DETERMINATION OF KINEMATIC VISCOSITY OF DIFFERENT BIODIESEL FUELS AT LOW TEMPERATURES

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ABSTRACT

The use of biodiesel as an alternative fuel for diesel engine still has some challenges. One of the main challenges is being a significant amount of unsaturated fatty acid compounds because it is derived from vegetable oils and fats. Therefore, viscosity of biodiesel is affected by fatty acid composition, temperature, pressure, chain length and degree of saturation. The most serious problem that is faced to biodiesel is its utilization at low temperatures. There is a contrary relationship between viscosity and temperature. Viscosity increases by decreasing temperature. Additionally, the cloud point and pour point of biodiesel are higher than petrodiesel. Due to these reasons, there is a need to determine the biodiesel properties, especially at low temperatures such as viscosity and cold flow properties because; the major concern about biodiesel is its use at low temperatures. The kinematic viscosity and cloud point and pour point of five biodiesel fuel blends (100% UCOME, 75% UCOME + 25% UFOME, 50% UCOME + 50% UFOME, 25% UCOME + 75% UFOME and 100% UFOME) are measured from 20 ^{0}C down to -10 ^{0}C. The variations of these properties with temperature and blend composition are also observed.

Keywords: Biofuels, Biodiesel, Kinematic Viscosity, Cold Flow Properties, Cloud Point, Pour Point, Frying Oil, Canola Oil.

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LIST OF SYMBOLS USED

LIST OF QUANTITIES

A	Area
D	Capillary diameter
dv	Changing in velocity
dx	Changing in separation height
E_a	Activation energy for flow
F	Force
g	Acceleration due to gravity
G	Universal gas constant
h	Plank's constant
H	Capillary height
K	Viscometer constant
L	Length of viscometer
N_A	Avogadro's number
p	Flow pressure
Q	Flow rate
R	Capillary radius
r	Radian length
t	Time
T	Absolute temperature
T_{cp}	Cloud point temperature
T_{pp}	Pour point temperature
V	Volume
v	Velocity
v	Flow velocity
v_r	Velocity in radian direction
v_z	Velocity in flow direction
v_θ	Velocity in angular direction
x	Elemental length

y	Correction factor
z	Length in flow direction
θ	Angular length
ρ	Density
μ	Dynamic viscosity
ν	Kinematic viscosity
σ	Shear stress
ε	Strain rate

ABBREVIATIONS USED

Ave.	Average
ASTM	American Society for Testing and Materials
CGS	Centimeter-gram-second
CFPP	Cold Filter plugging Point
CP	Cloud Point
DIN	German Institute for Standardization
EN	European Standard
FAME	Fatty Acid Methyl Ester
HC	Kinetic Energy Correction (Hagenbach-Couette Korrektion)
ISO	International Standard Organization
PP	Pour Point
St	Stokes
SSU	Saybolt Universal Seconds
UCO	Used Canola Oil
UFO	Used Frying Oil
UCOME	Used Canola Oil Methyl Ester
UFOME	Used Frying Oil Methyl Ester

CHAPTER 1

INTRODUCTION

It is estimated that, the world will need 50% more energy in 2030 than today. In the past 30 years, the transportation sector has experienced a steady growth especially due to the increasing numbers of cars around the world. The global transportation energy use is expected to increase by an average of 1.8% per year from 2005 to 2035. It is expected that about 12.7 billion metric tons of carbon dioxide (CO_2) will be released to the atmosphere from 2007 to 2035 [1]. The rising prices day after day of crude oil, environmental degradation and the possibility depletion of fossil fuel in the future have led to systematic efforts by many researchers to determine the suitability of vegetable oil and its derivatives (biodiesel) as an alternative to fossil fuels or blend to the diesel [2, 3].

Biodiesel is derived from vegetable oils and animal fats, which is defined as the monoalkyl esters of long chain fatty acids [2, 4, 5, 6]. It is gaining attention as an alternative fuel and is typically obtained by transesterification of vegetable oils; animal fats or used frying oils that comprise mainly of triglycerides. The transesterification is achieved with monohydric alcohols like methanol and ethanol in the presence of an alkali catalyst [4]. The most used alcohol is methanol, which creates a mixture of fatty acid methyl esters (FAME). Biodiesel can be used mixed with petrodiesel or alone in a diesel engine. Due to the worldwide increase in the production and use of this biofuel, it is extremely important to be able to estimate the dependence of its physicochemical properties with the temperature [7].

The properties of biodiesel fuel are determined by the amounts of each fatty acid that is present in its molecules, so a change in the nature of the fatty acid profile, in biodiesel leads to overall changes in the fuel properties [8, 9]. Transesterification does not change the fatty acid composition of the feedstock so this composition plays an important role to determine some main parameters of the biodiesel like viscosity and cold flow properties. Chain length, branching of the chain, and degree of saturation mostly affects the physical and fuel properties of a fatty ester molecule [8, 10].

One of the most important properties of biodiesels is viscosity because it affects the fuel injection system, especially in cold weather because of the increase of viscosity with a decreasing temperature [4, 8, 11]. Kinematic viscosity is reduced by shorter fatty acids chain length and the presence of cis (when the two hydrogen atoms are on the same side as a double bond) [8, 12]. A biodiesel of high viscosity causes to form larger droplets upon injection and then causes poorer atomization, poor combustion and increasing emission. While a low viscosity biodiesel may not provide enough lubrication for the fuel injection pumps, resulting in leakage or increased wear [11]. The viscosity of biodiesel is approximately 1.5 times more than petrodiesel [4] and it must be in the range of 1.9-6.0 mm^2/s (ASTM D6751) and 3.5-5.0 mm^2/s (EN 14214) at 40^0C [1, 5, 11, 12] as shown in Table 1.1. All the biodiesel properties must meet the ASTM D-6751 [6] and EN 14214 [13] specifications in USA and Europe, respectively [5, 14].

Table 1.1 Kinematic viscosity at 40 ^0C in biodiesel and petrodiesel fuel standards [12, 15].

Standard	Location	Fuel	Method [a]	Kinematic viscosity (mm^2/s)
ASTM D975	United States	Petrodiesel	ASTM D445	1.9-4.1 [b]
ASTM D6751	United States	Biodiesel	ASTM D445	1.9-6.0
EN 590	Europe	Petrodiesel	ISO 3104	2.0-4.5
EN 14214	Europe	Biodiesel	ISO 3104	3.5-5.0

[a] ASTM = American Society for Testing and Materials; ISO = International Standard Organization

[b] Specification for low-sulfur No.2 diesel fuel to which biodiesel is usually compared. Specification for No.1 diesel fuel is 1.3-2.4 mm^2/s.

Biodiesel possesses some advantages over petroleum diesel, such as reducing global warming gas emissions, hydrocarbons (HC), carbon monoxide (CO) and other air toxics [4, 16, 17]. Biodiesel improves lubricity and reduces premature wearing of fuel pumps [4]. The environmental benefits of biodiesel are renewability, biodegradability and cleaner burning [8,11]. It has a higher flash point that makes it easier to store [8].

Regardless of advantages, biodiesel faces some technical challenges such as reducing of nitrogen oxides (NO_x) exhaust emission and improving oxidative stability but the main two more important problems of biodiesel are cold flow properties [4, 12, 18] and high cost [1, 3, 5]. Biodiesel starts to become gel at low temperature, which makes the filters to be clogged or even become so thick that it cannot be pumped from the fuel tank to the engine [4] due to an increase in viscosity with decreasing of temperature [11]. The viscosity of biodiesel is slightly greater than that of petrodiesel but approximately an order of magnitude less than that of the parent vegetable oil or fat [12].

Cloud point (CP) and pour point (PP) are two main parameters of the cold flow properties. The cloud point, which usually occurs at a higher temperature than the pour point, is the temperature at which a liquid fatty material becomes cloudy due to the formation of crystals and solidification of saturates. Crystallization of the saturated fatty acid methyl ester components of biodiesel during cold seasons causes fuel starvation and operability problems as solidified material clog fuel lines and filters. With decreasing temperature more solids form and material approaches the pour point, the lowest temperature at which it will cease to flow [1, 2]. Unlike diesel fuel, the esters have relatively high cloud point and pour point. While the cloud point and pour point of diesel fuel are around -15 ^{0}C and -27 ^{0}C respectively, the respective values for FAME are about 15–25 ^{0}C higher [19].

Recently, more than 95% of the world biodiesel is produced from edible oils [1] such as soybean, rapeseed, canola, palm, and sunflower oils. These oils can replace only a few percent of the petrodiesel market because the biodiesel from these feedstocks is more expensive than petrodiesel [5, 20]. Using edible oils causes food versus crisis [1] because it is a potential source for food so they should not be used for fuel purposes due to effects on food prices and land-use change [3].

When vegetable oils are used for frying of food materials and in the presence of moisture and air during heat processing is thermolytically degraded with time. These develop high free fatty acid contents, which make them, unfit for human consumption and the two main factors which affect the cost of biodiesel are the high cost of vegetable oil and the cost of production process. Frying or used cooking oil can be a good source for the production of biodiesel [3, 5, 20] to reduce the using of edible oil because feedstock alone represents

75% of the overall biodiesel production cost as shown in Figure 1.1 [1].

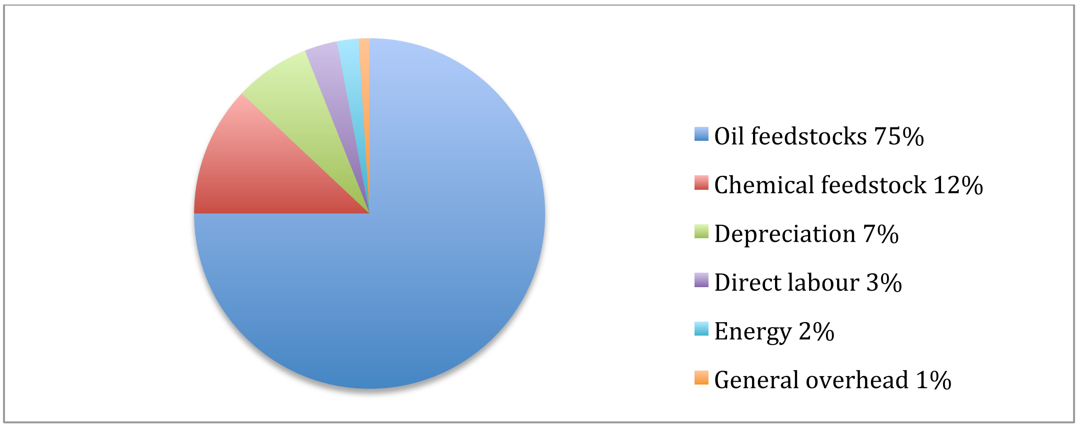

Figure 1.1 Biodiesel production cost [1].

1.1 Literature Review

There are some papers in the literatures on the properties of biodiesel blended with petrodiesel or with another kind of biodiesel at low temperatures. A report on the kinematic viscosity of biodiesel and some of its components are a study of the low temperature viscosities of biodiesel/diesel blends [21], a report on the low temperature properties (-3 to 15 ^{0}C) of soybean, used soybean, mustard, and used mustard oils [22], a report on two soy methyl esters, one of them genetically modified at (2–100) ^{0}C in steps of 20 ^{0}C [23], a work has been reported on the kinematic viscosity of biodiesel and a variety of fatty acid alkyl esters at temperature from 40 ^{0}C down to -10 ^{0}C in increments of 5 ^{0}C and compared with petrodiesel [18].

Cold flow properties of neat esters of branched chain alcohols with fatty acids and blends of these esters with fossil diesel fuel are another study. According to this study fossil fuel blending with fatty esters of branched alcohols up to 10-volume % does not substantially change the cold flow properties of fossil fuel [19]. By transforming Eyring's equation into this equation ($\ln \nu = a + bn_1 + c/T + dn_1/T$ where, a, b, c and d, T, n_1 and ν are thermodynamically related constant, absolute temperature, mole fraction and kinematic viscosity of biodiesel, respectively), another work has been done to predict kinematic viscosity of biodiesel blends of different degree of blending by using above equation at any temperatures from pour point to 100 ^{0}C. According to this work, the numeric values for (a, b, c and d) are changed as the composition of biodiesel are changed [24]. Another study has been conducted to investigate the effect of temperature and blending percentage of Jatropha based biodiesel on the viscosity and specific gravity in the temperature range 15 – 60 ^{0}C together with the pure fuels. In this study, the viscosity of Jatropha biodiesel was found to be 42.09% higher than that of number two-diesel fuel [17]. A very recent work has been done to evaluate the viscosity and cloud point of binary mixtures caster oil biodiesel, palm oil biodiesel and diesel fuel, in this study, the palm oil biodiesel caused a problematic high cloud point, while the caster oil biodiesel could lower the cloud point but increased the viscosity of the blends [8].

There are also some works in the literature on the kinematic viscosity of some biodiesels at various temperatures and 40 ^{0}C, which they are out of the range of this work [10, 25, 26]. Two other papers, presented several models that they were previously proposed to predict the viscosity of biodiesels and their blend with other fuels such as: Andrade's, Ceriani's, Krisnangkura's, and Yuan's models [11, 27]. There is a work to study the variation of density and kinematic viscosity as a function of percent volume and temperature by mixing biodiesel and ultra low sulfur diesel, which density and kinematic viscosity increase by increasing in the concentration of biodiesel, and both of them decrease as temperature increases [28]. There were not found any previous work on determining the viscosity and cold flow properties of biodiesel derived from used frying oil (UFO) and used canola oil (UCO).

1.2 Purpose

The aim of this work is to determine experimentally the kinematic viscosity and cold flow properties (cloud point and pour point) of five different blends of biodiesel fuel derived from methyl esters used frying oil (UFO) and used canola oil (UCO) at low temperatures (from 20 ^{0}C down to -10 ^{0}C), which were produced in (April, 2012) in northern Cyprus. This work is concerned to determine the effect of both temperature and blend composition on the biodiesel properties.

CHAPTER 2

THEORY AND METHODS

Theoretically studying of viscosity and cold flow properties of biodiesel is very important to understand the effecting of temperature on the viscosity and both cloud point (CP) and pour point (PP).

2.1 Viscosity

Viscosity is a fundamental characteristic property of all fluids [29]. It is the integral of the interaction forces of molecules. In a solid case the interaction forces among molecules are very strong, they cannot slide over each other. When energy or heat is applied up to a certain level, molecules can then slide over each other or become melted. Initially, they slide over each other very slowly. If the amount of heat or temperature greatly exceeds the melting point, they move pass each other very rapidly and the liquid becomes less viscous [27, 30].

When a liquid flows, there is an internal friction or resistance to flow, which is mainly, depends on the interaction forces of molecules. Viscosity is a measure of this friction or resistance to flow [28, 29, 30]. Viscosity is governed by the strength of intermolecular forces and especially by the shapes of the molecules of a liquid. Liquids whose molecules are polar or can form hydrogen bonds are usually more viscous than similar nonpolar substances [31].

2.1.1 Types of Viscosity

Viscosity is expressed in two distinct types [29, 32]:

1. Dynamic (absolute) viscosity
2. Kinematic viscosity

Dynamic (absolute) viscosity is the tangential force per unit area required to move one layer against another layer at unit velocity as shown in Figure 2.1 when the two layers are maintained at a unit distance. In Figure 2.1, force F causes upper and lower layers to slide in a relative velocity [29, 32].

Since the viscosity of a fluid is defined, as the measure of how resistive the fluid is to flow [33], in mathematical form, it can be described as:

Shear stress = μ *Strain rate

Where μ is the dynamic viscosity.

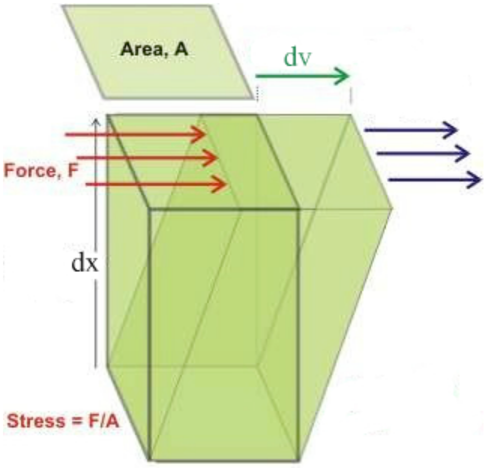

Figure 2.1 Shear of a liquid layer [33].

If (σ) is shear stress and (ε) is strain rate, then the expression becomes:

$$\mu = \frac{\sigma}{\varepsilon} \tag{2.1}$$

If changing in velocity will be denoted by dv and changing in separation height by dx and the strain rate is generally expressed as:

$$\varepsilon = \frac{1}{x}\frac{dx}{dt} = \frac{v}{x} \qquad 2.2$$

where: x is the length
t is the time
v is the velocity

Because dx/dt is velocity (v)
So, the dynamic viscosity can be written as

$$\mu = \sigma\frac{x}{v} \qquad 2.3$$

On the other hand, the shearing stress between the layers of non-turbulent fluid moving in straight parallel lines can be defined for a Newtonian fluid as [32]:

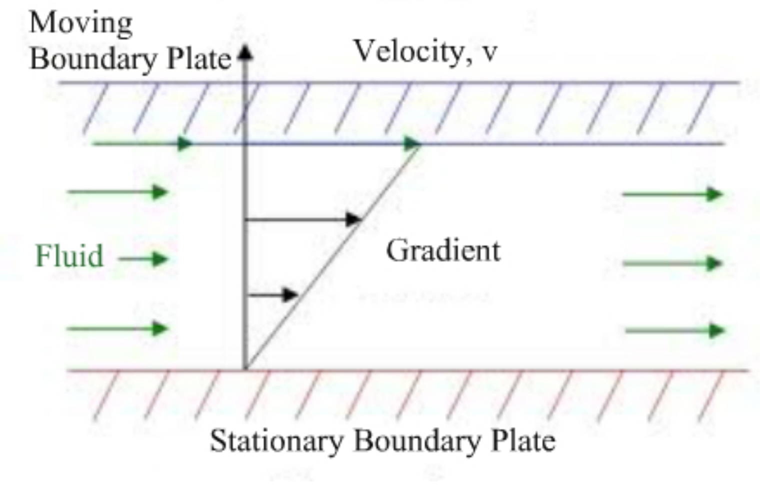

Figure 2.2 Shear stress of Newtonian fluid [34].

Figure 2.2 represents shear stress as proportional to the strain rate. The dynamic viscosity can be expressed like

$$\sigma = \mu\frac{dv}{dx} \qquad 2.4$$

Fluids for which the shearing stress is linearly related to the rate of shearing strain are designated as Newtonian Fluids. Newtonian materials are referred to as true liquids since their viscosity or consistency is not affected by shear such as agitation or pumping at a constant temperature [32]. The viscosity of Newtonian fluids is independent of shear strain rate and a plot of shear strain rate against shear stress is linear and passes through the origin [33] as shown in Figure 2.3.

