time curve, Temperature 80 °C Viscosity@100 °C=32.0 cSt

N=300,400,500 rpm

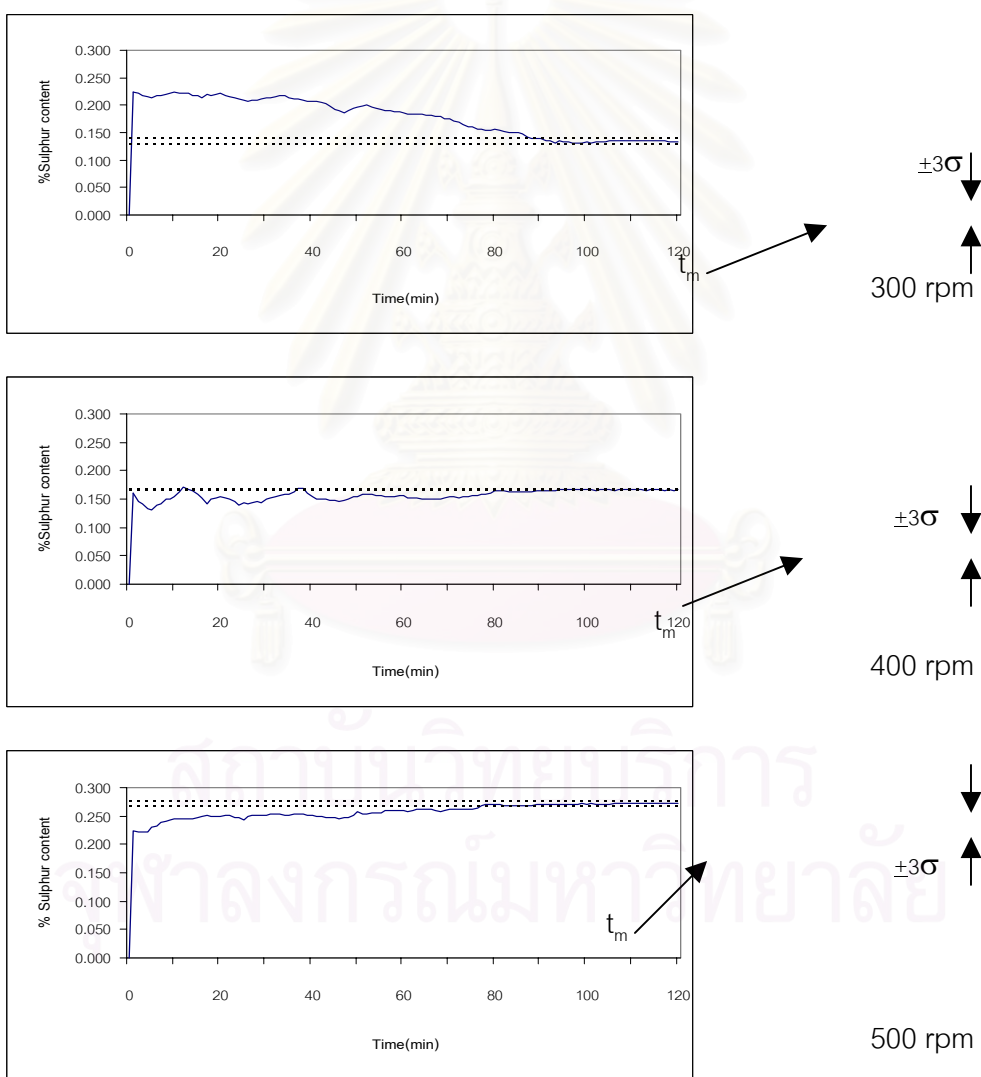


Figure G-8 Mixing Time curve, Temperature 100 °C Viscosity@100 °C=32.0 cSt

N=300,400,500 rpm

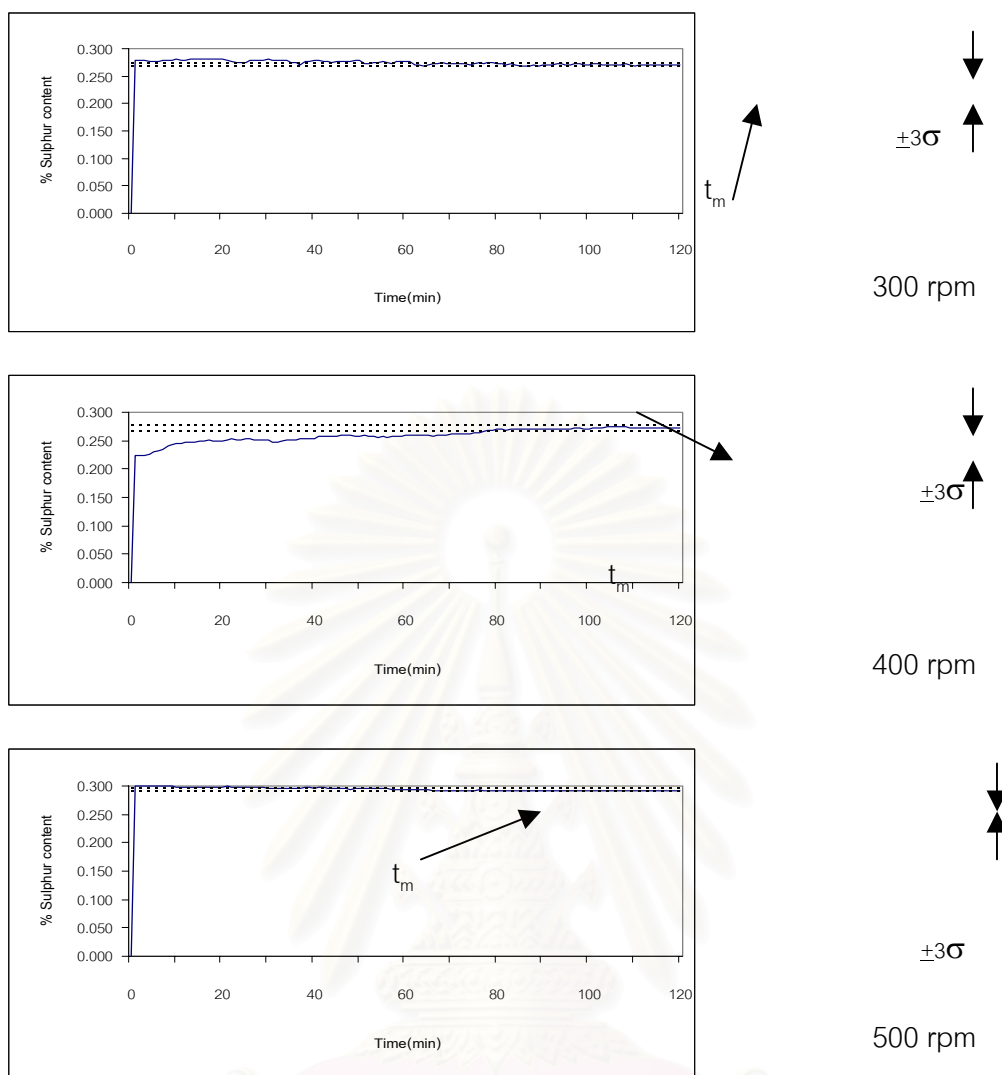


Figure G-9 Mixing Time curve, Temperature 120 °C Viscosity@100 °C=32.0 cSt  
N=300,400,500 rpm

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## APPENDIX H

### SOLUBILITY OF SULPHUR IN LUBRICATING OILS vs. TEMPERATURE

Turbidity method is used to detect %Sulphur do not dissolve by used UV Spectro photometer at a wave length 600 nm as detector. The mass needed to saturated the liquid ( $C_s$ ) at a specific temperature is measured as Absorbance and converted to %wt by calibration curve. From this %wt can calculation value of saturation concentration (Solubility) of Sulphur in lubricating oil.

Accurate value of Solubility is essential for a proper calculation of mass diffusion solubility of Sulphur in lubricating oils vs. temperature.

Table H-1 Solubility of Sulphur in lubricating oil at various temperatures

Temperature (°C)	Solubility (g of solute/1000 cm <sup>3</sup> of lubricating oil)		
	@ 100 °C = 4.7 cSt	@ 100 °C = 11.2 cSt	@ 100 °C = 32.0 cSt
80	494.385	473.634	389.851
90	525.616	503.554	414.478
100	558.819	535.364	440.661
110	594.120	569.183	468.498
120	631.651	605.139	498.093

## APPENDIX I

### Test Method for determination of Sulphur content in Lubricating Oil by Ultraviolet Transmittance (UV Spectro photometric method)

#### 1. Scope

This method describes a procedure for the determination of the transmittances of Sulphur powder that do not dissolve at wavelength 600 nm. The results provide a measure of the content of sample that does not dissolve with respect to ultraviolet absorbing compounds.

Note 1. General information on spectroscopy can be obtained from the following standards:

ASTM E 131: Standard definitions of terms and symbols relating to molecular spectroscopy.

ASTM E 169: Standard recommended practices for general techniques of ultraviolet quantitative analysis.

ASTM E 275: Standard recommended practice for describing and measuring performance of spectrophotometers.

#### 2. Apparatus

2.1 *Ultraviolet Spectrophotometer System*, suitable for measurement at wavelength 600 nm, having a spectral bandwidth 2.0 nm or less at 220 nm, wavelength accuracy  $\pm 0.5$  nm or less at 220 nm and a photometric accuracy of  $\pm 0.5$  %T

2.2 *Quart cell*, Have path length of  $50 \pm 0.1$  mm and 10 mm

#### 3. Prepare Calibration Standard

3.1 Weigh accurately 0.1 g of Sulphur powder into a suitable container. Add base oil to make the total mass equal to 100 g. Cap/stopper the sample container and homogenize the solution.

3.2 Weigh accurately 0.2 g of Sulphur powder into a suitable container. Add base oil to make the total mass equal to 100 g. Cap/stopper the sample container and homogenize the solution.

3.3 Weigh accurately 0.3 g of Sulphur powder into a suitable container. Add base oil to make the total mass equal to 100 g. Cap/stopper the sample container and homogenize the solution.

#### 4. Procedure

##### 4.1 Prepare the calibration curve

4.1.1 Adjust the spectrophotometer to the optimum instrument settings, selecting the slit width to give a spectral bandwidth of 2.0 nm or less.

4.1.2 Fill two 50 mm cells with freshly base oil. Make sure the cell windows are clear. Place the cells in the cell compartment of the spectrophotometer and measure the absorbance at 600 nm. Set zero absorbance.

4.1.3 Empty the sample cell. Fill the 0.1%wt of Sulphur to the sample cell without changing the adjustment of the spectrophotometer measure the absorbance at 600 nm.

4.1.4 Empty the cells and rinse with Toluene solvent and Acetone solvent. Clean the cells and dry with cell tissue.

4.1.5 Same as 4.1.3 to 4.1.4. Measure absorbance of standard at 0.2%wt. and 0.3%wt. of Sulphur in base oil

4.1.6 Used the calibration data of various concentrations created calibration curve and created linear equation from this calibration curve.

## 4.2 Measurement sample

4.2.1 Adjust the spectrophotometer to the optimum instrument settings, selecting the slit width to give a spectral bandwidth of 2.0 nm or less.

4.2.2 Fill two 50 mm cells with freshly base oil. Make sure the cell windows are clear. Place the cells in the cell compartment of the spectrophotometer and measure the absorbance at 600 nm. Set zero absorbance.

4.2.3 Empty the sample cell. Fill sample to the sample cell without changing the adjustment of the spectrophotometer measure the absorbance at 600 nm.

4.2.4 Empty the cells and rinse with Toluene solvent and Acetone solvent. Clean the cells and dry with cell tissue.

4.2.5 Used the absorbance from reading 4.2.3 to find out the concentration of Sulphur by calculated from equation of 4.1.6



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## APPENDIX J

### PREDICTION ACCURACY OF THE PRESENT GENERAL CORRELATIONS

Table J-1 shows, the relative errors between the experimental mixing time and the calculated mixing time. The calculated mixing time is obtained from the correlation. The relative errors are based on the experimental mixing time.

From Figure 5.4-5.6, A linear regression analysis was provided by curve fitting as below :-

For temperature 80 °C with base oil type 1

CURVE EQUATION FORM		Y=A+B*X			
A	B	R-SQUARED	ADJ.R <sup>2</sup>		
+6433.283700	+9.401407	0.996849	0.993698		
OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	3111.2402	36000.0000	35683.3200	316.6797	0.88
2	4148.3203	44800.0000	45433.3320	-633.3320	1.41
3	5185.4004	55500.0000	55183.3480	316.6523	0.57
MEAN ABSOLUTE %ERROR		0.9546328			
MEAN SQUARE ERROR		200554.7			

For temperature 80 °C with base oil type 2

CURVE EQUATION FORM		Y=A+B*X			
A	B	R-SQUARED	ADJ.R <sup>2</sup>		
+9933.315400	+2.891494	0.995530	0.971060		
OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	8403.9648	34800.0000	34233.3280	566.6719	0.63
2	11205.2861	41200.0000	42333.3320	-713.3320	0.75
3	14006.6074	51000.0000	50433.3400	566.6602	0.11
MEAN ABSOLUTE %ERROR		0.830091			
MEAN SQUARE ERROR		64222.8			



For temperature 80 °C with base oil type 3

CURVE EQUATION FORM  $Y=A+B*X$

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+9466.625000	+1.721002	0.989106	0.978212