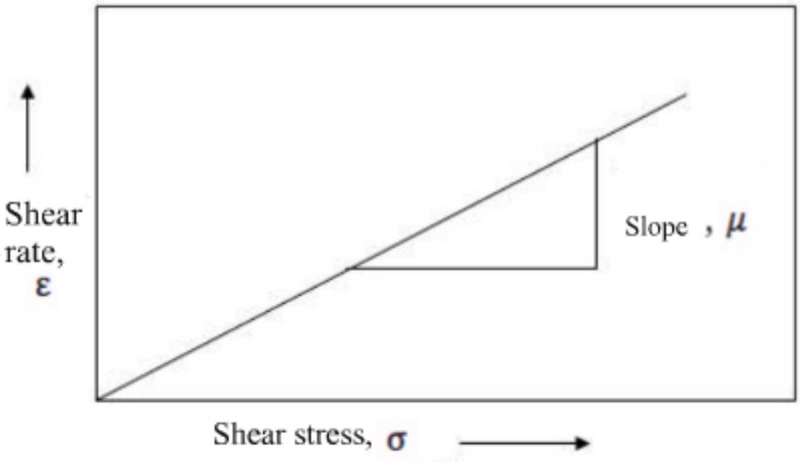

Figure 2.3 Shear stress and shear strain relations.

Kinematic viscosity is dynamic viscosity over density (density is obtained by dividing the mass of the fluid by the volume of the fluid) at that temperature and pressure - a quantity in which no force is involved. It can be obtained by dividing the absolute viscosity of a fluid with its mass density so; it is requires knowledge of density of the liquid (ρ) [29, 32, 35] and is defined as

$$\nu = \frac{\mu}{\rho} \qquad 2.5$$

Where:

ν is kinematic viscosity, μ is dynamic viscosity and ρ is density of the fluid

2.1.2 Viscosity Units and Conversion

Common units for viscosity are poise (P), Stokes (St), Saybolt Universal Seconds (SSU) and degree Engler (degree Engler is used in Great Britain as a scale to measure kinematic viscosity). The most suitable unit to dynamic viscosity of liquids is Centipoise (cP). It is 1/100 of Poise.

In the SI System (Système International d'Unités) the dynamic viscosity units are (N·s/m^2), (Pa·s) or (kg/m·s)

Where N is Newton, Pa is Pascal and s is second
1 Pa·s = 1 N·s/m^2 = 1 kg/m·s

In the metric system CGS (centimeter-gram-second) the dynamic viscosity units are (g/cm·s), (dyne·s/cm^2) or (poise (P)) where,

1 poise = dyne·s/cm^2 = g/cm·s = 1/10 Pa·s

In British system of units, the dynamic viscosity is expressed in (lb/ft·s) or (lb·s/ft^2).
In brief, the unit of dynamic viscosity is Force / area x time.

For the SI system, kinematic viscosity is reported using Stokes (St). The kinematic viscosity is expressed as Stokes (St) or m^2/s, where 1 stoke =10^{-4} m^2 /s. Stokes is a large unit; it is usually divided by 100 to give the unit called Centistokes (cSt) [29, 32].

Where:

1 St = 10^{-4} m^2/s
1 St = 100 cSt.
1 cSt = 10^{-6} m^2/s

2.1.3 Factors Influence Viscosity

Generally there are some factors, which affect the Newtonian fluid such as temperature, pressure and composition. Additionally biodiesel as a Newtonian fuel is affected by some more factors like chain length and degree of saturation. Viscosity is inversely proportional to temperature [36]. It will increase with decreasing temperature [4, 5, 28, 37]. Viscosity increases with chain length (number of carbon atoms) and with increasing degree of saturation [10, 17, 36]. Also viscosity is affected by composition, for instance, the viscosity of biodiesel is less than that of the parent vegetable oil or fat [8, 12, 35] because of using alkyl esters in producing biodiesel fuel by transesterification [18].

2.1.4 Measuring of Viscosity

The measurement of viscosity is of significant importance in both industry and academia. Accurate knowledge of viscosity is necessary for various industrial processes. Various theories that are developed for prediction or estimation of viscosity must be verified using experimental data [29]. Viscosity is measured by a device, which is called viscometer. Various types of viscometers and rheometers are used for measuring viscosity. Rheometer is used for those fluids that cannot be defined by a single value of viscosity and therefore require more parameters to be set and measured than in the case for a viscometer [38]. Capillary viscometers are most widely used for measuring viscosity of Newtonian liquids.

2.2 Viscometers

Viscometers are used for measuring viscosity of Newtonian fluids. Viscometers used to measure the viscosity of liquids can be broadly classified into seven categories [29]:

1. Capillary viscometers
2. Orifice viscometers
3. High temperature high shear rate viscometers
4. Rotational viscometers
5. Falling ball viscometers
6. Vibrational viscometers

7. Ultrasonic viscometers

One of the most common instruments for measuring kinematic viscosity is the glass capillary viscometer [29, 38].

2.2.1 Capillary Viscometers

The most accurately determined of the viscosity of Newtonian fluids is by using capillary viscometers [29, 39, 40]. They are simple in operation, easy temperature control; require a small volume of sample liquid, and cheap. In capillary viscometers, the sample liquid flowing through a fine bore (capillary) is measured, usually by taking the time required for a known volume of liquid to pass through two marked levels as shown in Figure 2.4. The liquid may flow through the capillary tube either under the influence of gravity (Gravity Type Viscometer) or an external force. However, most of the capillary viscometers must be first calibrated with one or more liquids of known viscosity to obtain “constants” for that particular viscometer [29] but the viscometers used in this work are calibrated by the manufacturer.

Figure 2.4 Capillary viscometer [39].

Several types of capillary viscometers have been designed. Glass capillary viscometers are most suitable for measuring of the viscosity of Newtonian liquids [29,38]. Kinematic viscosity is generally measured by using these viscometers [29].

Glass capillary viscometers allow a very accurate determination of viscosities using standard testing methods like ASTM, DIN and ISO. There are some models of glass capillary viscometers such as Ubbelohde (It’s been used in this work and will be explained in detail later in this chapter), Cannon-Fenske and U-tube [40].

2.2.2 Theory of Capillary Viscometers

Consider fully developed laminar flow through a straight vertical tube of circular cross section as shown in Figure 2.5. Rotational symmetry is considered to make the flow two-dimensional axisymmetric and z-axis is taken as the axis of the tube along which all the fluid particles travel [41], that mean

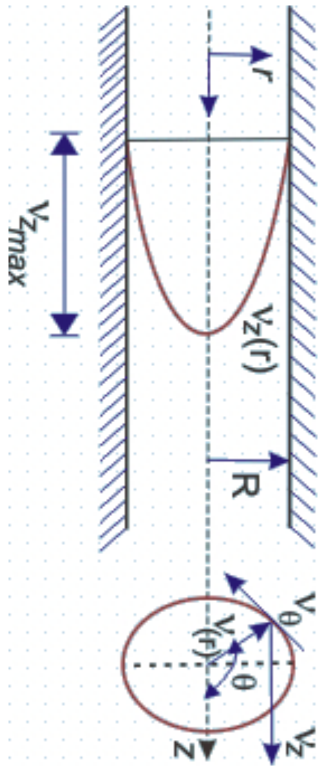

Figure 2.5 Hagen -poiseuille flow [41].

$$v_z \neq 0,\ v_r = 0,\ v_\theta = 0 \qquad 2.6$$

Now, from continuity equation in cylindrical coordinate

$$\underbrace{\frac{\partial v_r}{\partial r}}_{0} + \underbrace{\frac{\partial v_r}{r}}_{0} + \frac{\partial v_z}{\partial z} = 0 \qquad 2.7$$

For rotational symmetry

$$\frac{1}{r} \cdot \frac{\partial v_\theta}{\partial \theta} = 0 \qquad 2.8$$

$$\frac{\partial v_z}{\partial z} = 0 \quad \text{which means} \quad v_z = v_z(r, t) \qquad 2.9$$

Inserting

$$v_r = 0 \quad , \quad v_\theta = 0 \quad , \quad \frac{\partial v_z}{\partial z} = 0 \quad \text{and} \quad \frac{\partial}{\partial \theta}(\text{any quantity}) = 0$$

in the Navier-Stokes equations in cylindrical coordinate system in z direction, can be obtained

$$\frac{\partial v_z}{\partial t} = -\frac{1}{\rho} \cdot \frac{\partial p}{\partial z} + v\left(\frac{\partial^2 v_z}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial v_z}{\partial r}\right) \quad \text{in z direction} \qquad 2.10$$

For steady flow, the governing equation becomes

$$\frac{\partial^2 v_z}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial v_z}{\partial r} = \frac{1}{\mu}\frac{dp}{dz} \qquad 2.11$$

By using boundary conditions and solving Equation 2.11

At

$r = 0$; v_z is finite

$r = R\ ; \qquad v_z = o$

It can be obtained Equation 2.12

$$v_z = \frac{R^2}{4\mu}\left(-\frac{dp}{dz}\right)\left(1-\frac{r^2}{R^2}\right) \tag{2.12}$$

While $$-\frac{dp}{dz} = \frac{\Delta p}{L} \tag{2.13}$$

It may be noted from Figure 2.5 that the velocity distribution across the capillary is parabolic. The overall flow rate (Q) can be obtained by integrating the following expression [29].

$$Q = \int_0^R 2\pi v_z\, r\, dr \tag{2.14}$$

By substituting both 2.12 and 2.13 into 2.14, it's obtained

$$Q = \pi\frac{R^4}{8\mu}\left(\frac{\Delta p}{L}\right) \tag{2.15}$$

This is known as Poiseuille's equation and is used for calculation of viscosity when using a capillary viscometer.

$$Q = \frac{V}{t} \tag{2.16}$$

where:

Q is overall flow rate

V is volume

t is time

$$\upsilon = \frac{\mu}{\rho} \tag{2.17}$$

For vertical tube arrangement, which is the case for most of the capillary viscometer, the hydrostatic pressure, **ρ**gh, depends on the height, h, of the liquid. Therefore, the pressure difference, **Δ**p, in terms of hydrostatic pressure is given by

$$\Delta p = \rho g H$$

then,

$$\upsilon = \frac{\pi g H R^4}{8LV} \cdot t \qquad 2.18$$

K is a constant for viscometer

$$K = \frac{\pi g H R^4}{8LV} \qquad 2.19$$

or

$$K = \frac{\pi g H D^4}{128LV} \qquad 2.20$$

so,

$$\upsilon = Kt \qquad 2.21$$

A number of viscometers are designed based on Equation 2.21. The instrument is calibrated for the value of K, which is obtained by using a liquid of known viscosity and density with Equation 2.22. Once the value of K is known, the viscosity of test liquid can be obtained by measuring the time required for a known volume of sample to flow between two graduation marks [29].

$$\mu = K\rho t \qquad 2.22$$

2.2.3 Kinetic Energy Corrections (HC)

Some factors can affect the experiment and gives errors in the measurements. To improve the accuracy of the measurement, various corrections are made to the experimentally determined data [29]. The most significant factor is kinetic energy correction HC (Hagenbach-Couette korrektion) [42]. For the absolute measurements the kinetic energy correction HC is subtracted from the determined efflux time and the Equation 2.21 becomes as

$$\upsilon = K(t - y) \qquad 2.23$$

where: y is the kinetic energy correction (HC)

2.3 Cold Flow Properties

One of the main problems faced to the use of biodiesel, as a fuel for engine is its properties at low temperatures especially in the cold regions of the world. The behavior of biodiesel at low temperature is an important quality standard because at low temperature crystallization or full solidification of the fuel may cause clogging of filters and finally fuel starvation and stalling of engine. The most important parameters of biodiesel at low temperature applications are cloud point (CP), cold filter plugging point (CFPP) and pour point (PP) [1, 5, 10, 37, 43, 44].

2.3.1 Cloud Point

The cloud point is defined as the temperature of the fuel at which the first cloud of wax crystal can be observed when the fuel is cooled under controlled conditions during a standard test [1, 2, 4, 5, 8, 14, 43, 44] as shown in Figure 2.6. Normally for measuring cloud point ASTM D2500-09 method is used [1, 2, 14]. Usually the cloud point occurs at a higher temperature than the pour point [2, 10].

Figure 2.6 Cloud point.

2.3.2 Cold Filter Plugging Point

Cold filter plugging point is defined as the lowest temperature of a fuel at which the fuel still passes through a standardized filter in a specific time, after this degree of temperature the fuel starts to clog the filter due to crystal formation. CFPP is typically used as indicator of low temperature operability. It is measured per ASTM D6371 [1, 37] (CFPP is not tested in this work).

2.3.3 Pour Point

Pour point is defined as the temperature at which the amount of wax crystal out of solution is sufficient to gel the fuel, so it is the lowest temperature at which the fuel can still flow when the glass jar test is tipped [1, 2, 4, 5, 8, 14, 10, 43, 44] as shown in Figure 2.7. It is usually a few degrees below the cloud point [5]. The pour point is measured according ASTM D-97-05 method [1, 2, 10, 14, 37].

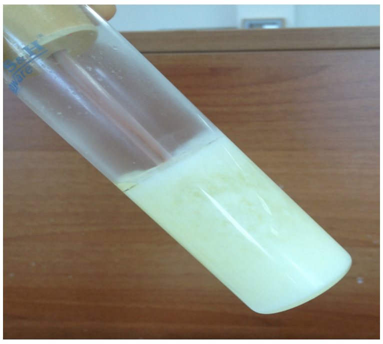

Figure 2.7 Pour point.

2.3.4 Cloud Point (CP) and Pour Point (PP) Measurements

The cloud point and pour point are measured per ASTM standards, D 2500-09 for cloud point and D 97-05 for pour point. The assembly used for measuring the cloud point and pour point is called the cloud point and pour point measurement apparatus. It is mainly consist of a glass test jar that is isolated from an aluminum cylinder by means of a cork support, stopper and ring assembly [2, 4]. The cylinder is immersed into an antifreeze-cooling bath. A thick layer of Styrofoam isolates the whole system.

2.4 Biodiesel Production and standards

Biodiesel is derived from biological sources such as vegetable oils and animal fats. Because of high viscosity of vegetable oils and fats, several methods are used to convert them to a less viscous fuel, which is called biodiesel (Fatty Acid Alkyl Esters) [10, 11, 14, 43, 45]. Biodiesel is a diesel engine fuel comprised of mono-alkyl esters of long chain fatty acids. It is designated by B100 and meeting the specification of ASTM D 6751 [10, 12, 21, 43]. There are several conversion methodologies to produce biodiesel such as transesterification, Supercritical process, Ultra- and high-shear in-line and batch reactors, Ultrasonic reactor, Lipase-catalyzed method and etc. [46], but the most practical and common method is transesterification because of its low cost and simplicity [1, 14].

The produced biodiesel with any of these methods must meet the international biodiesel standard specifications. These specifications include The American Society for Testing and Materials (ASTM D 6751-3) or the European Union (EN 14214) standards for biodiesel fuel and some other standards in the world such as (DIN 51606) in Germany, (CSN) in Czech republic, (ON) in Austria and etc. [1]. In this work, the ASTM D 445-09 is used for kinematic viscosity, ASTM D 2500-09 for cloud point and ASTM D 97-05 for pour point.

The list and standard specification of ASTM D 445-09, ASTM D 2500-09 and ASTM D97-05 are given in Appendix 1.

2.4.1 Biodiesel Production by Transesterification

Transesterification is the most practical and common way to produce biodiesel. It is a chemical reaction between triglyceride (such as vegetable oils and animal fats) and alcohol (such as methanol or ethanol) in the presence of an alkaline catalyst (such as NaOH, KOH or etc.) to produce fatty acid esters and glycerol [1, 43, 45, 47, 48]. Generally, transesterification process includes two main types; catalytic and non-catalytic process as shown in Figure 2.8, but the most frequently used process is the catalytic transesterification process by using alkaline catalyst [1]. The biodiesel samples in this work have been produced by this process.

Biodiesel production by transesterification consists of three consecutive reversible reactions. In the transesterification process, the glycerol inside the triglycerides is replaced with a short chain of alcohol. At the first step of reaction the triglycerides are converted to diglycerides and then diglycerides are converted to monoglycerides, and finally monoglycerides are converted to glycerol. The whole process is consisting of three steps. In each step, an ester is produced; so three ester molecules are produced from one triglyceride molecule [1, 43, 47] as shown in Figure 2.9. In spite of biodiesel production, by-product of this reaction is glycerol, which can be used in the cosmetic industry [1].

2.4.2 Standards of Biodiesel.

Globally, there are some standard specifications for biodiesel fuel. Biodiesel producers, engine designers and consumers must know these standards. As mentioned before, there are several standards all over the world, but the ASTM D 6751 and EN 14214 are the most common standards for biodiesel. Table 2.1 shows some main required properties of biodiesel for ASTM D 6751 in USA and EN 14214 within Europe.

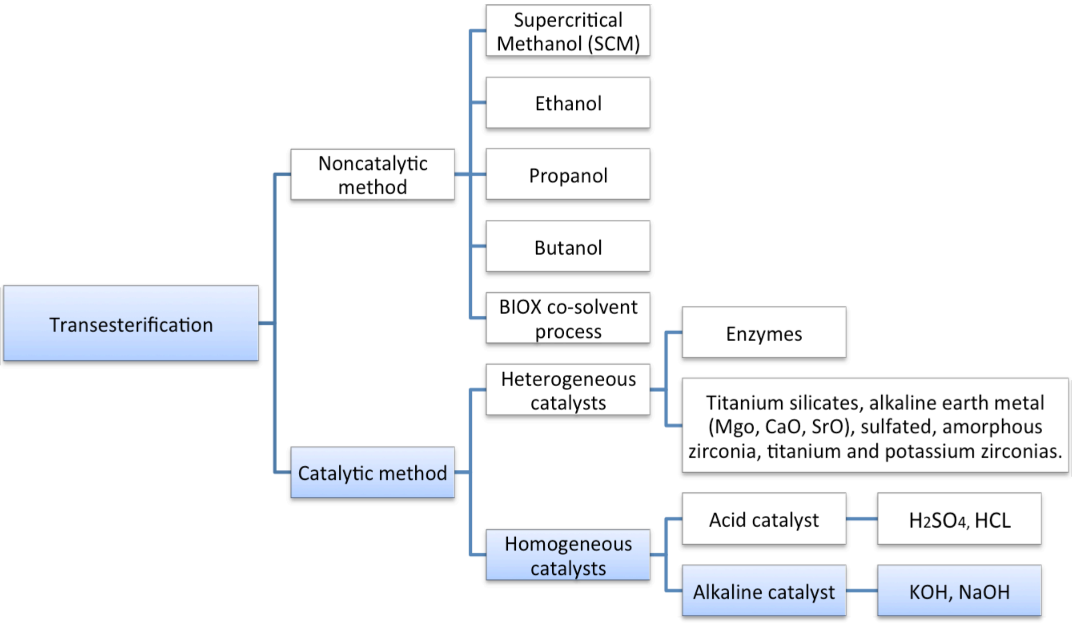

Figure 2.8 Classification of transesterification process [1].