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	13422.4277	32100.0000	32566.6562	-466.6563	1.45
2	17896.5703	41200.0000	40266.6640	933.3359	2.27
3	22370.7129	47500.0000	47966.6760	-466.6758	0.98

MEAN ABSOLUTE %ERROR 1.567204

MEAN SQUARE ERROR 435556.8

For temperature 100 °C with base oil type 1

CURVE EQUATION FORM  $Y=A+B*X$

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+17833.322300	+9.298175	0.970050	0.940099

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	1855.2039	34500.0000	35083.3320	-583.3320	1.69
2	2473.6050	42000.0000	40833.3360	1166.6641	2.78
3	3092.0063	46000.0000	46583.3360	-583.3359	1.27

MEAN ABSOLUTE %ERROR 1.912237

MEAN SQUARE ERROR 680554

For temperature 100 °C with base oil type 2

CURVE EQUATION FORM  $Y=A+B*X$

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+5433.377400	+7.904857	0.998486	0.996972

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	3093.0310	29700.0000	29883.3438	-183.3438	0.62
2	4124.0415	38400.0000	38033.3360	366.6641	0.95
3	5155.0518	46000.0000	46183.3240	-183.3242	0.40

MEAN ABSOLUTE %ERROR 0.6569014

MEAN SQUARE ERROR 67221.74

For temperature 100 °C with base oil type 3

CURVE EQUATION FORM  $Y=A+B*X$

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+13766.644500	+2.986518	0.987666	0.915333

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	4972.3506	27600.0000	28616.6602	-101.6602	0.68
2	6629.8008	35600.0000	33566.6640	203.3359	0.71
3	8287.2510	37500.0000	38516.6720	-101.6671	0.72

MEAN ABSOLUTE %ERROR 2.035431

MEAN SQUARE ERROR 206725

For temperature 120 °C with base oil type 1

CURVE EQUATION FORM  $Y=A+B*X$

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+2366.680400	+4.191763	0.911865	0.823730

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	465.1955	4200.0000	4316.6699	-116.6699	2.78
2	620.2607	5200.0000	4966.6665	233.3335	4.49
3	775.3259	5500.0000	5616.6631	-116.6631	2.12

MEAN ABSOLUTE %ERROR 3.128728

MEAN SQUARE ERROR 27222.22

For temperature 120 °C with base oil type 2

CURVE EQUATION FORM  $Y=A+B*X$

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+11300.038100	+2.760165	0.989726	0.979452

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	923.8477	13800.0000	13850.0098	-50.0098	0.36
2	1231.7970	14800.0000	14700.0000	100.0000	0.68
3	1539.7462	15500.0000	15549.9902	-49.9902	0.32

MEAN ABSOLUTE %ERROR 0.453527

MEAN SQUARE ERROR 5000

For temperature 120 °C with base oil type 3

CURVE EQUATION FORM  $Y=A+B*X$

	A	B	R-SQUARED	ADJ.R <sup>2</sup>	
	+7097.364700	+15.113331	0.989468	0.978935	
OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	1161.2824	24300.0000	24648.2090	-348.2090	1.43
2	1548.3764	31200.0000	30503.0234	696.9766	2.23
3	1935.4705	36000.0000	36348.7700	-348.7695	0.97
MEAN ABSOLUTE %ERROR		1.545221			
MEAN SQUARE ERROR		242888.7			



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Table J-1 Prediction accuracy of data from table 5.4 by correlation

Viscosity mineral oil @100°C (cSt)	Temperature (°C)	Speed (rpm)	Mixing time, $t_m$ (min)		%Error
			Experiment	Calculation	
4.7	80	300	120	119	0.88
		400	112	114	-1.41
		500	111	110	0.57
11.2		300	116	114	1.63
		400	103	106	-2.75
		500	102	101	1.11
32.0		300	107	109	-1.45
		400	103	101	2.27
		500	95	96	-0.98
4.7	100	300	115	117	-1.69
		400	105	102	2.78
		500	92	93	-1.27
11.2		300	99	100	-0.62
		400	96	95	0.95
		500	92	92	0.00
32.0		300	92	95	-3.68
		400	89	86	3.37
		500	75	77	-2.71
4.7	120	300	14	14	0.00
		400	13	12	7.69
		500	11	11	0.00
11.2		300	46	46	0.00
		400	37	37	0.00
		500	31	31	0.00
32.0		300	81	82	-1.23
		400	78	76	2.56
		500	72	73	-1.39

## APPENDIX K

### SAMPLES OF CALCULATION

In this part the method of processing the data obtained from condition experiment and standard configuration of tank.