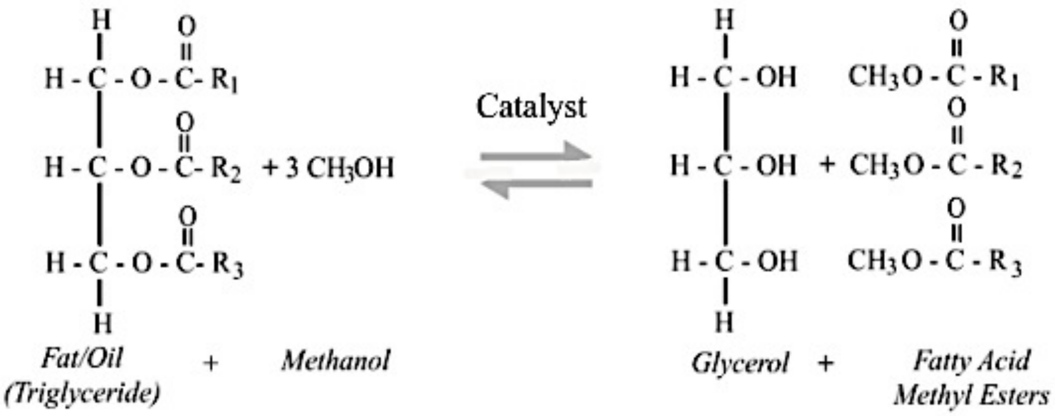

Figure 2.9 Transesterification of triglycerides with methanol, R_1, R_2 and R_3, are the hydrocarbon chain length in the range (C12-C22).

Table 2.1 International standard requirements for biodiesel per ASTM D6751 and EN 14214 [1, 4, 36, 43].

		ASTM D6751		EN 14214	
Properties	Units	Test method	Limits	Test method	Limits
Kinematic Viscosity, at 40 °C	mm²/s	D445	1.9-6.0	EN ISO 3104	3.5-5.0
Flash point	°C	D93	130 min.	ISO 3679	>101
Density at 15°C	kg/m³	-	880	EN ISO 3675 / EN ISO 12185.	860-900
Water & Sediment	vol.%	D2709	0.050 max.	EN ISO 12937	500 max.
Cloud Point	°C	D2500	Report	-	-
pour point	°C	D-97	Report	-	-
Sulfated Ash	% mass	D874	0.020 max.	EN 14214	0.020 max.
Sulfur	% mass	D5453	0.05 max.	EN ISO 20846	10 max.
Cetane number	-	D613	47 min.	EN ISO 5165	51 min.
Acid value	mg KOH/g	D664	0.5 max.	EN 14104	0.5 max.
Copper Strip Corrosion	-	D130	No. 3 max.	EN ISO 2160	class 1
Free Glycerin	% mass	D6584	0.020 max.	EN 14105/14106	0.020 max.
Carbon Residue	% mass	D4530	0.050 max.	EN ISO 10370	0.03 max.

Some international standards for biodiesel are given in Appendix 2.

2.5 Experimental Set-ups and Methods

2.5.1 Kinematic Viscosity

The set-up that is used for measuring kinematic viscosity is shown in Figure 2.10 and described in details as below:

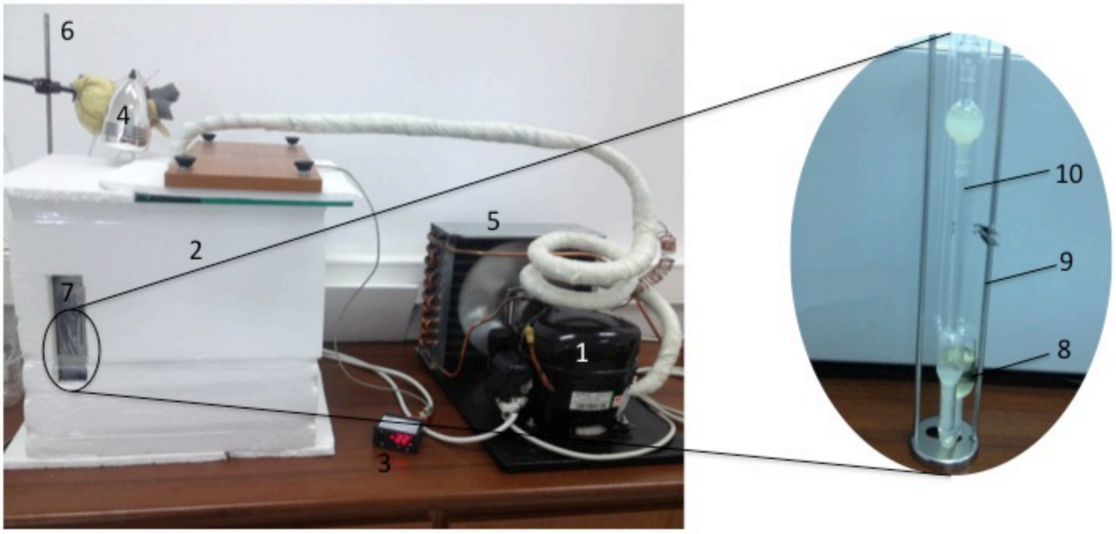

Figure 2.10 Cooling bath system for measuring kinematic viscosity.

1. Compressor
2. Cooling bath glass aquarium, inner volume (250 mm width, 350 mm length and 370 mm depth)
3. Thermostat
4. Mixer
5. Radiator
6. Holder
7. Alcohol as a cooling bath liquid
8. Biodiesel samples
9. Capillary holder
10. Ubbelohde capillary viscometer

The biodiesel samples (8) are poured into the Ubbelohde capillary viscometer (10) and the viscometer is placed in a special holder (9) which keeps the viscometer in a straight vertical position to give the biodiesel samples correct horizontal level inside the viscometer. All together is immersed into the cooling bath liquid (alcohol is used as a cooling bath liquid)(7). A coil connected with a compressor (1) cools down the liquid bath and the compressor is cooled down by a radiator (5). A mixer (4) is used to give a uniform temperature in each point inside the cooling bath. It is held by a separated holder (6) not to make any vibration. The bath temperature is controlled by a thermostat (3), which is automatically starts up and shuts down the compressor. The cooling bath is thermally isolated by a 3cm thickness of Styrofoam layer to keep the temperature inside the cooling bath.

2.5.1.1 Biodiesel samples (specimens)

Five different samples of biodiesel were used in this work. Previously the biodiesel samples had been produced from used frying oil (UFO) and used canola oil (UCO) by transesterification method in (April, 2012). The samples were as below:

1. 100 % UCOME
2. 75 % UCOME + 25 % UFOME
3. 50 % UCOME + 50 % UFOME
4. 25 % UCOME + 75 % UFOME
5. 100 % UFOME

2.5.1.2 Ubbelohde viscometer

Ubbelohde suspended-level viscometer is a measuring instrument that uses a capillary-based method of measuring viscosity. It is recommended for higher viscosity cellulosic polymer solutions. One of the advantages of this instrument is that the values obtained are independent of the total volume. The device was invented by the German chemist Leo Ubbelohde (1877-1964) [49]. Figure 2.11 shows some details for Ubbelohde viscometer.

1 Capillary tube
2 Venting tube
3 Filling tube
4 Reservoir
5 Reference level vessel
6 Dome-shaped top part
7 Capillary
8 Measuring sphere
9 Pre-run sphere
M_1 Upper timing mark

M_2 Lower timing mark

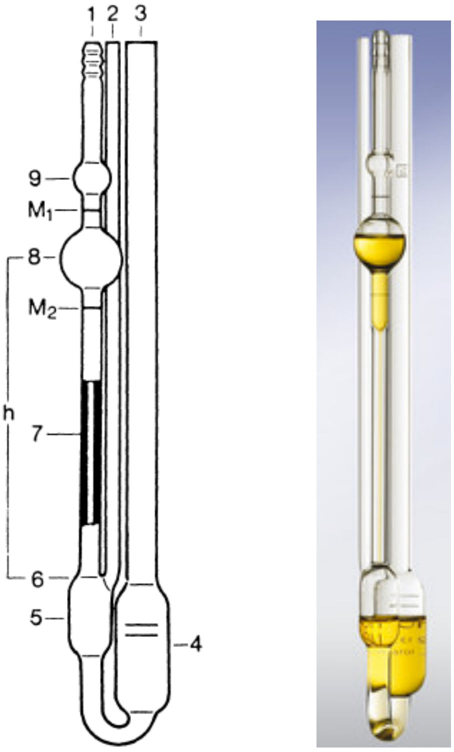

Figure 2.11 Ubbelohde viscometer [39, 42].

The viscometer basically consists of the capillary tube (1), venting tube (2) and the filling tube (3), the capillary (7) with the measuring sphere (8), the pre-run sphere (9) (for Ubbelohde Viscometers) and the reference level vessel (5). Above and below the measuring sphere (8) are printed on timing marks M1 and M2. These marks not only define the flow-through volume of the sample, but also the mean hydrostatic head (h). The capillary (7) ends in the upper part of the reference level vessel (5). The sample runs down from the capillary (7) as a thin film on the inner surface of the reference level vessel (5) (suspended level bulb) [42].

A biodiesel sample is introduced into the reservoir through the filling tube (3). The U–tube at the bottom must be filled completely and should be free from air bubbles and particulate matter. After desired temperature is obtained, a plug or finger is placed over venting tube (2) then sucked through the capillary tube (1) and measuring bulb. The suction is disconnected from capillary tube (1) and the plug is removed from venting tube (2).

The liquid is allowed to travel back through the measuring bulb and the time it takes for the liquid to pass through two calibrated marks (upper timing mark and lower timing mark) is a measure for viscosity. The Ubbelohde device has a third arm extending from the end of the capillary and open to the atmosphere. In this way the pressure head only depends on a fixed height and no longer on the total volume of liquid [29, 49, 50].

Ubbelohde viscometer is useful for the determination of the kinematic viscosity of transparent Newtonian liquids in the range of 0.3 to 100,000 cSt (mm^2/s). It possesses the same constant at all temperatures. This property is advantageous when measurements are to be made at a number of different temperatures. The liquid is induced to flow only down the walls of the bulb below the capillary, thus forming a suspended level, ensuring that the lower liquid level is automatically fixed and coincides with the lower end of the capillary, avoiding the need to fill the viscometer with a definite volume of the liquid and application of corrections for the expansion of glass due to changes in temperature [29, 50].

Ubbelohde type viscometer has some advantages such as speed, low susceptibility to errors and accuracy (within ± 0.1%). It needs a small sample size (about 15 mL is enough). The equipment is cheaper than the other models providing the same type of accuracy. The main concern with this viscometer is the prospect of clogging (specially, in small capillaries) [29].

To get more accuracy of the viscometer, the constant temperature of cooling bath should maintain at a constant ± 0.01 ^{0}C. Only 0.1 ^{0}C difference in temperature may cause an error by 0.6 % [42]. There are many different types of Ubbelohde viscometers covering the kinematic viscosity in the range of even less than 0.3 to above 100,000 cSt (mm^2/s). In Table 2.2, the size number of Ubbelohde viscometers, their constants and corresponding kinematic viscosity range has been tabulated.

According to their kinematic viscosity range, two viscometer of size Ic and II were chosen in this work for measuring kinematic viscosity. The manufacturer has done their calibrations and their constant K is given in Table 2.2 for manual measurements. Their overall length is approximately 285 mm and their filling quantity is (15-20) ml. They were purchased from SI Analytics GmbH, Mainz, in Germany.

Table 2.2 Different types of Ubbelohde viscometers for transparent fluids [29, 42, 51, 50].

Size no:	Capillary diameter (mm) (±2%)	Constant K, (mm²/s)/s (approx.)	Kinematic viscosity range mm²/s (approx.)
0	0.24	0.001	0.3[a] to 1
0c	0.36	0.003	0.6 to 3
0b	0.46	0.005	1 to 5
I	0.58	0.01	2 to 10
Ic	**0.78**	**0.03**	**6 to 30**
Ib	0.88	0.05	10 to 50
II	**1.03**	**0.1**	**20 to 100**
IIc	1.36	0.3	60 to 300
IIb	1.55	0.5	100 to 500
III	1.83	1	200 to 1000
IIIc	2.43	3	600 to 3000
IIIb	2.75	5	1000 to 5000
IV	3.27	10	2000 to 10 000
IVc	4.32	30	6000 to 30 000
IVb	5.2	50	10 000 to 50 000
V	6.25	100	20 000 to 100 000

200 Sec: minimum flow time for all units, but [a]300 sec: minimum flow time.

The manufacturer's certificate for both Ic and II capillary type viscometer are given in Appendix 3.

2.5.1.3 Alcohol (Ethanol)

The water is freezing by 0^0C but pure alcohol will not be frozen up to -114 ^{0}C. Thus, alcohol was used as a cooling bath liquid. The purity of alcohol that used as bath liquid was 97% and purchased from local shop in Nicosia, Cyprus. The alcohol used was (22) liter in a transparent glass bath of (10mm) thickness and (250*350*370) mm^3 volume.

2.5.1.4 Temperature measurement

An accurate thermostat was used to control and keep the temperature constant inside the cooling bath, which is connected to a compressor and automatically turns the compressor on and off. It was measuring the temperature by 0.1 ^{0}C for avoiding or reducing errors as shown in Figure 2.12.

Figure 2.12 Reading of thermostat.

2.5.1.5 Accessories

For measuring kinematic viscosity, some accessories were used which some of them were shown in Figure 2.10. These include:

1. A mixer: used for getting uniform temperature at any point inside the cooling bath.
2. Glass pipette: used for transporting a measured volume of Biodiesel sample into the viscometer and cleaning the viscometer after changing the samples.
3. Holder: It was used for holding the mixer separately from the whole set-up to avoid making vibration.
4. Vacuumed Syringe: used for suction process during measurement.
5. Stop watch: used for accurate measurement of time as required by the standard procedure.
6. Viscometer Holder: used to keep the ubbelohde capillary viscometer vertically upright in the cooling bath.

2.5.1.6 Methodology

The kinematic viscosity is determined by measuring the time for a known volume of liquid flowing under gravity to pass through a calibrated glass capillary viscometer tube according to ASTM standard D445. The following steps are necessary for measuring the kinematic viscosity of the biodiesel samples.

1. Before first use, the viscometer has been cleaned with 15 % H_2O_2 and 15 % HCl, then rinsed viscometer with a suitable solvent (Acetone was used for this work) to be completely dry and dust-free and be ready to use for manual measuring.
2. The samples were filtered to clean it from possibility of lint, dust, or any other solid materials by using fine mesh screen.
3. Enough volume of sample (15 ml) was introduced through filling tube into the lower reservoir by using glass pipette.
4. The viscometer was placed into the holder, and inserted it into the constant temperature bath. It was immersed vertically into alcohol inside the cooling bath.
5. It was left for a long enough time to get the same temperature of the bath.
6. There was applied suction to capillary tube (1) as in Figure 2.11, closing venting tube (2) by a finger or rubber stopper. This will cause the successive filling of the reference level vessel (5), the capillary tube (1), the measuring sphere (8), and the pre-run sphere (9). Filled to approximately 10 mm above the upper timing mark M_1. Now suction is disconnected from the capillary tube (1) and the venting tube (2) opened again. This caused the liquid column to separate at the lower end of the capillary (7) and to form the suspended level at the dome-shaped top part (6), finally the liquid started to come down.
7. The time interval (efflux time t) was measured; it is a period of time, which is taken by an amount of the sample from the upper edge of the upper timing mark M_1 to the upper edge of the lower timing mark M_2.
8. The kinematic viscosity of the sample was calculated by multiplying the efflux time (t) by the viscometer constant (K). For absolute measurement, the kinetic energy correction (y) was subtracted from efflux time (t) then multiplied by constant (K). Equation 2.23 was used for determining kinematic viscosity (υ).

$v = K(t - y)$

9. Without changing the sample, the steps 6 to 8 were repeated four or more than four times for each degree of temperature from 20 ^{0}C down to pour point by stepwise (interval) of 5 ^{0}C but when it is approaching the onset of crystallization, by stepwise of 1 ^{0}C. If a measurement is different by ($\pm 1\%$) with each other, it will be cancelled and taken one more measurement

But after changing the sample, the procedure was started one more time from step 1 to 9 then the steps 6 to 8 were repeated again for each degree of temperature. This process was continued for the all samples.

The measurements of the kinematic viscosity for each sample have been conducted. As mentioned before, fore more accurate value, four or more measurements have been conducted for each sample at the same degree of temperature. Here there are two ways to taking average, either taking the average of the four efflux times (t) and then calculate the kinematic viscosity or calculate the kinematic viscosity for each efflux time (t) and then take the average for four different kinematic viscosity. Both ways gave the same result for kinematic viscosity.

In this work the average of four efflux times (t) were taken and then calculated the kinematic viscosity by using Table 2.2 for (K), Table 2.3 for (y) and Equation 2.23.

2.5.1.7 Calculation of kinematic viscosity

The Equation 2.23 is used for determining the kinematic viscosity. The number of seconds stated for the various capillaries in the Table 2.3 of the kinetic energy correction (HC) is subtracted from the determined efflux time (t) by experiment and then the corrected flow time multiplied by the viscometer constant K gives the kinematic viscosity (mm^2/s) directly.

$$v = K(t - y)$$

Table 2.3 Table of the kinetic energy correction (HC) [42].

Ubbelohde viscometers ISO 3105/ASTM D2515 (Ref. No. 525… 526…)

Correction seconds a):

Flow time [s]	Capillary No. 0	0b	0c	I	Ic
50	– b)	(5.06) b)	(6.69) b)	(2.45) b)	0.41
75	– 2)	2.25	2.98	1.09	0.18
100	(3.69) b)	1.26	1.67	0.61	0.10
125	2.36	0.81	1.07	0.39	0.07
150	1.64	0.56	0.74	0.27	0.05
175	1.21	0.41	0.55	0.20	0.03
200	0.92	0.32	0.42	0.15	0.03
225	0.73	0.25	0.33	0.12	0.02
250	0.59	0.20	0.27	0.10	0.02
275	0.49	0.17	0.22	0.08	0.02
300	0.41	0.14	0.19	0.07	0.01
325	0.35	0.12	0.16	0.06	0.01
350	0.30	0.10	0.14	0.05	0.01
375	0.26	0.09	0.12	0.04	0.01
400	0.23	0.08	0.11	0.04	0.01
425	0.20	0.07	0.09	0.03	0.01
450	0.18	0.06	0.08	0.03	< 0.01
475	0.16	0.06	0.07	0.03	< 0.01
500	0.15	0.05	0.06	0.02	< 0.01

a) The correction seconds stated are related to the respective theoretical constant.

b) For precision measurements, these flow times should not be applied.
A selection of a viscometer with a smaller capillary diameter is suggested.

An example:

Determining the kinematic viscosity of (75% UCOME)&(25%UFOME) at 20 ^{0}C. As mentioned before, the average of four efflux times (t) are taken at 20 ^{0}C and then calculated the kinematic viscosity by using Table 2.2 for (K), Table 2.3 for (y) and Equation 2.23.

Capillary Ic constant (K) = 0.02799 (mm^2/s)/s

Flow time (averaged) t = 372.75 s

Kinetic energy correction (HC) y for 372.75 = 0.01 s

Kinematic viscosity = 0.02799*(372.75-0.01) =10.4329926 mm^2/s

Table 2.4 Calculation of kinematic viscosity of (75% UCOME + 25%UFOME) at 20 ^{0}C.

Times (T ^{0}C @ 20)	Sec.	Ave. Time (sec.)	Ave. Time (±1%)	Capillary Constant (K) (mm^2/sec^2)	Kinetic Energy Correction (y) (sec.)	Kinematic Viscosity (mm^2/sec)
Time 1	373.1	372.75	376.4775	0.02799	0.01	10.4329926
Time 2	372.2					
Time 3	374.1		369.0225			
Time 4	371.6					

In the same manner, the kinematic viscosity for all samples has been calculated and tabulated which are shown and discussed in the Chapter Three.

2.5.2 Cloud Point and Pour Point Set-up

The assembly used for measuring the cloud point and pour point is shown in Figure 2.13 and called cloud point and pour point measurement apparatus. It has been manufactured following the ASTM and EN-ISO standards.

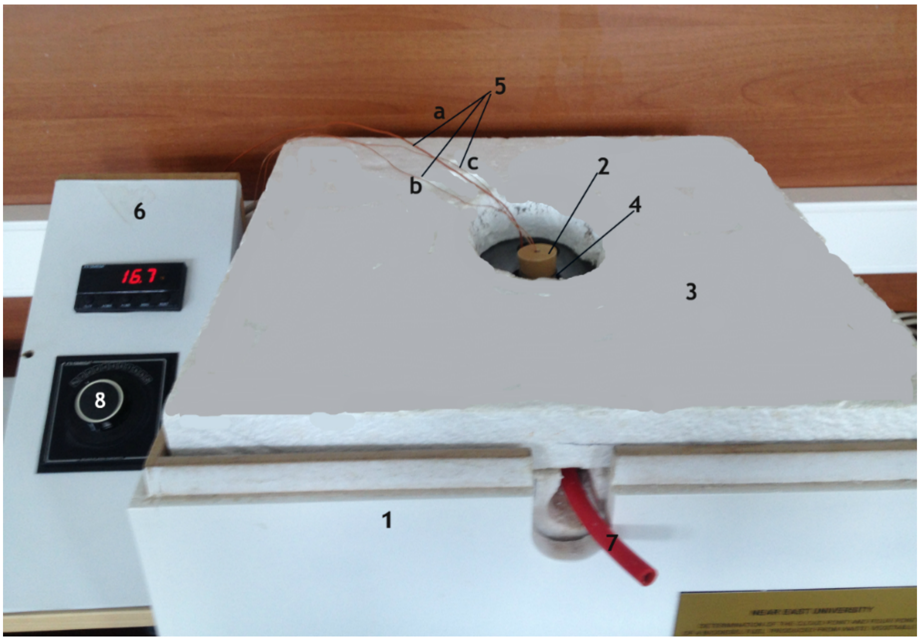

Figure 2.13 Cloud point and pour point measurement apparatus.

1. Cooling bath: isolated by a thick layer of Styrofoam block (11cm) inside a wooden box.
2. Cork: to close the mouth of the glass test jar.
3. A block of Styrofoam: to keep the whole system cold for a long time.
4. Glass test jar: Test sample is poured into the glass test jar by amount of (45ml).
5. Thermocouples: Three (T type) thermocouples are used
    a. For measuring temperature inside the cooling bath.
    b. For measuring the cloud point, which is placed in the bottom of the glass test jar.
    c. For measuring the pour point, which is placed in the upper layer of the sample inside the glass test jar.

6. Multi-thermometer: Used for reading the cooling bath temperature, (CP) and (PP).
   This thermometer was calibrated with the thermometer that shown in Figure 2.12. Both of them were calibrated with mercury thermometer.

7. Hose: For keeping the standard level of the cooling bath liquid.
8. Regulator: For showing the readings among the three different temperatures.

The assembly consists of: A glass test jar (4), which has been placed into an aluminum cylinder inside the cooling bath liquid (in this case, alcohol has been used as a cooling liquid). The glass test jar has been isolated from the aluminum cylinder by means of a cork support, stopper and ring assembly. The cylinder has been immersed into an 8-liter stainless steel cooling bath-containing alcohol at -25 ^{0}C. The cooling bath has been put inside an 11cm thick of Styrofoam block in order to isolate it from any vibrations and heat transfer to keep the cooling bath temperature very cold for a long period of time. The whole system has been covered by a 3cm thick of wooden box.

The same samples, which were used for determining kinematic viscosity, were used for determining their cloud point and pour point.

2.5.2.1 Methodology

The cloud point is defined as the temperature at which a cloud of wax crystals first appear in a liquid when it is cooled under controlled conditions during a standard test but the pour point is the temperature at which the amount of wax crystal is sufficient to solidify the fuel, thus it is the lowest temperature at which a fuel can still flow. The samples were tested as per American standard test method for cloud point and pour point, ASTM D2500 and ASTM D97 respectively.

After preparing the apparatus and cooling down the cooling bath liquid, the cloud point test consisted of the cooling of the sample in the glass test jar under prescribed conditions and inspected at stepwise of 1 ^{0}C until a cloud (fog) appeared into the sample, then this degree was recorded as cloud point for that sample when the reading was taken from that thermocouple which was placed in the bottom of the test jar because temperature at the bottom of the jar is normally higher than the top. For determining the pour point, the observation of the samples starts at a temperature that is at least 9 ^{0}C above the expected pour point. The same sample was immersed into a -25 ^{0}C cooling bath and inspected at stepwise of 1 ^{0}C until the sample was totally ceased to flow. Reading of the test thermometer was taken and the degree before this reading was recorded as pour point but the reading was taken from that thermocouple which was placed at the top layer of the sample. All the measurements were done three times for each sample and the results averaged (The standard deviation of the test results is ±2 ^{0}C). In Table 2.5 an example (100% UCOME) has been shown for averaging the cloud point and pour point.

In brief, the following steps are necessary for determining the cloud point and pour point of biodiesel samples:

1. By using the set-up in Figure 2.10, alcohol was cooled down to -25 ^{0}C, and then transferred to cooling bath in cloud and pour point apparatus.
2. A specific sample of biodiesel was poured into the test jar, put it into the aluminum cylinder which was immersed in the cooling bath liquid and then placed the thermocouples in their determined position as mentioned before.
3. It was inspected at stepwise of 1 ^{0}C until a cloud appeared into the sample at the bottom of the test jar. This degree was recorded as cloud point and the procedure was repeated three times then the average was taken.
4. The same sample was heated to 9 ^{0}C above the expected pour point then one more time immersed it into the cylinder. For a greater degree of accuracy, pour point measurements were done with an increment of 1 ^{0}C. It was inspected at stepwise 1 ^{0}C until it was totally ceased to flow then the degree before ceased degree was recorded as a pour point. Again this procedure was repeated three times and the results were averaged.

After these steps, the test jar was cleaned and another sample was prepared for doing the same procedure.

Table 2.5 Taking average of cloud point and pour point.

Sample	Cloud Point (CP) ^{0}C	Ave. Cloud Point ^{0}C	Pour Point (PP) ^{0}C	Ave. Pour Point ^{0}C
100% UCO	-1	-1	-10.5	-10.5
	-1		-10.5	
	-1		-10.5	

In the same manner, the cloud point and pour point have been determined for all samples and they are shown and discussed in Chapter Three.

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Reliability of the Results

For more accuracy and reliability, some steps were conducted before starting the experiments and during the experiments such as:

- The manufacturer had calibrated the capillary viscometer. It has been determined by using comparative measurements with reference viscometers, of which the constants were determined at the Physikalisch-Technischen Bundesanstalt, D-38116 Braunschweig. It was checked again by selecting a calibrated viscometer of known viscometer constant K_2 (in this work, it was 0.002856 mm^2/s^2) as a reference viscometer. It was used distilled water as the basic kinematic viscosity standard. The kinematic viscosity of distilled water is 1.004 (mm^2/s) at 20 ^{0}C as given in the literature [32]. By following the D446-07 method, the calibrated viscometer and the viscometer to be calibrated were mounted in the same bath to determine the flow times (t_1 and t_2), and then calculated the K_1 by using equation 3.1.

$$K_1 = t_2 * K_2/t_1 \tag{3.1}$$

- The constancy of temperature: instead of using mercury thermometer, a very accurate thermometer was used for measuring the temperature inside the cooling bath and used thermocouples for determining cloud and pour point.
- Following the sample filling procedure, the measurements were taken after at least 15 minutes or for a period of time to equalize the temperature. Actually, the period of time depends on the temperature of the laboratory and that degree of temperature, which is taken.

- The actual time measurement was performed for each degree of temperature with a stopwatch by observing the exact starting and ending time of flowing. Normally, the time measurement includes a very small degree of measuring uncertainty.
- In practice, the repeating accuracy is the most important criterion in most cases. Thus the repeatability test was done to get precise results as in Table 3.1 and 3.2 for both measuring kinematic viscosity and determining cloud and pour point respectively.

Table 3.1 Reliability results for biodiesel samples at 20 ^{0}C.

Biodiesel type	Measured kinematic viscosity (mm^2/s)	Average Kinematic viscosity (mm^2/s)	Absolute error in (%)
100% UCOME	7.3356192	7.35171345	0.219398657
	7.3412172		0.142976971
	7.3636092		0.161547818
	7.3664082		0.199483243
100% UFOME	7.4475792	7.41539070	0.432200842
	7.3748052		0.5503264
	7.3943982		0.283897343
	7.4447802		0.394766524
50%UCOME+ 50%UFOME	9.6058881	9.63737685	0.327806754
	9.6562701		0.195657845
	9.6310791		0.065389869
	9.6562701		0.195657845

Table 3.2 Accuracy of cloud point and pour point results for (100% UCOME).

Sample	Cloud Point (CP)	Ave. Cloud Point (^{0}C)	Absolute error (%)	Pour Point (PP)	Ave. Pour Point (^{0}C)	Absolute error (%)
100% UCO	-1	-1	0	-10.5	-10.5	0
	-1		0	-10.5		0
	-1		0	-10.5		0

From Table 3.1, it is clear that the measurements, which have been done by the viscometers, are accurate because the absolute error is less than 0.5%. At the same time the errors that are noticed from Table 3.2 for cloud point and pour point is zero. Thus, there is more than 99% probability that all results to be given and discussed in this chapter are true.

3.2 Kinematic Viscosity

Viscosity is a measure of the resistance offered by a fluid to flow. According to Krisnangkura et al [24]. Viscosity may be considered the integral of the interaction forces of molecules [30]. The kinematic viscosities of five biodiesel samples have been measured in this work are reported in Table 3.3. Also the relationship between the kinematic viscosity and temperature of different composition of biodiesel such as (100% UCOME, (75% UCOME + 25% UFOME), (50% UCOME + 50% UFOME), (25% UCOME + 75% UFOME and 100% UFOME) are given in Figures 3.1 - 3.5 respectively. It can be easily obtained any magnitude of viscosity from Table 3.3 or from the charts for the known temperatures. The measurements indicate that all samples have the same qualitative behavior when they are cooled down, it is making them more viscous, but all the samples are different at the temperatures approaching the onset of crystallization because of different chemical composition.

From the Figures 3.1 – 3.5 it is seen that the viscosity rapidly increases as the temperature decreases. The viscosity of each biodiesel varies from another biodiesel at the same temperature according to their chemical composition and mixed percentages for instance the viscosity of (75% UCOME+25% UFOME) at 20 ^{0}C is (10.43299260) mm^2/s, while the viscosity of (25% UCOME+75%UFOME) at the same temperature is (9.63737685) mm^2/s. Viscosity of 100% UFOME could not be determined less than 15 ^{0}C and the viscosity of 100% UCOME could not be determined at less than -8 ^{0}C because they are approaching to become gel. Thus by mixing any two portions of UCOME and UFOME, the lowest determined degree of temperature occurs between 15^{0}C and -8^{0}C.

Table 3.3 Kinematic viscosities of five biodiesel fuels from 20 ^{0}C down to -10 ^{0}C.

Temperature (^{0}C)	Kinematic viscosity (mm^2/s)				
	100%UCOME	75%UCOME + 25%UFOME	50%UCOME + 50%UFOME	25%UCOME + 75%UFOME	100%UFOME
20	7.35171345	10.43299260	9.63737685	9.63737685	7.41539070
15	8.61224310	12.31532010	11.47282110	11.42663760	8.71160760
10	9.78852285	14.51953260	13.42092510	13.42792260	nd[a]
8	10.41479910	15.55726185	14.35159260	nd[a]	-
5	11.83459185	17.57044260	nd[a]	-	-
2	13.14382410	20.28419105	-	-	-
0	14.03040735	nd[a]	-	-	-
-3	15.59015010	-	-	-	-
-5	18.28278810	-	-	-	-
-7	21.51664030	-	-	-	-
-8	23.86299105	-	-	-	-
-10	nd[a]	-	-	-	-

[a] : (not determined), Formation of crystals at this temperature.

In Table 3.3, the increment for determination of kinematic viscosity was (5 ^{0}C), but when the measurements were approaching to the lowest determined temperature, the stepwise has been decreased to (1 ^{0}C) or (2 ^{0}C) in order to determine the highest kinematic viscosity of the samples.

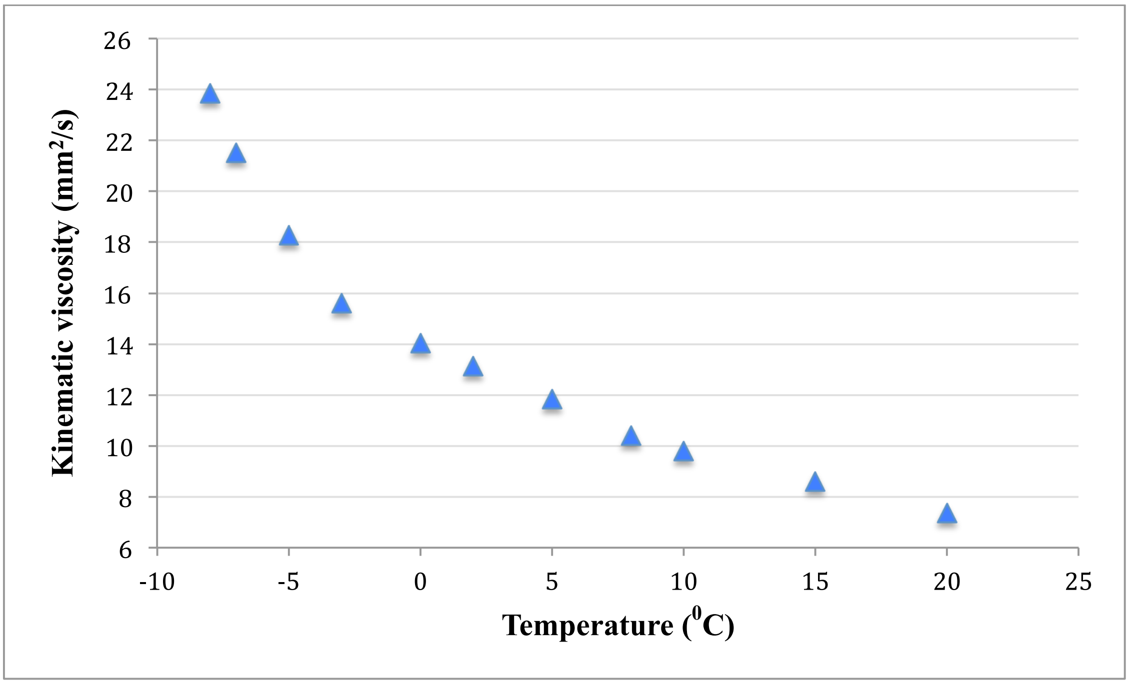

Figure 3.1 Kinematic viscosity-temperature relationship of 100% UCOME.

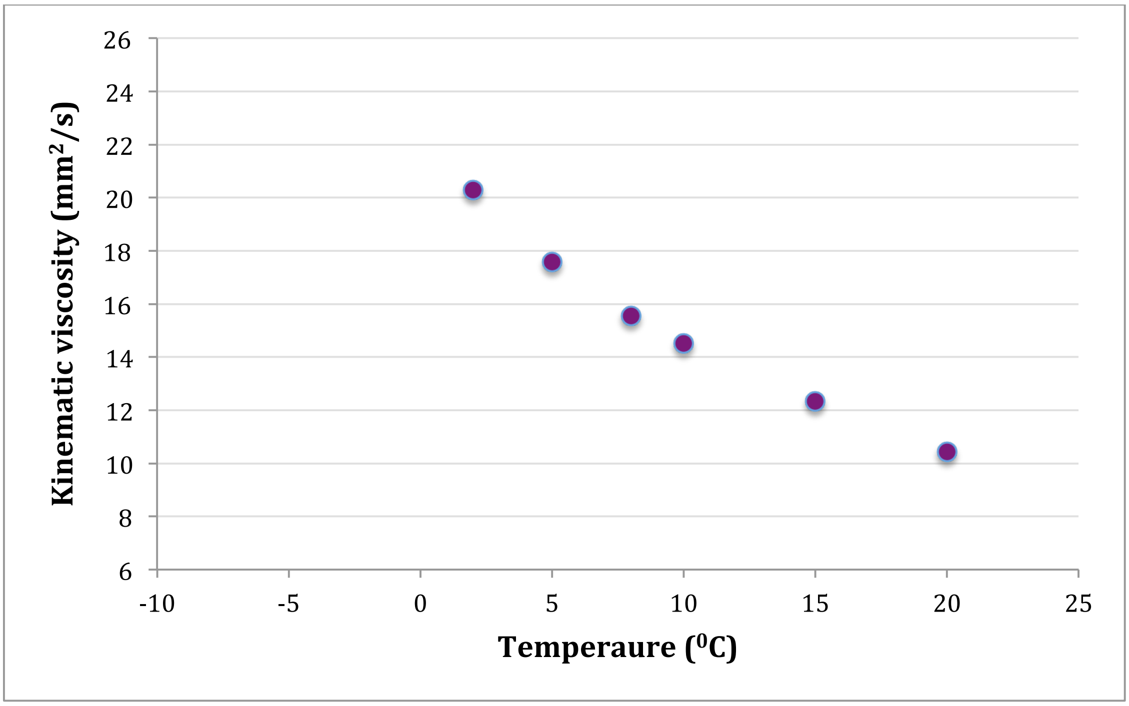

Figure 3.2 Kinematic viscosity-temperature relationship of (75% UCOME + 25%UFOME).

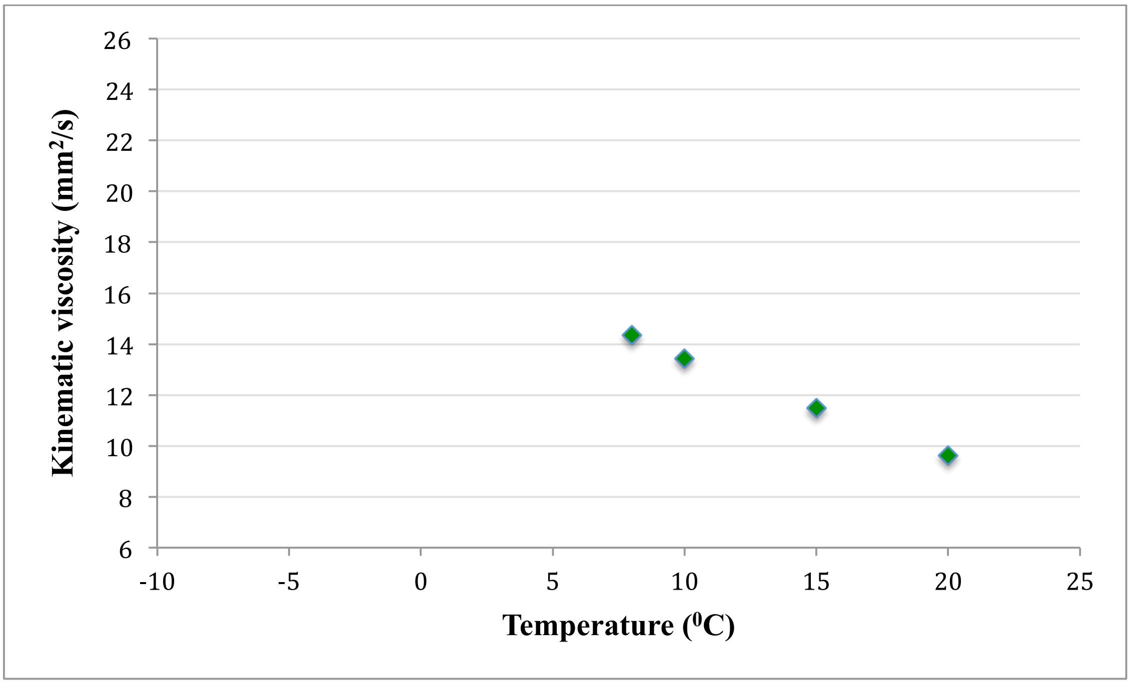

Figure 3.3 Kinematic viscosity-temperature relationship of (50%UCOME + 50%UFOME).