#### K.1 Sample calculation for Reynolds number, $Re$

The following is a condition for experiment.

-Diameter of impeller: 8.33 cm

-Viscosity of lubricating oil @80°C: 9.614 Poise ..... (Appendix E)

-Density of lubricating oil@80°C: 0.8621 g/cm<sup>3</sup> ..... (Appendix E)

-Speed of impeller: 300 rpm.

-Temperature: 80 °C

$$Re = \frac{D_i^2 \rho N}{\mu} \quad (K1)$$

whereas

$$D_i = 8.33 \text{ cm}$$

$$N = 300 \text{ rpm}$$

$$\rho = 0.8621 \text{ g/cm}^3$$

$$\mu = 9.614 \text{ Poise}$$

$$\begin{aligned} Re &= (8.33^2 \times 0.8621 \times 300) / (9.614 \times 10^{-2} \times 60) \\ &= 3.111 \times 10^3 \end{aligned}$$

#### K.2 Sample calculation for Sherwood number, $Sh$

$$Sh = \frac{KT}{D_v} \quad (K2)$$

whereas

- $K$  = Dissolution rate coefficient (cm/sec)  
 $T$  = Tank diameter (cm)  
 $D_v$  = Diffusion coefficients (cm<sup>2</sup>/sec)

### F2.1 Calculation of Dissolution rate coefficient ( $K$ )

The dissolution rate coefficients are calculated from the equation (K3) sample of dissolution rate coefficient calculation:

$$K = \frac{\left[ 1 - \left( \frac{W}{W_0} \right)^{1/3} \right] 3W_0^{1/3}}{\alpha_w n^{1/3} C_s t} \quad (K3)$$

whereas

- $K$  = Dissolution rate coefficient (cm/sec)  
 $\alpha_w$  = Shape factor relating the surface area with mass (Appendix F)  
 $W_0$  = Initial mass of solid (g)  
 $W$  = The mass remaining at time  $t$  (g)...(Appendix N)  
 $t$  = Time (sec)  
 $C_s$  = Saturation concentration (g/cm<sup>3</sup>)...(Appendix H)  
 $n$  = Number of particle  
 $n = \frac{6W}{d_p^3 \pi \rho_s}$ , when  $d_p$  = diameter of particle (cm)

$$\begin{aligned}
 K &= \frac{[1 - (0.029/0.3)^{1/3}] \times 3 \times 0.3^{1/3}}{0.96 \times 1053.3943^{1/3} \times 0.4944 \times 7200} \\
 &= 3.1251 \times 10^{-5} \text{ cm/sec}
 \end{aligned}$$

### K2.2 Calculation of Diffusion coefficient ( $D_v$ )

The diffusion coefficients are calculated from the equation (K4)

$$D_v = \frac{kT}{6\pi r\mu} \quad (K4)$$

whereas

$k$  = Boltzmann constant =  $1.38 \times 10^{-16}$  ergs/K

$r$  = Solute particle radius (cm)

$T$  = absolute temperature (K)

$\mu$  = Solvent viscosity (cSt or  $1 \times 10^{-2}$  cm<sup>2</sup>/s)

Sample of diffusion coefficients calculation:

$$\begin{aligned} D_v &= \frac{(1.38 \times 10^{-16} \times 353)}{6\pi \times 4.05 \times 10^{-3} \times 4.7} \\ &= 1.357 \times 10^{-13} \text{ cm}^2/\text{s} \end{aligned}$$

$$Sh = \frac{KT}{D_v} \quad (K2)$$

$$\begin{aligned} Sh &= \frac{(4.6888 \times 10^{-5})(25)}{(1.357 \times 10^{-13})} \\ &= 8.638 \times 10^9 \end{aligned}$$

### K.3 Sample calculation for Schmidt number, Sc

The following is a condition for experiment.

-Viscosity of lubricating oil @80°C: 9.614 Poise ..... (Appendix E)

-Density of lubricating oil@80°C: 0.8621 g/cm<sup>3</sup> ..... (Appendix E)

$$Sc = \frac{\mu}{\rho D_v} \quad (K5)$$

$$\begin{aligned} Sc &= \frac{(9.614 \times 10^{-2})}{(0.8621)(1.357 \times 10^{-13})} \\ &= 8.217 \times 10^{11} \end{aligned}$$

#### K.4 Sample calculation to find required mixing time

For batch experiment, data were prepared for finding required mixing time as follows:-

K.4.1 The average and standard deviation of a set of experiment (VK@100°C =4.7 cSt, Temperature 80°C, 300 rpm) was evaluated to set upper and lower limits.