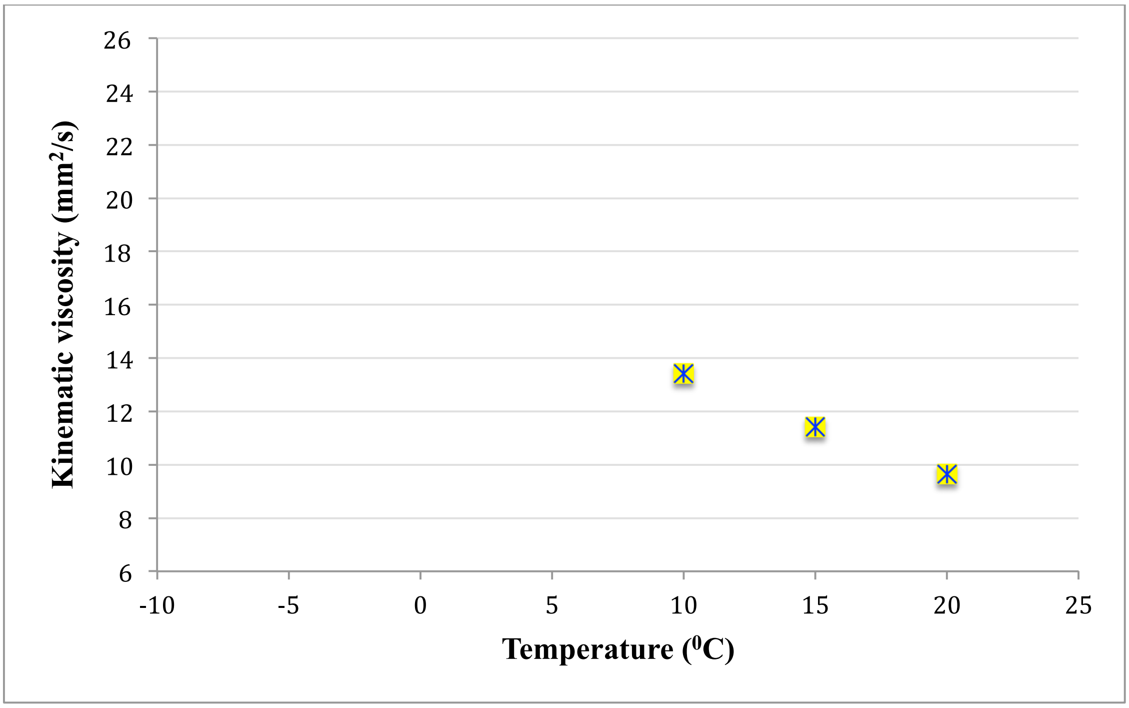

Figure 3.4 Kinematic viscosity-temperature relationship of (25%UCOME + 75%UFOME).

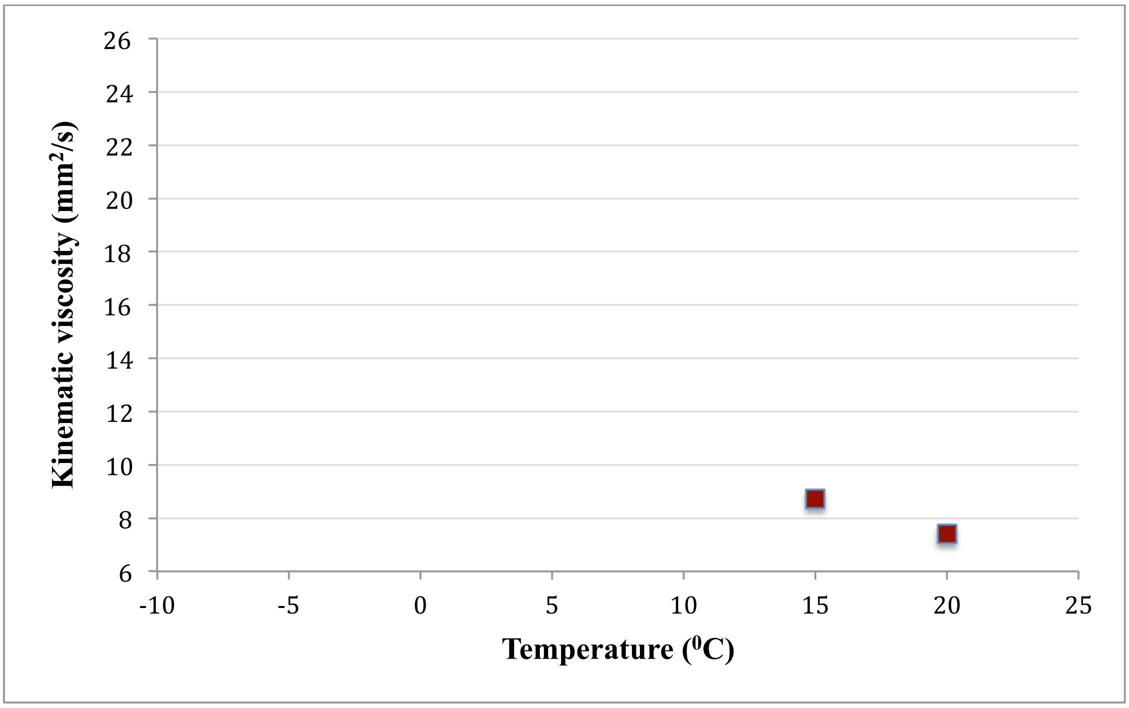

Figure 3.5 Kinematic viscosity-temperature relationship of 100%UFOME.

The lowest kinematic viscosity is 7.35171345 mm^2/s for 100% UCOME at 20 ^{0}C. By mixing only 25% of UFOME with 75% of UCOME, the kinematic viscosity gets the highest value (10.43299260 mm^2/s) for the same degree of temperature among the biodiesel samples and the lowest determination of temperature changed from -8 ^{0}C to 2 ^{0}C. When two equal portions of UCOME and UFOME (50% UCOME + 50% UFOME) was tested, the kinematic viscosity was decreased, but again the lowest determined temperature increased by 6 ^{0}C from 2 ^{0}C to 8 ^{0}C. This is true for all degrees of temperatures. The value of kinematic viscosity of (25% UCOME + 75% UFOME) is less than the kinematic viscosity of (75% UCOME + 25% UFOME) for the temperatures (20 ^{0}C, 15 ^{0}C and 10 ^{0}C) and it is equal in both (50% UCOME + 50% UFOME) and (25%UCOME + 75%UFOME) at 20 ^{0}C and again it is almost equal at 10 ^{0}C.

In Figure 3.6, illustrated the kinematic viscosity and temperature relationships of the biodiesel samples.

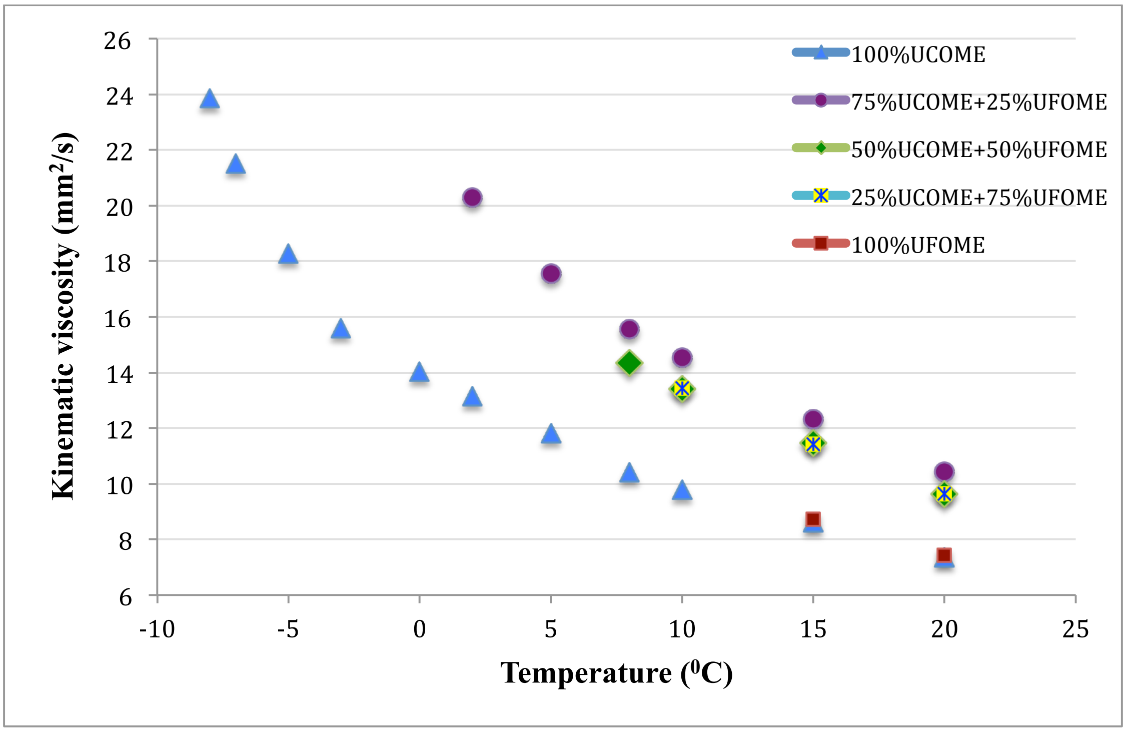

Figure 3.6 Kinematic viscosity-temperature relationships of the biodiesel samples.

From the figure, it can be seen that as the temperature decrease, the kinematic viscosity rapidly increase and this increasing is different from a biodiesel to another. This increasing of viscosity is in a good agreement with the other works that previously reported in the literatures.

It cannot be found an overall theory to determine the viscosity of the liquids so far because of its complex nature. In spite of the large deviation in the measure viscosity data, there are some models to predict and estimate the kinematic viscosity such as Krisnangkura's model [24], Yuan's model, and Ceriani's model [11]. The effect of temperature on the kinematic viscosity of liquid is described by means of the Arrenhius equation [30] as

$$v = A_1 . exp \frac{E_a}{GT} \quad 3.1$$

v being the kinematic viscosity, E_a the activation energy for flow, G the universal gas constant and T the absolute temperature. Additionally,

$$A_1 = \frac{N_A h}{V} \qquad 3.2$$

where N_A, V and h are the Avogadro's number, the molecular volume and the Plank's constant, respectively.

In the case of vegetable oils, the Equation 3.1 can be written as the Andrade equation:

$$v = A_1 . exp^{\frac{B}{T}} \qquad 3.3$$

where T is the absolute temperature and A_1 and B are specific constants to be adjusted for each specific oil. By applying logarithms to both sides of equation and Equation 3.3 can be linearized by applying the least-squares method and making $^1/_T$ the independent variable and it becomes as follow [30]:

$$\ln(v) = A + {}^B/_T \qquad 3.4$$

This is the most common equation that is empirically used to determine the relationship between viscosity and temperature, with the constant A and B are determined empirically. A modified form of Andrade equation is especially useful when dealing with wide temperature ranges and it has been reported by (Tat and Van Gerpen) and (Azian et al.) [21, 52].

$$\ln(v) = A + {}^B/_T + {}^C/_{T^2} \qquad 3.5$$

Thus, Equation 3.4 is used in analysis, validation and discussion of the data that obtained for the five biodiesel samples in this work. A relationship between $\ln(v)$ and $^1/_T$ is plotted, where *v* is the measured kinematic viscosity and *T* is the absolute temperature in Kelvin and Figures 3.7-3.11 show the regressions.

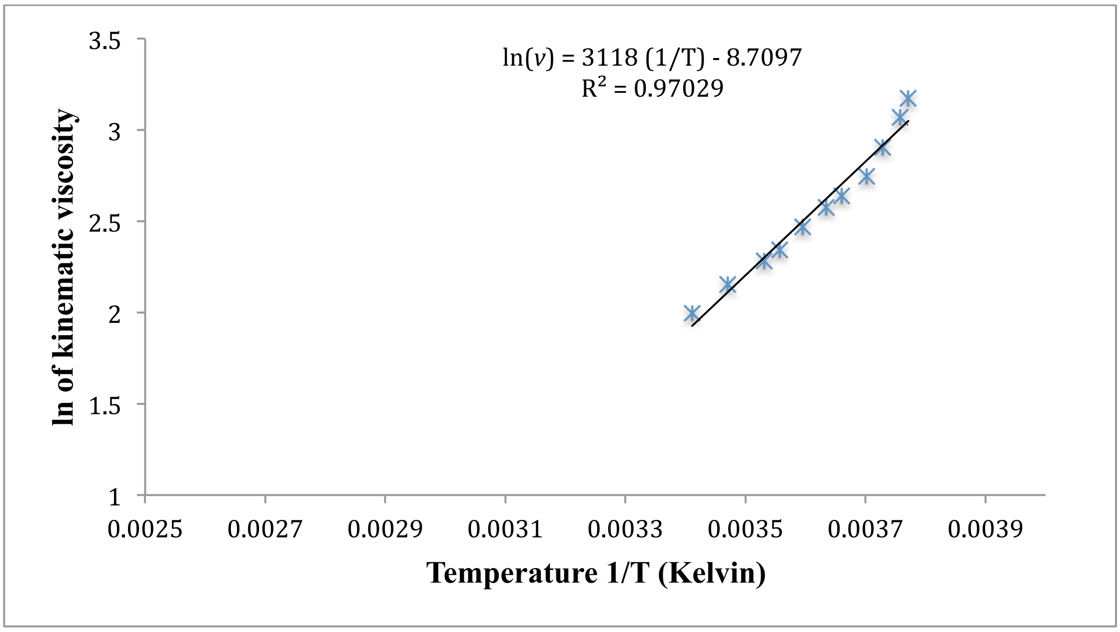

Figure 3.7 Empirical model for 100% UCOME.

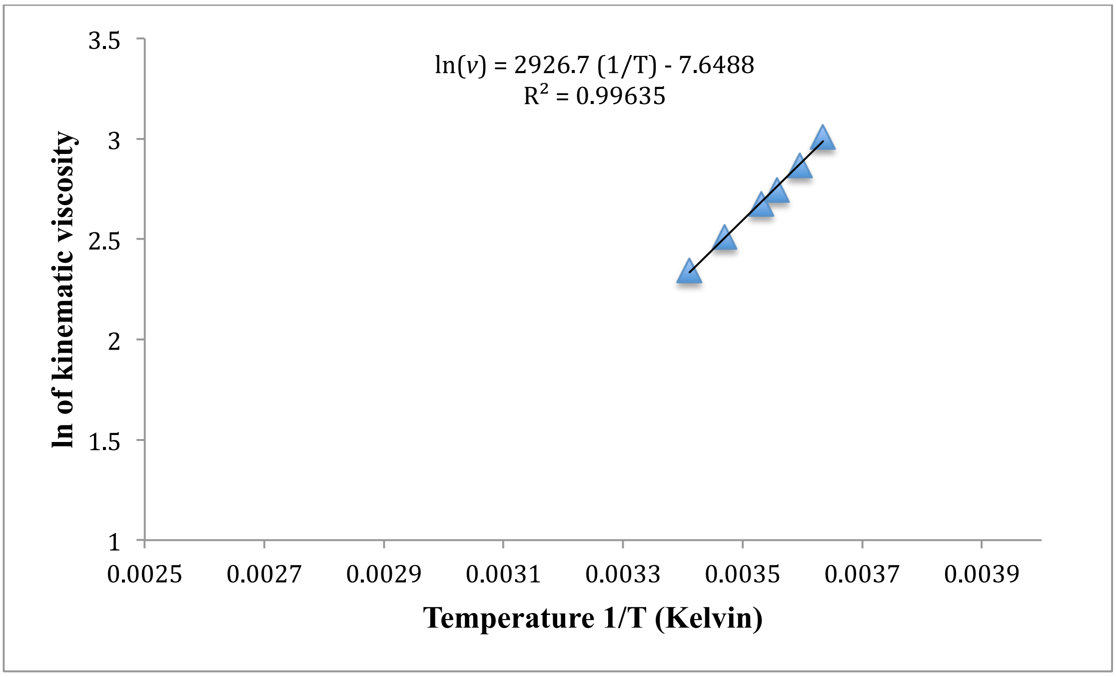

Figure 3.8 Empirical model for (75% UCOME + 25%UFOME).

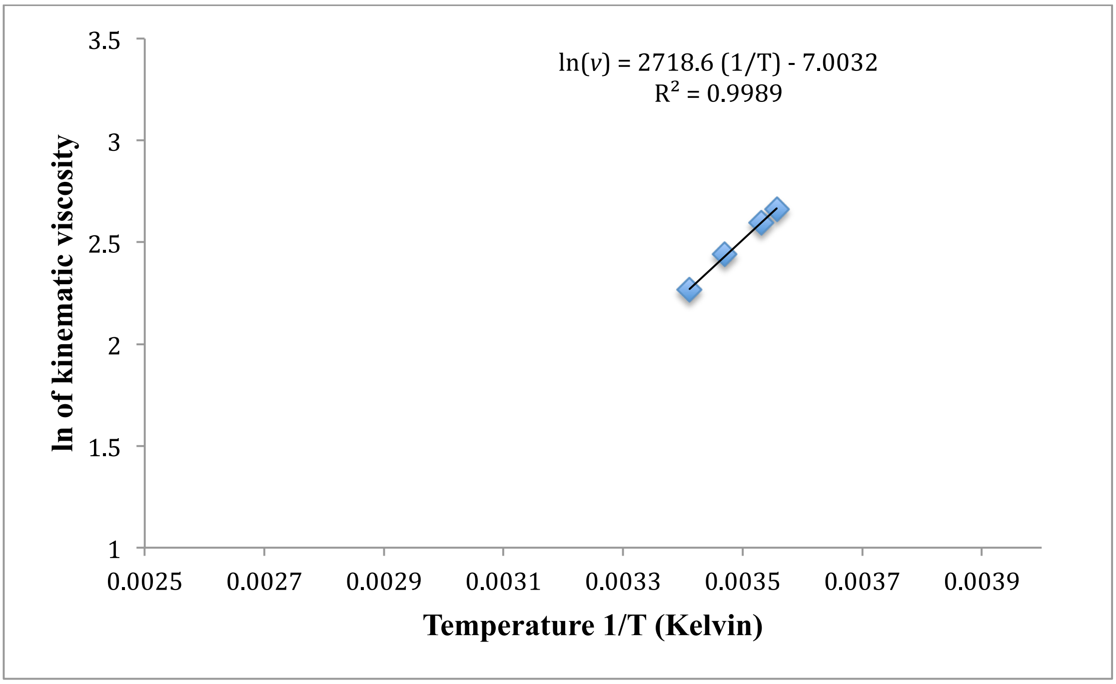

Figure 3.9 Empirical model for (50%UCOME + 50%UFOME).

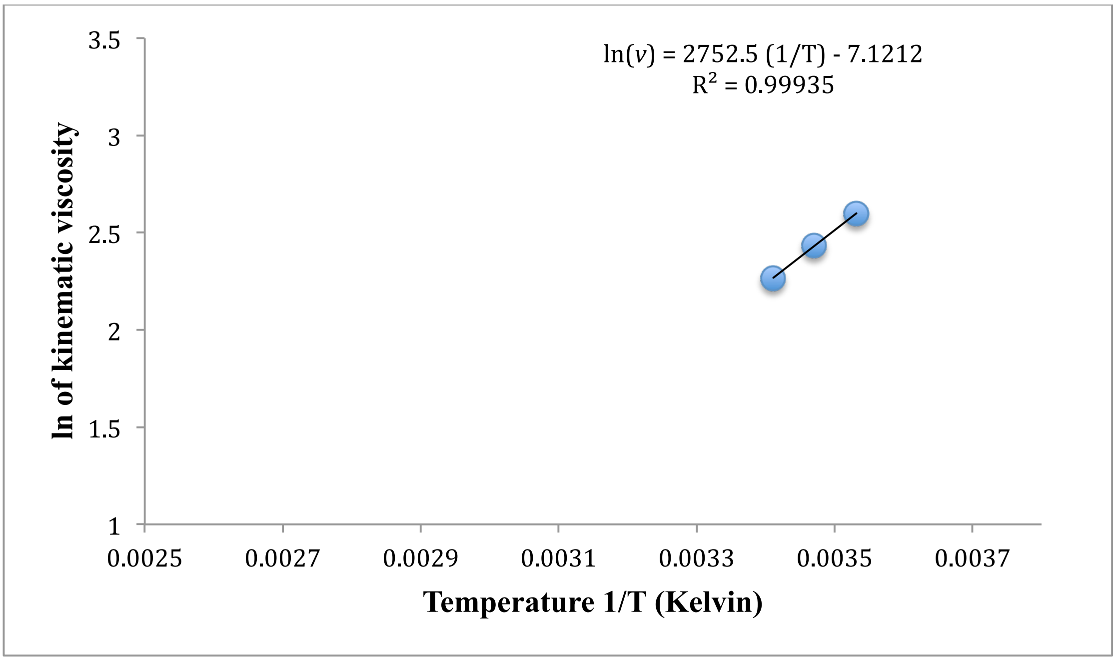

Figure 3.10 Empirical model for (25%UCOME +75%UFOME).

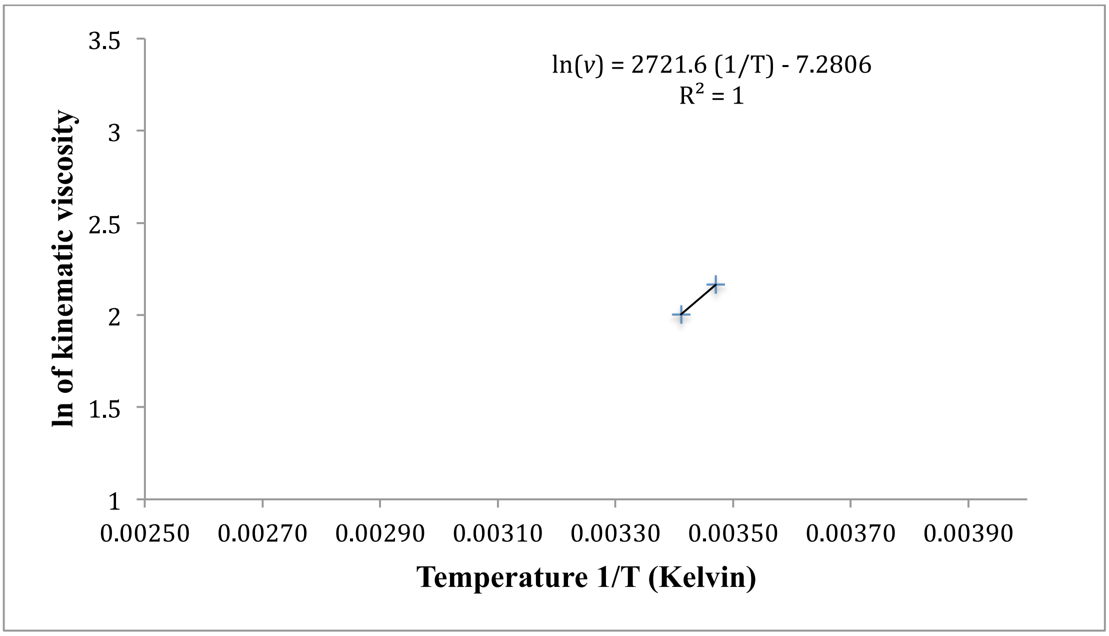

Figure 3.11 Empirical model for 100%UFOME.

The correlation coefficient for all samples are greater than 0.99, but for 100% UCOME is 0.97 as given in Table 3.4.

Table 3.4 Viscosity correlation coefficient and constants for the samples from 20 ^{0}C to -10 ^{0}C.

Biodiesel samples	A	B	R^2
100% UCOME	3118	-8.7097	0.97029
75%UCOME+25%UFOME	2926.7	-7.6488	0.99635
50%UCOME+50%UFOME	2718.6	-7.0032	0.9989
25%UCOME+75%UFOME	2752.5	-7.1212	0.99935
100% UFOME	2721.6	-7.2806	1

Except the temperature, there is a good effectiveness between the viscosity and the different percentage of the samples as in Figure 3.12.

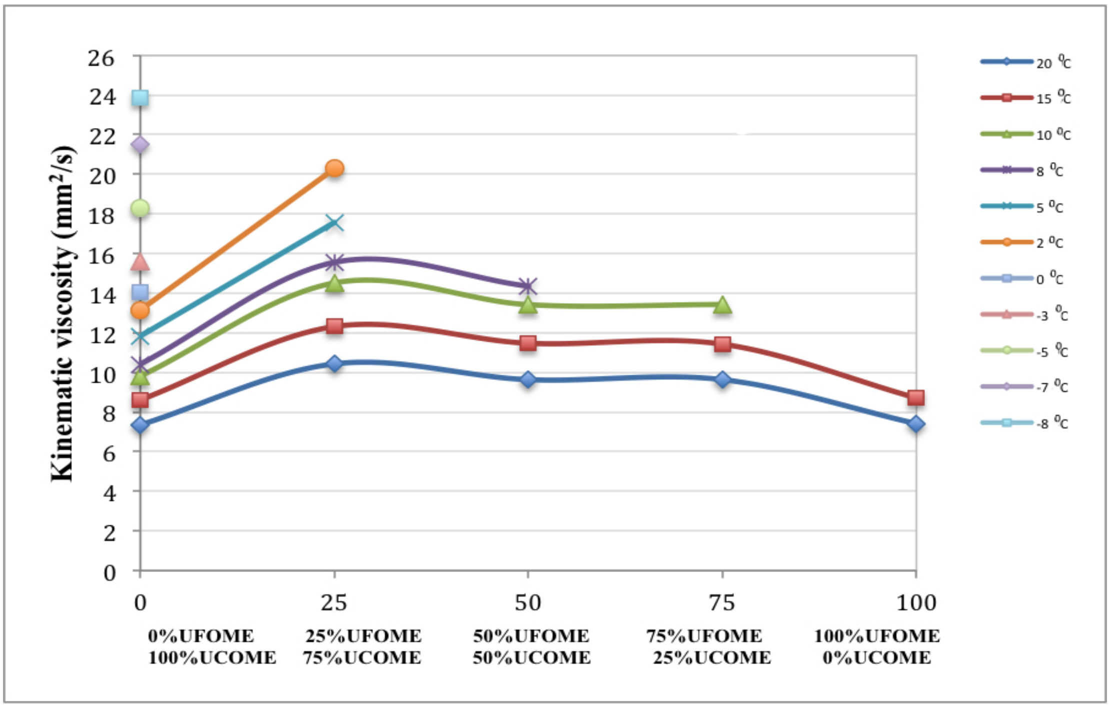

Figure 3.12 Kinematic viscosity and percentage composition relationship.

From the figure, the kinematic viscosity 100% UFOME is a little greater than the kinematic viscosity of 100% UCOME at the same temperature and pressure, especially at both 20 ^{0}C and 15 ^{0}C because it could not be determined the kinematic viscosity for lower degrees of the temperature for the all samples due to the solidification or the onset of crystallization. As the percentage of UCOME is increased by 25%, the viscosity increases, but at 75% UCOME and 25% UFOME gets the highest value of kinematic viscosity for all cases then after this point, it drops down again. The kinematic viscosity of both 50% UFOME and 75% UFOME are nearly equal to each other at 20 ^{0}C, 15 ^{0}C and 10 ^{0}C but at 8 ^{0}C, the viscosity for 75% UFOME cannot be determined.

It can be said that all biodiesel samples have approximately the same kinematic viscosity at a particular temperature except their point of crystallization. Also, it can be found the empirical coefficients for the kinematic viscosity and composition relationship by using forth order of polynomial regression as in Equation 3.6 and shown an example in Figure 3.13.

$$v = \mathrm{A}x^4 + \mathrm{B}x^3 + \mathrm{C}x^2 + \mathrm{D}x + \mathrm{E} \qquad 3.6$$

where v is kinematic viscosity and x any required percentage of UFOME in the mixture.

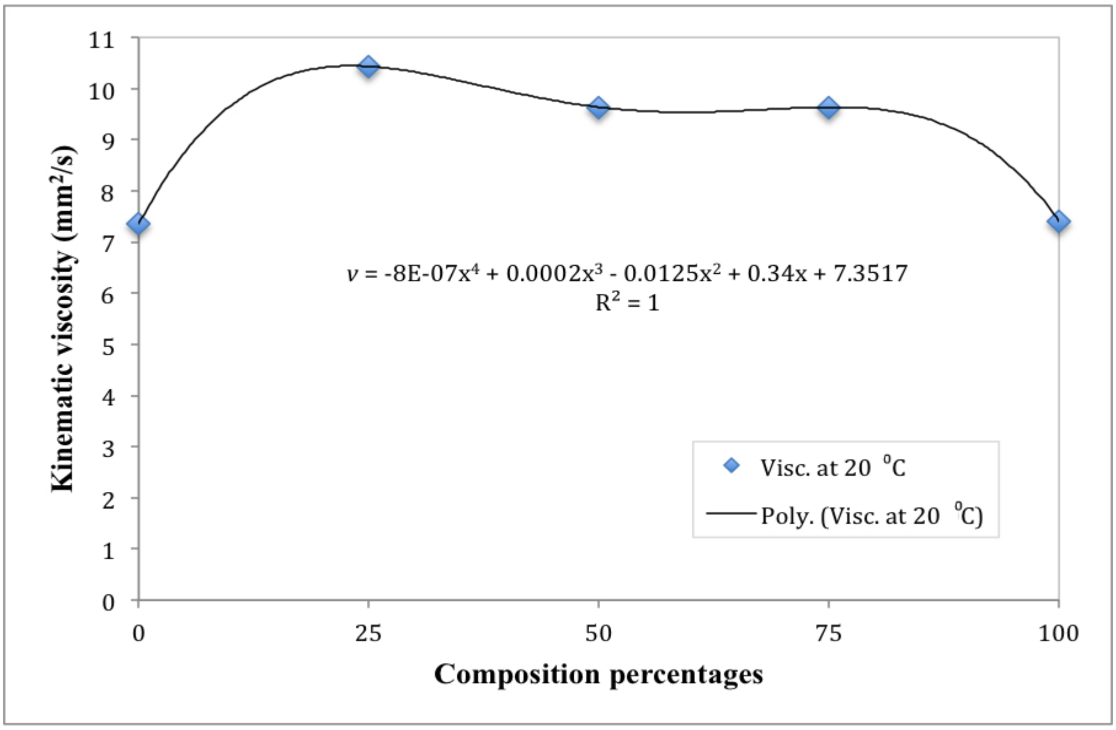

Figure 3.13 Polynomial regressions for percentage compositions at 20 ^{0}C.

In the same way, the regression can be calculated for the other degree of temperatures less than 20 ^{0}C and tabulated in Table 3.5.

Table 3.5 Polynomial coefficients for kinematic viscosity and composition relationship.

Temperature (^{0}C)	A	B	C	D	E	R^2
20	-8.00E-07	2.00E-04	-0.0125	0.3400	7.3517	1
15	-9.00E-07	2.00E-04	-0.0144	0.3983	8.6122	1
10	-	7.00E-05	-0.0102	0.3983	9.7885	1
8	-	-	-0.0051	0.3327	10.415	1
5	-	-	-	0.2294	11.835	1
2	-	-	-	0.2856	13.144	1
0	-	-	-	-	14.030	1
-3	-	-	-	-	15.590	1
-5	-	-	-	-	18.283	1
-7	-	-	-	-	21.517	1
-8	-	-	-	-	23.863	1

In all cases R=1 which means that all correlations are within the acceptable range.

3.3 Cloud Point (CP) and Pour Point (PP)

The cloud point is of concern in cold filter plugging and so it is an estimate of low temperature operability. Pour point is meaningful for improving or handling bulk fuels at low temperatures. All cloud point and pour point results were visually measured. The cloud point was measured by inspecting the biodiesel sample at any degree of temperature until the cloud of wax crystals was visible and the pour point was determined by cooling down the samples until the biodiesel was totally ceased and then the degree before ceased point was recorded. The cloud point and pour point of five different percentages of UCOME and UFOME (100% UCOME, (75% UCOME + 25% UFOME), (50% UCOME + 50% UFOME), (25% UCOME + 75% UFOME) and 100% UFOME) are shown in Figures 3.14 and 3.15. The measured cloud point and pour point are presented in Table 3.6.

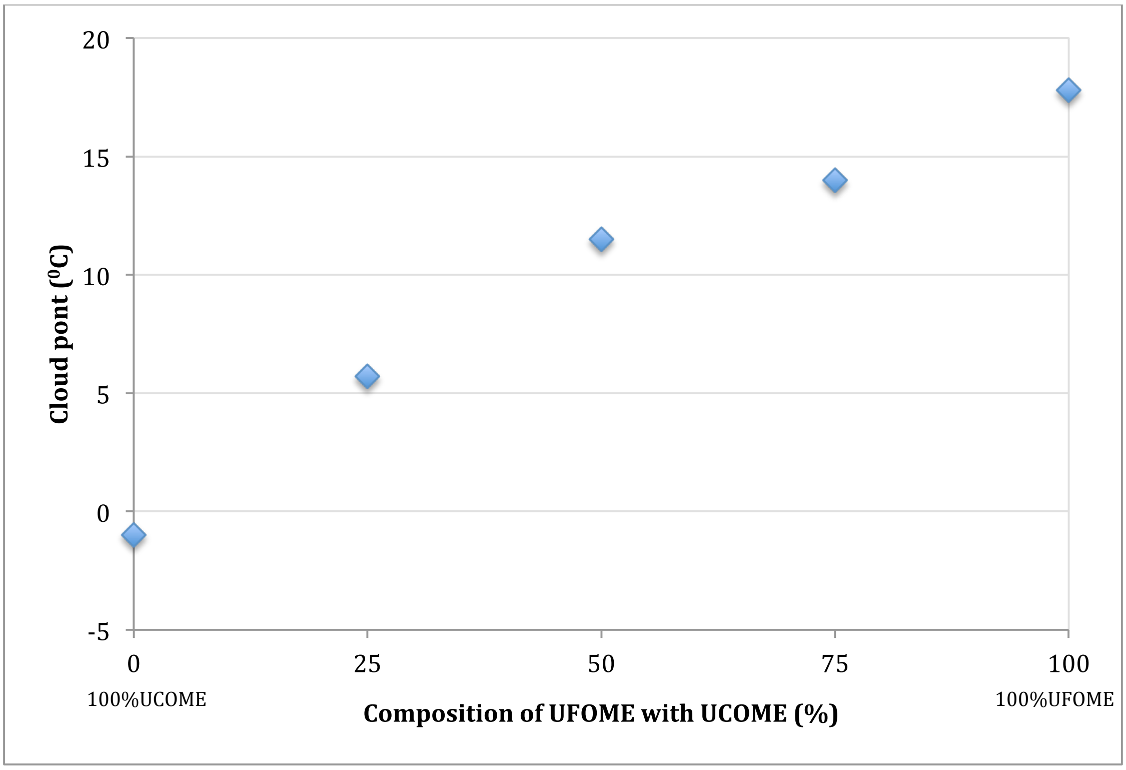

Figure 3.14 Cloud point of different percentage of UFOME with UCOME.

From the figure, as the percentage ratio of UFOME is increased, the cloud point of the mixture will be increased, which at 100% UFOME, the cloud point gets the highest value and at 100% UCOME reaches the lowest value. The cloud point of 100% UFOME is 17.8 ^{0}C, while the cloud point for 100% UCOME is -1 ^{0}C. For each increasing of UFOME by 25%, there is an increase in cloud point.

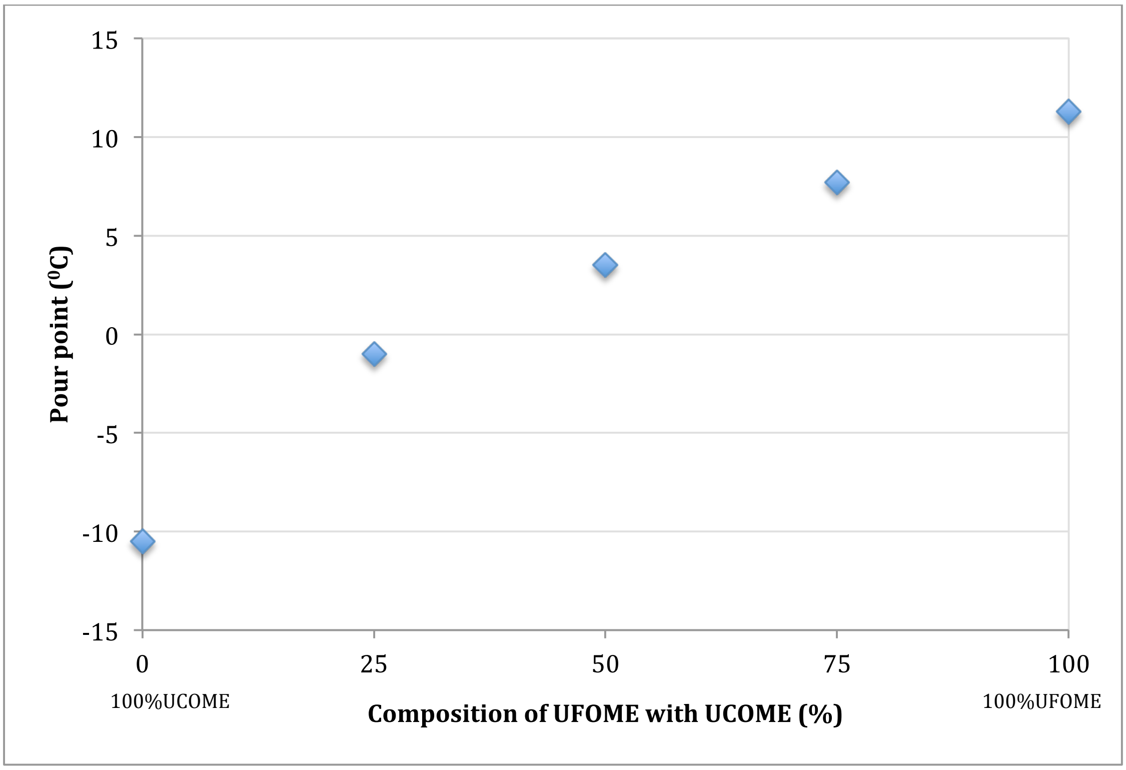

Figure 3.15 Pour point of different percentage of UFOME with UCOME.