K.4.2 All data were normalized by dividing with the average.

K.4.3 The upper limit, its mean plus 3 times of standard deviation, and the lower limit, its mean minus 3 times of standard deviation were calculated.

K.4.4 The shortest time that counts at that time and after was not exceed these limits was verified.

For another method of determining, the followings step had been done.

K.4.5 The standard deviation of a set of experiment was calculated by moving average method (Hald, 1952)

K.4.6 The shortest time that the standard deviation at that time and after was fairly constant was verified.

Some of data for analyzed data to find required mixing time are shown below:- ( for VK. @ 100 ° C =4.7 cSt., temperature 80 °C, 300 rpm.)



Table K-1 Analyzed data to find required mixing time

Time (Min)	Conc.of Sulphur (%Wt.)	Time (Min)	Conc.of Sulphur (%Wt.)	Time (Min)	Conc.of Sulphur (%Wt.)
0	0.000	26	0.188	52	0.209
1	0.295	27	0.187	53	0.201
2	0.294	28	0.190	54	0.200
3	0.294	29	0.200	55	0.197
4	0.294	30	0.201	56	0.196
5	0.293	31	0.200	57	0.191
6	0.292	32	0.195	58	0.192
7	0.293	33	0.178	59	0.195
8	0.291	34	0.169	60	0.193
9	0.291	35	0.146	61	0.195
10	0.200	36	0.133	62	0.197
11	0.190	37	0.130	63	0.199
12	0.190	38	0.123	64	0.201
13	0.186	39	0.121	65	0.202
14	0.181	40	0.119	66	0.201
15	0.181	41	0.144	67	0.203
16	0.180	42	0.155	68	0.206
17	0.180	43	0.190	69	0.207
18	0.179	44	0.201	70	0.209
19	0.180	45	0.213	71	0.211
20	0.179	46	0.209	72	0.216
21	0.179	47	0.220	73	0.219
22	0.181	48	0.223	74	0.220
23	0.186	49	0.225	75	0.225
24	0.190	50	0.221	76	0.231
25	0.190	51	0.218	77	0.238

Table K-1 Analyzed data to find required mixing time(continue)

Time (Min)	Conc.of Sulphur (%Wt.)	Time (Min)	Conc.of Sulphur (%Wt.)	Time (Min)	Conc.of Sulphur (%Wt.)
78	0.241	93	0.212	108	0.213
79	0.246	94	0.228	109	0.211
80	0.244	95	0.236	110	0.210
81	0.241	96	0.244	111	0.215
82	0.238	97	0.249	112	0.219
83	0.226	98	0.255	108	0.213
84	0.208	99	0.253	109	0.211
85	0.199	100	0.252	110	0.210
86	0.187	101	0.248	111	0.215
87	0.171	102	0.241	112	0.219
88	0.165	103	0.235	113	0.221
89	0.160	104	0.231	114	0.225
90	0.162	105	0.225	115	0.231
91	0.179	106	0.221	116	0.229
92	0.206	107	0.220	117	0.228

Mean = 0.1220

Standard deviation = 0.0012

Upper limit = 0.1256

lower limit = 0.1184

## APPENDIX L

### CURVE FITTING

To draw the curve in order to correlate two variables the least square method is used. The logarithmic functions of Sherwood number and Reynolds number are obtained as straight lines. The slope of each line indicates whether the variable has influence on the Sherwood number or not

#### 1. The influence of Reynolds Number

Figure 5.8-5.10 demonstrates the influence of Reynolds number for High Speed Shear mixer respectively. The curves are drawn by least square method. Data from table 5.8-5.10 are plotted. Each set of data gave one straight line. Table L-1 is a summary of the slope and intercept of each straight line. The average slope is 0.378 and the exponent ( $p$ ) of the Reynolds number in the correlation. This average slope is used to draw all curves again as shown in Figure 5.8-5.10

#### 2. The influence of Schmidt Number on Sherwood Number

Figure 5.11-5.13 demonstrates the influence of the Schmidt number for High Speed Shear mixer respectively. The curves are drawn by least square method. Data from table 5.11-5.13 are plotted the same as above. Table L-2 is a summary of the slope and intercept of each straight line. The fitted curves of the data have the average slope 0.454 and the exponent of the Schmidt number in the correlation for High Speed Shear mixer respectively.