From Figure 3.15, the 100% UCOME biodiesel has the lowest pour point but again the 100% UFOME has the highest pour point. By increasing only 25% of UFOME (25% UFOME+75% UCOME), there is a big increasing in pour point from -10.5 ^{0}C to -1 ^{0}C and then by increasing each 25% of UFOME, there is a regularly increase of pour point by about 4 ^{0}C.

Table 3.6 Cloud point and pour point of five biodiesel samples.

Biodiesel samples	UFOME (%)	Cloud Point (CP)	Pour Point (PP)
100% UCOME	0	-1	-10.5
75% UCOME + 25% UFOME	25	5.7	-1
50% UCOME + 50% UFOME	50	11.5	3.5
25% UCOME + 75% UFOME	75	14	7.7
100% UFOME	100	17.8	11.3

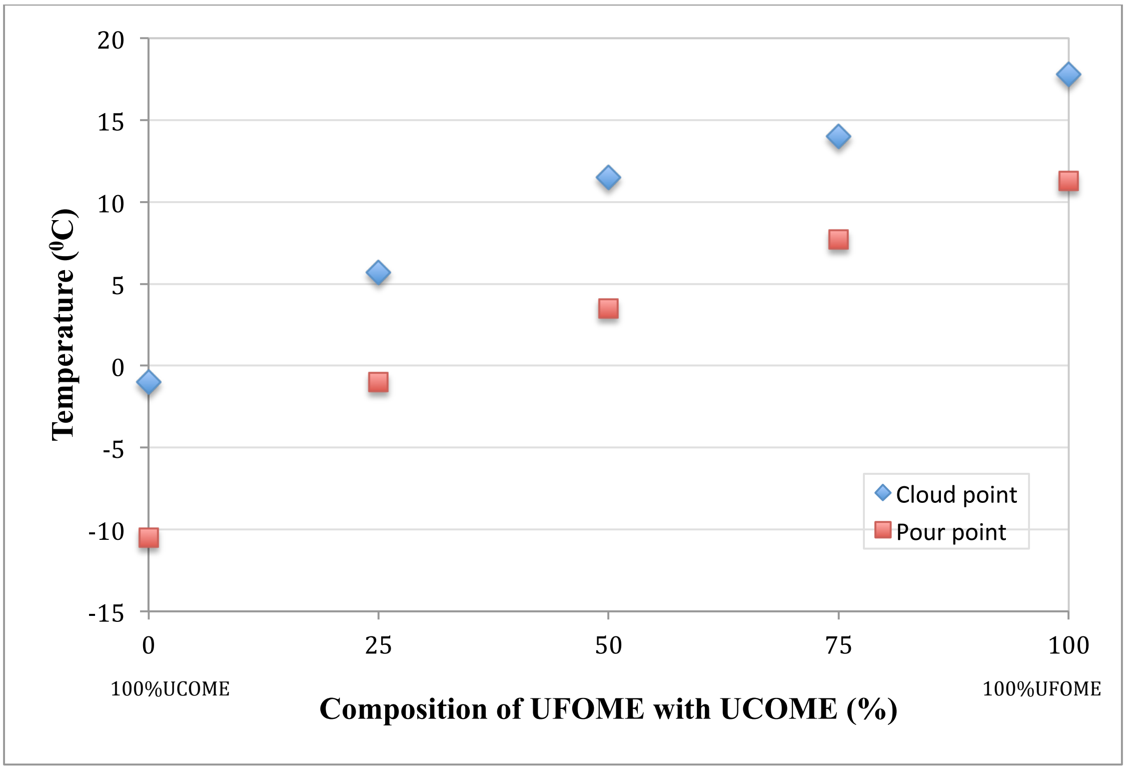

Figure 3.16 The cloud and pour points of different biodiesel composition.

From the Figure 3.16, the pour point is always and slightly lower than the cloud point and this is in a good agreement with previous literatures, as mentioned in Chapter 2 but the difference is varied from biodiesel sample to another sample. The pour point of 100% UCOME is lower from its cloud point by 9.5 ^{0}C but the pour point of 100% UFOME is lower from its cloud point by 6.5 ^{0}C and 50% UCOME+ 50% UFOME by 8 ^{0}C. Both cloud point and pour point increase with increasing the percentage of UFOME to the mixture.

The measurements of the cloud point and pour point have been correlated as function of percentage composition by empirical second order polynomial equations. From the regression coefficient, it was observed that the regression analysis of the data shows that the polynomial equation is better fitted for the measured values than a linear equation [4, 14]. The proposed equations for calculating cloud point and pour point as a function of composition respectively are:

$$T_{cp} = -0.001x^2 + 0.2837x + 272.33 \qquad 3.7$$

$$T_{pp} = -0.0014x^2 + 0.3458x + 263.2 \qquad 3.8$$

where x is the percentage of biodiesel.

The measured and calculated values for both cloud point and pour point are in very good agreement. Equation 3.7 gave a regression coefficient (R^2) of 0.99366 while Equation 3.8 gave a regression coefficient (R^2) of 0.99261 as shown in Figure 3.17.

The results of measured, calculated values and percentage error of the cloud point and pour point of five different biodiesel samples are presented in Table 3.7 and 3.8 respectively.

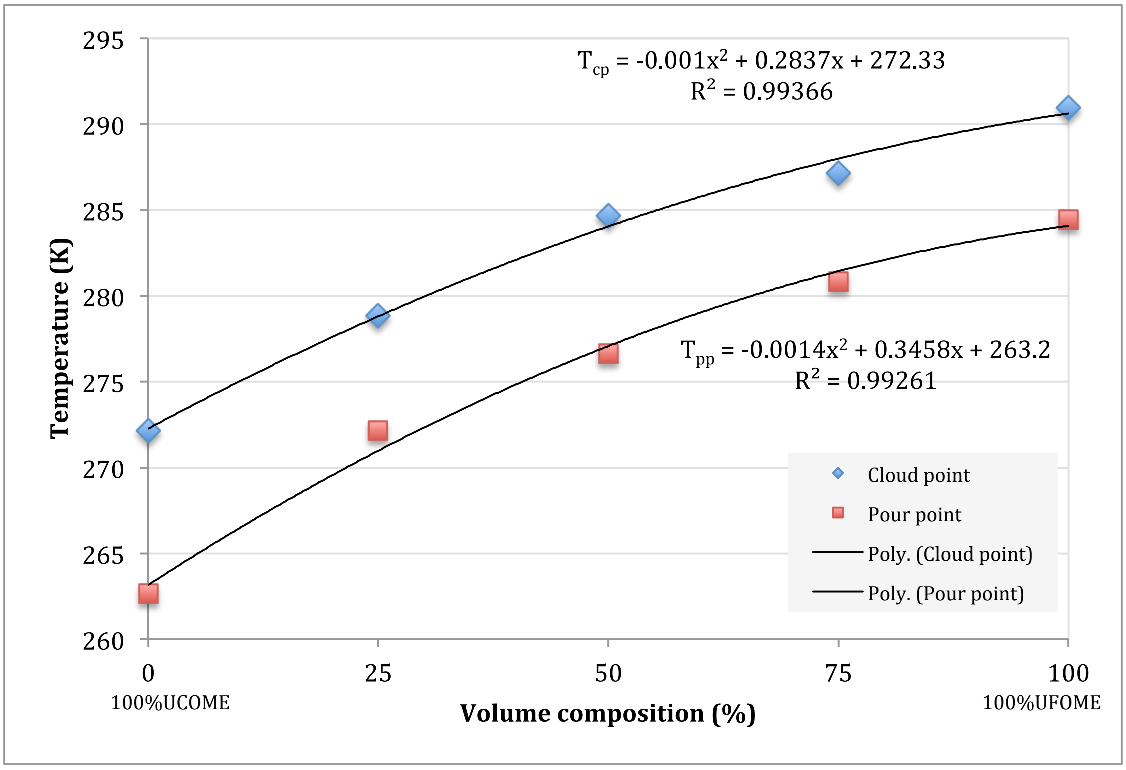

Figure 3.17 Polynomial regressions for cloud point and pour point.

Table 3.7 Measured and calculated cloud point values for the five-biodiesel samples.

Biodiesel samples	UFOME (%)	Measured cloud point (K)	Calculated cloud point (K)	Absolute error in (%)
100% UCOME	0	272.15	272.33	0.0661
75% UCOME + 25% UFOME	25	278.85	278.80	0.0188
50% UCOME + 50% UFOME	50	284.65	284.02	0.2231
25% UCOME + 75% UFOME	75	287.15	287.98	0.2899
100% UFOME	100	290.95	290.70	0.0859

Table 3.8 Measured and calculated pour point values for the five-biodiesel samples.

Biodiesel samples	UFOME (%)	Measured pour point (K)	Calculated pour point (K)	Absolute error in (%)
100% UCOME	0	262.65	263.20	0.2094
75% UCOME + 25% UFOME	25	272.15	270.97	0.4336
50% UCOME + 50% UFOME	50	276.65	276.99	0.1229
25% UCOME + 75% UFOME	75	280.85	281.26	0.1460
100% UFOME	100	284.45	283.78	0.2355

Complete experimental results and data for kinematic viscosity and both cloud and pour points are given in Appendix 4.

CHAPTER 4

CONCLUSIONS

The kinematic viscosity of five biodiesel samples was determined from (20 ^{0}C) down to (-10 ^{0}C) by using Ubbelohde viscometer per ASTM D445-09 and the experimental data were recorded. Also the cloud point and pour point of the biodiesel samples were tested per ASTM D2500-09 and ASTM D97-05 respectively. Based on the results obtained, the following conclusions can be drawn:

- The kinematic viscosity of the samples is a little high and it varies by changing the percentage composition. The highest kinematic viscosity is in (75%UCOME+ 25%UFOME) at any degree of temperature.
- The kinematic viscosity of 100%UFOME cannot be determined at less than 15 ^{0}C because of solidification.
- In all cases, the viscosity of the biodiesel samples increase logarithmically with decrease in temperature, which has been also predicted empirically.
- The cloud point of biodiesel samples always occurs a few degree of temperature above the pour point.
- The cloud point and pour point of 100%UCOME sample increase with increase in concentration of UFOME.
- These biodiesels cannot be used directly in engine because of high viscosity and high cloud point at these degree of temperatures.

These experiments were conducted as accurate as possible, which was discussed in chapter3, but definitely there are some errors such as:

- Equipment's inaccuracy probability.
- Reading and averaging error.
- Non-uniform perfect distribution of temperature in the cooling bath and impossibility of getting the exact temperature inside the capillary tube.
- Quickly changing temperature in the cooling bath, especially at very low temperature.

From the outlook of this work, there are some possibility future works, which are suggested to be done in the future such as:

- Improving cold flow properties of the biodiesel samples by adding some kind of improvers such as conventional diesel fuel (petrodiesel) or alkyl esters.
- Testing the other properties of biodiesel such as density, specific gravity and cold filter plugging point (CFPP).
- Investigation of the effect of pressure on the kinematic viscosity of the biodiesel samples.

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APPENDICES

APPENDIX 1.

ASTM D445-09, ASTM D2500-09 and ASTM D97-05

Designation: D 445 – 09

British Standard 2000: Part 71:1990

Designation: 71/1/97

Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)[1]

This standard is issued under the fixed designation D 445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method specifies a procedure for the determination of the kinematic viscosity, ν, 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, η, can be obtained by multiplying the kinematic viscosity, ν, by the density, ρ, of the liquid.

NOTE 1—For the measurement of the kinematic viscosity and viscosity of bitumens, see also Test Methods D 2170 and D 2171.

NOTE 2—ISO 3104 corresponds to Test Method D 445.

1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.

1.3 The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm^2/s (see Table A1.1) at all temperatures (see 6.3 and 6.4). The precision has only been determined for those materials, kinematic viscosity ranges and temperatures as shown in the footnotes to the precision section.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers
D 1193 Specification for Reagent Water
D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
D 1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer
D 1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer
D 2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards
D 2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)
D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
D 6071 Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atomic Absorption Spectroscopy
D 6074 Guide for Characterizing Hydrocarbon Lubricant Base Oils
D 6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material
E 1 Specification for ASTM Liquid-in-Glass Thermometers
E 77 Test Method for Inspection and Verification of Thermometers

2.2 *ISO Standards:*[3]

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved July 1, 2009. Published August 2009. Originally approved in 1937. Last previous edition approved in 2006 as D 445–06.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

ISO 3104 Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity

ISO 3105 Glass Capillary Kinematic Viscometers—Specification and Operating Instructions

ISO 3696 Water for Analytical Laboratory Use—Specification and Test Methods

ISO 5725 Accuracy (trueness and precision) of measurement methods and results.

ISO 9000 Quality Management and Quality Assurance Standards—Guidelines for Selection and Use

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

2.3 *NIST Standards:*[4]

NIST Technical Note 1297, Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results

NIST GMP 11

NIST Special Publication 819

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *automated viscometer*, *n*—apparatus which, in part or in whole, has mechanized one or more of the procedural steps indicated in Section 11 or 12 without changing the principle or technique of the basic manual apparatus. The essential elements of the apparatus in respect to dimensions, design, and operational characteristics are the same as those of the manual method.

3.1.1.1 *Discussion*—Automated viscometers have the capability to mimic some operation of the test method while reducing or removing the need for manual intervention or interpretation. Apparatus which determine kinematic viscosity by physical techniques that are different than those used in this test method are not considered to be Automated Viscometers.

3.1.2 *density*, *n*—the mass per unit volume of a substance at a given temperature.

3.1.3 *dynamic viscosity*, *n*—the ratio between the applied shear stress and rate of shear of a liquid.

3.1.3.1 *Discussion*—It is sometimes called the coefficient of dynamic viscosity or, simply, viscosity. Thus dynamic viscosity is a measure of the resistance to flow or deformation of a liquid.

3.1.3.2 *Discussion*—The term dynamic viscosity can also be used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusodial time dependence.

3.1.4 *kinematic viscosity*, *n*—the resistance to flow of a fluid under gravity.

3.1.4.1 *Discussion*—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, ν, where $\nu = \eta/\rho$, and η is the dynamic viscosity coefficient.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity (determined value) is the product of the measured flow time and the calibration constant of the viscometer. Two such determinations are needed from which to calculate a kinematic viscosity result that is the average of two acceptable determined values.

5. Significance and Use

5.1 Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

6. Apparatus

6.1 *Viscometers*—Use only calibrated viscometers of the glass capillary type, capable of being used to determine kinematic viscosity within the limits of the precision given in the precision section.

6.1.1 Viscometers listed in Table A1.1, whose specifications meet those given in Specifications D 446 and in ISO 3105 meet these requirements. It is not intended to restrict this test method to the use of only those viscometers listed in Table A1.1. Annex A1 gives further guidance.

6.1.2 *Automated Viscometers*—Automated apparatus may be used as long as they mimic the physical conditions, operations or processes of the manual apparatus. Any viscometer, temperature measuring device, temperature control, temperature controlled bath or timing device incorporated in the automated apparatus shall conform to the specification for these components as stated in Section 6 of this test method. Flow times of less than 200 s are permitted, however, a kinetic energy correction shall be applied in accordance with Section 7 on Kinematic Viscosity Calculation of Specifications D 446. The kinetic energy correction shall not exceed 3.0 % of the measured viscosity. The automated apparatus shall be capable of determining kinematic viscosity of a certified viscosity reference standard within the limits stated in 9.2.1 and Section 17. The precision shall be of statistical equivalence to, or better (has less variability) than the manual apparatus.

NOTE 3—Precision and bias of kinematic viscosity measurements for flow times of less than 200 s has not been determined. The precision stated in Section 17 is not know to be valid for kinematic viscosity measurements with flow times less than 200 s.

6.2 *Viscometer Holders*—Use viscometer holders to enable all viscometers which have the upper meniscus directly above the lower meniscus to be suspended vertically within 1° in all directions. Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0.3° in all directions (see Specifications D 446 and ISO 3105).

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[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

6.2.1 Viscometers shall be mounted in the constant temperature bath in the same manner as when calibrated and stated on the certificate of calibration. See Specifications D 446, see Operating Instructions in Annexes A1–A3. For those viscometers which have Tube L (see Specifications D 446) held vertical, vertical alignment shall be confirmed by using (*1*) a holder ensured to hold Tube L vertical, or (*2*) a bubble level mounted on a rod designed to fit into Tube L, or (*3*) a plumb line suspended from the center of Tube L, or (*4*) other internal means of support provided in the constant temperature bath.

6.3 *Temperature-Controlled Bath*—Use a transparent liquid bath of sufficient depth such, that at no time during the measurement of flow time, any portion of the sample in the viscometer is less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 *Temperature Control*—For each series of flow time measurements, the temperature control of the bath liquid shall be such that within the range from 15 to 100°C, the temperature of the bath medium does not vary by more than ±0.02°C of the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature must not exceed ±0.05°C.

6.4 *Temperature Measuring Device in the Range from 0 to 100°C*—Use either calibrated liquid-in-glass thermometers (Annex A2) of an accuracy after correction of ±0.02°C or better, or any other thermometric device of equal or better accuracy.

6.4.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. The two thermometers shall agree within 0.04°C.

6.4.2 Outside the range from 0 to 100°C, use either calibrated liquid-in-glass thermometers of an accuracy after correction of ±0.05°C or better, or any other thermometric device of equal or better accuracy. When two temperature measuring devices are used in the same bath, they shall agree within ±0.1°C.

6.4.3 When using liquid-in-glass thermometers, such as those in Table A2.1, use a magnifying device to read the thermometer to the nearest ⅕ division (for example, 0.01°C or 0.02°F) to ensure that the required test temperature and temperature control capabilities are met (see 10.1). It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements. This information can be quite useful, especially when investigating issues or causes relating to testing accuracy and precision.

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within ±0.07 % (see Annex A3) of the reading when tested over the minimum and maximum intervals of expected flow times.

6.5.1 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 kinematic viscosity flow time measurements.