Table L-1 Slope for relation between the Reynolds number and Sherwood number for  
High Speed Shear mixer

Data from tables	Temperature	Base oil type	Slope	Intercept
5.8	80 °C	Type 1	0.021	$-2.450 \times 10^{11}$
		Type 2	0.087	$-1.271 \times 10^{11}$
		Type 3	1.603	$-2.059 \times 10^{10}$
5.9	100 °C	Type 1	0.021	$-1.970 \times 10^{11}$
		Type 2	0.065	$-1.850 \times 10^{11}$
		Type 3	1.083	$-3.260 \times 10^{10}$
5.10	120 °C	Type 1	0.007	$-6.486 \times 10^{11}$
		Type 2	0.061	$-1.592 \times 10^{11}$
		Type 3	0.452	$-8.553 \times 10^{10}$
Average			0.378	

Table L-2 Slope for relation between the Schmidt number and Sherwood number for  
High Speed Shear mixer

Data from tables	Slope	Intercept
5.11	0.455	$3.569 \times 10^4$
5.12	0.457	$3.489 \times 10^4$
5.13	0.451	$4.854 \times 10^4$
Average	0.454	

## APPENDIX M

### DETERMINATION OF THE CONSTANT $r$ IN THE CORRELATIONS

From the result the correlations are expressed in the form

$$Sh_T = r Re_a^p Sc_q$$

The values of  $r$  depend on the solid-liquid systems. To determine the constant,  $r$  the terms  $\frac{Sh}{Sc_q}$  and  $Re_a$  are plotted on log-log scale. The relations give straight line having same slope 0.691. The constant,  $r$  is obtained by calculating the values of antilogarithmic of the intercepts. Table M-1 show the results by the least squares method as described in Appendix L. The value of  $A$  is the intercept of least square line. From table M-2 to M-4 the constant,  $r$  obtained as follow:

Table M-1 The constant,  $r$  from calculating by least square method

System	Constant , $r$
1. 0.3wt% of Sulphur in mineral oil at 80°C	5.407x10 <sup>4</sup>
2. 0.3wt% of Sulphur in mineral oil at 100°C	7.359x10 <sup>4</sup>
3. 0.3wt% of Sulphur in mineral oil at 120°C	1.101x10 <sup>5</sup>

Table M-2 Calculate of least square line to evaluate the constant,  $r$ 

Data from Table 5.8 and 5.11

The constant,  $r$  is Anti-log  $A = 5.407 \times 10^4$ 

Base oil	Speed	$Re_a \times 10^3$	$Sh \times 10^{10}$	$Sc \times 10^{11}$	$cRe = \frac{Sh}{Sc}^{0.454}$	X	y
						Log (Re)	Log (cRe)
Type 1	300	3.111	0.576	8.217	2.243	3.493	4.351
	400	4.148	0.587	8.217	2.288	3.618	4.359
	500	5.185	0.619	8.217	2.411	3.715	4.382
Type 2	300	1.855	1.282	32.837	2.663	3.268	4.425
	400	2.474	1.304	32.837	2.710	3.393	4.433
	500	3.092	1.390	32.837	2.887	3.490	4.460
Type 3	300	0.465	4.002	374.155	2.755	2.668	4.440
	400	0.620	4.383	374.155	3.017	2.793	4.480
	500	0.775	4.499	374.155	3.097	2.889	4.491

Least square method is obtained by calculating the value of anti logarithmic of intercept as below:

CURVE EQUATION FORM  $Y = A + B \cdot X$ 

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+4.732950	-0.094641	0.508267	0.438019

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	3.4930	4.3510	4.4024	-0.0514	1.18
2	3.6180	4.3590	4.3905	-0.0315	0.72
3	3.7150	4.3820	4.3814	0.0006	0.01
4	3.2680	4.4250	4.4237	0.0013	0.03
5	3.3930	4.4330	4.4118	0.0212	0.48
6	3.4900	4.4600	4.4027	0.0573	1.29
7	2.6680	4.4400	4.4804	-0.0404	0.91
8	2.7930	4.4800	4.4686	0.0114	0.25
9	2.8890	4.4910	4.4595	0.0315	0.70

MEAN ABSOLUTE %ERROR 0.6197918

MEAN SQUARE ERROR 1.125355E-03

Table M-3 Calculate of least square line to evaluate the constant,  $r$ 

Data from Table 5.9 and 5.12

The constant,  $r$  is Anti-log  $A= 7.359 \times 10^4$ 

Base oil	Speed	$Re_a \times 10^3$	$Sh \times 10^{10}$	$Sc \times 10^{11}$	$cRe = \frac{Sh}{Sc}^{0.454}$	X	y
						Log (Re)	Log (cRe)
Type 1	300	8.404	0.601	2.879	3.770	3.924	4.576
	400	11.205	0.633	2.879	3.970	4.049	4.599
	500	14.007	0.719	2.879	4.512	4.146	4.654
Type 2	300	3.093	1.408	18.640	3.784	3.490	4.578
	400	4.124	1.493	18.640	4.010	3.615	4.603
	500	5.155	1.543	18.640	4.146	3.712	4.618
Type 3	300	0.924	4.565	163.819	4.572	2.966	4.660
	400	1.232	4.794	163.819	4.802	3.091	4.681
	500	1.540	5.232	163.819	5.240	3.187	4.719

Least square method is obtained by calculating the value of anti logarithmic of intercept as below:

CURVE EQUATION FORM  $Y=A+B \cdot X$ 

A	B	R-SQUARED	ADJ.R <sup>2</sup>
+4.866827	-0.065675	0.324034	0.227467

OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	3.9240	4.5760	4.6091	-0.0331	0.72
2	4.0490	4.5990	4.6009	-0.0019	0.04
3	4.1460	4.6540	4.5945	0.0595	1.28
4	3.4900	4.5780	4.6376	-0.0596	1.30
5	3.6150	4.6030	4.6294	-0.0264	0.57
6	3.7120	4.6180	4.6230	-0.0050	0.11
7	2.9660	4.6600	4.6720	-0.0120	0.26
8	3.0910	4.6810	4.6638	0.0172	0.37
9	3.1870	4.7190	4.6575	0.0615	1.30

MEAN ABSOLUTE %ERROR 0.661781

MEAN SQUARE ERROR 1.459253E-03

Table M-4 Calculate of least square line to evaluate the constant, *r*

Data from Table 5.10 and 5.13

The constant, *r* is Anti-log A= 1.101x10<sup>5</sup>

Base oil	Speed	Re <sub>a</sub> x10 <sup>4</sup>	Shx10 <sup>10</sup>	Scx10 <sup>11</sup>	cRe= Sh/Sc <sup>0.454</sup>	X	y
						Log (Re)	Log (cRe)
Type 1	300	1.342	0.539	1.711	4.282	4.128	4.632
	400	1.790	0.580	1.711	4.611	4.253	4.664
	500	2.237	0.601	1.711	4.772	4.350	4.679
Type 2	300	0.497	1.287	11.005	4.392	3.697	4.643
	400	0.663	1.366	11.005	4.663	3.822	4.669
	500	0.829	1.490	11.005	5.084	3.918	4.706
Type 3	300	0.116	4.368	95.897	5.579	3.065	4.747
	400	0.155	4.617	95.897	5.897	3.190	4.771
	500	0.194	4.718	95.897	6.026	3.287	4.780

Least square method is obtained by calculating the value of anti logarithmic of intercept as below:

CURVE EQUATION FORM		Y=A+B*X			
A	B	R-SQUARED	ADJ.R <sup>2</sup>		
+5.041634	-0.091477	0.617099	0.562398		
OBSN	X	Y	FITTED-Y	RESID.ERROR	%ERROR
1	4.1280	4.6320	4.6640	-0.0320	0.69
2	4.2530	4.6640	4.6526	0.0114	0.24
3	4.3500	4.6790	4.6437	0.0353	0.75
4	3.6970	4.6430	4.7034	-0.0604	1.30
5	3.8220	4.6690	4.6920	-0.0230	0.49
6	3.9180	4.7060	4.6832	0.0228	0.48
7	3.0650	4.7470	4.7613	-0.0143	0.30
8	3.1900	4.7710	4.7498	0.0212	0.44
9	3.2870	4.7800	4.7409	0.0391	0.82
MEAN ABSOLUTE %ERROR		0.6144454			
MEAN SQUARE ERROR		1.031005E-03			



## APPENDIX N

### THE MASS OF SULPHUR REMAINING IN LUBRICATING OILS AT TIME 120 MIN

Table N-1 The mass of Sulphur remaining in lubricating at time 120 min

Temperature (°C)	Speed	The mass remaining at time 120 min (g)		
		@ 100 °C = 4.7 cSt	@ 100 °C = 11.2 cSt	@ 100 °C = 32.0 cSt
80	300	0.029	0.041	0.053
	400	0.027	0.039	0.042
	500	0.022	0.032	0.039
100	300	0.010	0.014	0.019
	400	0.007	0.010	0.015
	500	0.002	0.008	0.009
120	300	0.006	0.008	0.009
	400	0.003	0.005	0.006
	500	0.002	0.002	0.005

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## VITA

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