7. Reagents and Materials

7.1 *Chromic Acid Cleaning Solution*, or a nonchromium-containing, strongly oxidizing acid cleaning solution. (**Warning—**Chromic acid is a health hazard. It is toxic, a recognized carcinogen, highly corrosive, and potentially hazardous in contact with organic materials. If used, wear a full face-shield and full-length protective clothing including suitable gloves. Avoid breathing vapor. Dispose of used chromic acid carefully as it remains hazardous. Nonchromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.)

7.2 *Sample Solvent*, completely miscible with the sample. Filter before use.

7.2.1 For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

7.3 *Drying Solvent*, a volatile solvent miscible with the sample solvent (see 7.2) and water (see 7.4). Filter before use.

7.3.1 Acetone is suitable. (**Warning—**Extremely flammable.)

7.4 *Water*, deionized or distilled and conforming to Specification D 1193 or Grade 3 of ISO 3696. Filter before use.

8. Certified Viscosity Reference Standards

8.1 Certified viscosity reference standards shall be certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. Viscosity standards shall be traceable to master viscometer procedures described in Test Method D 2162.

8.2 The uncertainty of the certified viscosity reference standard shall be stated for each certified value ($k = 2$, 95% confidence). See ISO 5725 or NIST 1297.

9. Calibration and Verification

9.1 *Viscometers*—Use only calibrated viscometers, thermometers, and timers as described in Section 6.

9.2 *Certified Viscosity Reference Standards* (Table A1.2)—These are for use as confirmatory checks on the procedure in the laboratory.

9.2.1 If the determined kinematic viscosity does not agree within the acceptable tolerance band, as calculated from Annex A4, of the certified value, recheck each step in the procedure, including thermometer and viscometer calibration, to locate the source of error. Annex A1 gives details of standards available.

NOTE 4—In previous issues of Test Method D 445, limits of ±0.35% of the certified value have been used. The data to support the limit of ±0.35% cannot be verified. Annex A4 provides instructions on how to determine the tolerance band. The tolerance band combines both the uncertainty of the certified viscosity reference standard as well as the uncertainty of the laboratory using the certified viscosity reference standard.

9.2.1.1 As an alternative to the calculation in Annex A4, the approximate tolerance bands in Table 1 may be used.

9.2.2 The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

9.3 The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration and this must, therefore, be supplied by the standardization laboratory together with the instrument constant. Where the acceleration of gravity, g, differs by more that 0.1 %, correct the calibration constant as follows:

$$C_2 = (g_2/g_1) \times C_1 \tag{1}$$

where the subscripts 1 and 2 indicate, respectively, the standardization laboratory and the testing laboratory.

10. General Procedure for Kinematic Viscosity

10.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.3.1 taking account of the conditions given in Annex A2 and of the corrections supplied on the certificates of calibration for the thermometers.

10.1.1 Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated.

10.1.2 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used (see 6.4).

10.1.3 They should be viewed with a lens assembly giving approximately five times magnification and be arranged to eliminate parallax errors.

10.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time for manual viscometers shall not be less than 200 s or the longer time noted in Specifications D 446. Flow times of less than 200 s are permitted for automated viscometers, provided they meet the requirements of 6.1.2.

10.2.1 The specific details of operation vary for the different types of viscometers listed in Table A1.1. The operating instructions for the different types of viscometers are given in Specifications D 446.

10.2.2 When the test temperature is below the dew point, fill the viscometer in the normal manner as required in 11.1. To ensure that moisture does not condense or freeze on the walls of the capillary, draw the test portion into the working capillary and timing bulb, place rubber stoppers into the tubes to hold the test portion in place, and insert the viscometer into the bath. After insertion, allow the viscometer to reach bath temperature, and the remove the stoppers. When performing manual viscosity determinations, do not use those viscometers which cannot be removed from the constant temperature bath for charging the sample portion.

TABLE 1 Approximate Tolerance Bands

NOTE—The tolerance bands were determined using Practice D 6617. The calculation is documented in Research Report RR: D02–1498.[A]

Viscosity of Reference Material, mm²/s	Tolerance Band
< 10	±0.30%
10 to 100	±0.32%
100 to 1000	±0.36%
1000 to 10 000	±0.42%
10 000 to 100 000	±0.54%
> 100 000	±0.73%

[A] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1498.

10.2.2.1 The use of loosely packed drying tubes affixed to the open ends of the viscometer is permitted, but not required. If used, the drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument.

10.2.3 Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers.

11. Procedure for Transparent Liquids

11.1 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample is thought or known to contain fibers or solid particles, filter through a 75 µm screen, either prior to or during charging (see Specifications D 446).

NOTE 5—To minimize the potential of particles passing through the filter from aggregating, it is recommended that the time lapse between filtering and charging be kept to a minimum.

11.1.1 In general, the viscometers used for transparent liquids are of the type listed in Table A1.1, A and B.

11.1.2 With certain products which exhibit *gel-like* behavior, exercise care that flow time measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameters.

11.1.3 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring a flow time.

11.1.4 Because this time will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial.

11.1.4.1 Thirty minutes should be sufficient except for the highest kinematic viscosities.

11.1.5 Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

11.2 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. With the sample flowing freely, measure, in seconds to within 0.1 s, the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum (see 10.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

11.2.1 Repeat the procedure described in 11.2 to make a second measurement of flow time. Record both measurements.

11.2.2 From the two measurements of flow time, calculate two determined values of kinematic viscosity.

11.2.3 If the two determined values of kinematic viscosity calculated from the flow time measurements agree within the stated determinability figure (see 17.1.1) for the product, use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If not, repeat the measurements of flow times after a thorough cleaning and drying of the viscometers and filtering (where required, see 11.1) of the sample until the calculated kinematic viscosity determinations agree with the stated determinability.

11.2.4 If the material or temperature, or both, is not listed in 17.1.1, use 1.5% as an estimate of the determinability.

12. Procedure for Opaque Liquids

12.1 For steam-refined cylinder oils and black lubricating oils, proceed to 12.3 ensuring a thoroughly representative sample is used. The kinematic viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure described in 12.1.1-12.2.2 shall be followed to minimize this.

12.1.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table A1.1, C.

12.1.2 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

12.1.3 Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

12.1.4 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

12.1.4.1 With samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60°C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

12.2 Immediately after completing 12.1.4, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

12.2.1 Immerse the flask in a bath of boiling water for 30 min. (**Warning—**Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures.)

12.2.2 Remove the flask from the bath, stopper tightly, and shake for 60 s.

12.3 Two determinations of the kinematic viscosity of the test material are required. For those viscometers that require a complete cleaning after each flow time measurement, two viscometers may be used. A single viscometer in which an immediate, repeat flow time measurement can be made without cleaning may also be used for the two measurements of flow time and calculation of kinematic viscosity. Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 75-µm filter into two viscometers previously placed in the bath. For samples subjected to heat treatment, use a preheated filter to prevent the sample coagulating during the filtration.

12.3.1 Viscometers which are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample. This is to ensure that the sample will not be cooled below test temperature.

12.3.2 After 10 min, adjust the volume of the sample (where the design of the viscometer requires) to coincide with the filling marks as in the viscometer specifications (see Specifications D 446).

12.3.3 Allow the charged viscometers enough time to reach the test temperature (see 12.3.1). Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring flow time.

12.4 With the sample flowing freely, measure in seconds to within 0.1 s, the time required for the advancing ring of contact to pass from the first timing mark to the second. Record the measurement.

12.4.1 In the case of samples requiring heat treatment described in 12.1 through 12.2.1, complete the measurements of flow time within 1 h of completing 12.2.2. Record the measured flow times.

12.5 Calculate kinematic viscosity, ν, in mm^2/s, from each measured flow time. Regard these as two determined values of kinematic viscosity.

12.5.1 For residual fuel oils, if the two determined values of kinematic viscosity agree within the stated determinability figure (see 17.1.1), use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If the calculated kinematic viscosities do not agree, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering of the sample. If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100°C use as an estimate of the determinability 1.0 %, and 1.5 % for temperatures outside this range; it must be realized that these materials can be non-Newtonian, and can contain solids which can come out of solution as the flow time is being measured.

13. Cleaning of Viscometer

13.1 Between successive determinations of kinematic viscosity, clean the viscometer thoroughly by several rinsings with the sample solvent, followed by the drying solvent (see 7.3). Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

13.2 Periodically clean the viscometer with the cleaning solution (**Warning**—see 7.1), for several hours to remove residual traces of organic deposits, rinse thoroughly with water (7.4) and drying solvent (see 7.3), and dry with filtered dry air or a vacuum line. Remove any inorganic deposits by hydrochloric acid treatment before the use of cleaning acid, particularly if the presence of barium salts is suspected. (**Warning—**It is essential that alkaline cleaning solutions are not used as changes in the viscometer calibration can occur.)

14. Calculation

14.1 Calculate each of the determined kinematic viscosity values, ν_1 and ν_2, from the measured flow times, t_1 and t_2, and the viscometer constant, C, by means of the following equation:

$$\nu_{1,2} = C \cdot t_{1,2} \tag{2}$$

where:

$\nu_{1,2}$ = determined kinematic viscosity values for ν_1 and ν_2, respectively, mm^2/s,
C = calibration constant of the viscometer, mm^2/s^2, and
$t_{1,2}$ = measured flow times for t_1 and t_2, respectively, s.

Calculate the kinematic viscosity result, ν, as an average of ν_1 and ν_2 (see 11.2.3 and 12.5.1).

14.2 Calculate the dynamic viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3} \tag{3}$$

where:

η = dynamic viscosity, mPa·s,
ρ = density, kg/m^3, at the same temperature used for the determination of the kinematic viscosity, and
ν = kinematic viscosity, mm^2/s.

14.2.1 The density of the sample can be determined at the test temperature of the kinematic viscosity determination by an appropriate method such as Test Methods D 1217, D 1480, or D 1481.

15. Expression of Results

15.1 Report the test results for the kinematic or dynamic viscosity, or both, to four significant figures, together with the test temperature.

16. Report

16.1 Report the following information:

16.1.1 Type and identification of the product tested,

16.1.2 Reference to this test method or a corresponding international standard,

16.1.3 Result of the test (see Section 15),

16.1.4 Any deviation, by agreement or otherwise, from the procedure specified,

16.1.5 Date of the test, and

16.1.6 Name and address of the test laboratory.

17. Precision

17.1 *Comparison of Determined Values*:

17.1.1 *Determinability (d)*—The difference between successive determined values obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, would in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C[5]	0.0020 y	(0.20 %)
Formulated oils at 40 and 100°C[6]	0.0013 y	(0.13 %)
Formulated oils at 150°C[7]	0.015 y	(1.5 %)
Petroleum wax at 100°C[8]	0.0080 y	(0.80 %)
Residual fuel oils at 80 and 100°C[9]	0.011 (y + 8)	
Residual fuel oils at 50°C[9]	0.017 y	(1.7 %)
Additives at 100°C[10]	$0.00106\ y^{1.1}$	
Gas oils at 40°C[11]	0.0013 (y+1)	
Jet fuels at –20°C[12]	0.0018 y	(0.18 %)

where: *y* is the average of determined values being compared.

17.2 *Comparison of Results*:

17.2.1 *Repeatability (r)*—The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C[5]	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C[6]	0.0026 x	(0.26 %)
Formulated oils at 150°C[7]	0.0056 x	(0.56 %)
Petroleum wax at 100°C[8]	$0.0141\ x^{1.2}$	
Residual fuel oils at 80 and 100°C[9]	0.013 (x + 8)	
Residual oils at 50°C[9]	0.015 x	(1.5 %)
Additives at 100°C[10]	$0.00192\ x^{1.1}$	
Gas oils at 40°C[11]	0.0043 (x+1)	
Jet fuels at –20°C[12]	0.007 x	(0.7 %)

where: *x* is the average of results being compared.

17.2.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D02-1331 and RR:D02-1132. These precision values were obtained by statistical examination of interlaboratory results from six mineral oils (base oils without additive package) in the range from 8 to 1005 mm^2/s at 40°C and from 2 to 43 mm $^2/s$ at 100°C, and were first published in 1989. Request See Guide D 6074.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1332. These precision values were obtained by statistical examination of interlaboratory results from seven fully formulated engine oils in the range from 36 to 340 mm $^2/s$ at 40°C and from 6 to 25 mm^2/s at 100°C, and were first published in 1991. See Guide D 6071.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1333. These precision values were obtained by statistical examination of interlaboratory results for eight fully formulated engine oils in the range from 7 to 19 mm^2 /s at 150°C, and first published in 1991. See Guide D 6074.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1334. These precision values were obtained by statistical examination of interlaboratory results from five petroleum waxes in the range from 3 to 16 mm^2/s at 100°C, and were first published in 1988.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1198. These precision values were obtained by statistical examination of interlaboratory results from fourteen residual fuel oils in the range from 30 to 1300 mm^2/s at 50°C and from 5 to 170 mm^2/s at 80 and 100°C, and were first published in 1984.

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1421. These precision values were obtained by statistical examination of interlaboratory results from eight additives in the range from 145 to 1500 mm^2/s at 100°C and were first available in 1997.

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1422. These precision values were obtained by statistical examination of interlaboratory results from eight gas oils in the range from 1 to 13 mm^2/s at 40°C and were first available in 1997.

[12] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1420. These precision values were obtained by statistical examination of interlaboratory results from nine jet fuels in the range from 4.3 to 5.6 mm^2/s at– 20°C and were first available in 1997.

Base oils at 40 and 100°C[5]	0.0065 x	(0.65 %)
Formulated oils at 40 and 100°C[6]	0.0076 x	(0.76 %)
Formulated oils at 150°C[7]	0.018 x	(1.8 %)
Petroleum wax at 100°C[8]	0.0366 $x^{1.2}$	
Residual fuel oils at 80 and 100°C[9]	0.04 (x + 8)	
Residual oils at 50°C[9]	0.074 x	(7.4 %)
Additives at 100°C[10]	0.00862 $x^{1.1}$	
Gas oils at 40°C[11]	0.0082 (x+1)	
Jet fuels at –20°C[12]	0.019 x	(1.9 %)

where: *x* is the average of results being compared.

17.3 The precision for used oils has not been determined but is expected to be poorer than that for formulated oils. Because of the extreme variability of such used oils, it is not anticipated that the precision of used oils will be determined.

17.4 The precision for specific automated viscometers has not been determined. However, an analysis has been made of a large data set including both automated and manual viscometers over the temperature range of 40 to 100°C. The reproducibility of automated viscometer data is not statistically significantly different than the reproducibility of manual viscometer data. It is also shown that there is no bias of the automated data in comparison to the manual data.[13]

18. Keywords

18.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

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[13] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1498.

ANNEXES

(Mandatory Information)

A1. VISCOMETER TYPES AND CERTIFIED VISCOSITY REFERENCE STANDARDS

A1.1 Viscometer Types

A1.1.1 Table A1.1 lists capillary viscometers commonly in use for viscosity determinations on petroleum products. For specifications, operating instructions, and calibration, refer to specifications in Specifications D 446.

A1.1.2 Table A1.2 lists certified viscosity reference standards.

TABLE A1.1 Viscometer Types

Viscometer Identification	Kinematic Viscosity Range,[A] mm²/s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine[B]	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube[B]	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL[B]	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch[B]	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL[B]	3.5 to 100 000
BS/IP/SL(S)[B]	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde[B]	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic[B]	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution[B](B)	0.5 to 100 000
Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow Types for Transparent and Opaque Liquids	
Cannon-Fenske opaque	0.4 to 20 000
Zeitfuchs cross-arm	0.6 to 100 000
BS/IP/RF U-tube reverse-flow	0.6 to 300 000
Lantz-Zeitfuchs type reverse-flow	60 to 100 000

[A] 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 s except where noted in Specifications D 446.

[B] In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

TABLE A1.2 Certified Viscosity Reference Standards

Designation	Approximate Kinematic Viscosity, mm²/s					
	20°C	25°C	40°C	50°C	80 °C	100°C
S3	4.6	4.0	2.9	...	...	1.2
S6	11	8.9	5.7	...	...	1.8
S20	44	34	18	...	...	3.9
S60	170	120	54	...	...	7.2
S200	640	450	180	...	...	17
S600	2400	1600	520	280	67	32
S2000	8700	5600	1700	...	...	75
S8000	37 000	23 000	6700	...	...	...
S30 000	...	81 000	23 000	11 000	...	...

A2. KINEMATIC VISCOSITY TEST THERMOMETERS

A2.1 Short-Range Specialized Thermometer

A2.1.1 Use a short-range specialized thermometer conforming to the generic specification given in Table A2.1 and Table A2.2 and to one of the designs shown in Fig. A2.1.

A2.1.2 The difference in the designs rests mainly in the position of the ice point scale. In Design A, the ice point is within the scale range, in Design B, the ice point is below the scale range, and in Design C, the ice point is above the scale range.

A2.2 Calibration

A2.2.1 Use liquid-in-glass thermometers with an accuracy after correction of 0.02°C or better, calibrated by a laboratory meeting the requirements of ISO 9000 or ISO 17025, and carrying certificates confirming that the calibration is traceable to a national standard. As an alternative, use thermometric devices such as platinum resistance thermometers, of equal or better accuracy, with the same certification requirements.

A2.2.2 The scale correction of liquid-in-glass thermometers can change during storage and use, and therefore regular re-calibration is required. This is most conveniently achieved in a working laboratory by means of a re-calibration of the ice point, and all of the main scale corrections altered for the change seen in the ice point.

TABLE A2.1 General Specification for Thermometers

NOTE—Table A2.2 gives a range of ASTM, IP, and ASTM/IP thermometers that comply with the specification in Table A2.1, together with their designated test temperatures. See Specification E 1 and Test Method E 77.

Immersion		Total
Scale marks:		
Subdivisions	°C	0.05
Long lines at each	°C	0.1 and 0.5
Numbers at each	°C	1
Maximum line width	mm	0.10
Scale error at test temperature, max	°C	0.1
Expansion chamber:		
Permit heating to	°C	105 up to 90, 120 between 90 and 95 130 between 95 and 105, 170 above 105
Total length	mm	300 to 310
Stem outside diameter	mm	6.0 to 8.0
Bulb length	mm	45 to 55
Bulb outside diameter	mm	no greater than stem
Length of scale range	mm	40 to 90

TABLE A2.2 Complying Thermometers

Thermometer No.	Test Temperature		Thermometer No.	Test Temperature	
	°C	°F		°C	°F
ASTM 132C, IP 102C	150		ASTM 128C, F/IP 33C	0	32
ASTM 110C, F/IP 93C	135	275	ASTM 72C, F/IP 67C	–17.8	0
ASTM 121C/IP 32C	98.9,	210,	ASTM 127C/IP 99C	–20	–4
	100	212	ASTM 126C, F/IP 71C	–26.1	–20
ASTM 129C, F/IP 36C	93.3	200	ASTM 73C, F/IP 68C	–40	–40
ASTM 48C, F/IP 90C	82.2	180	ASTM 74C, F/IP 69C	–53.9	–65
IP 100C	80				
ASTM 47C, F/IP 35C	60	140			
ASTM 29C, F/IP 34C	54.4	130			
ASTM 46C F/IP 66C	50	122			
ASTM 120C/IP 92C	40				
ASTM 28C, F/IP 31C	37.8	100			
ASTM 118C, F	30	86			
ASTM 45C, F/IP 30C	25	77			
ASTM 44C, F/IP 29C	20	68			

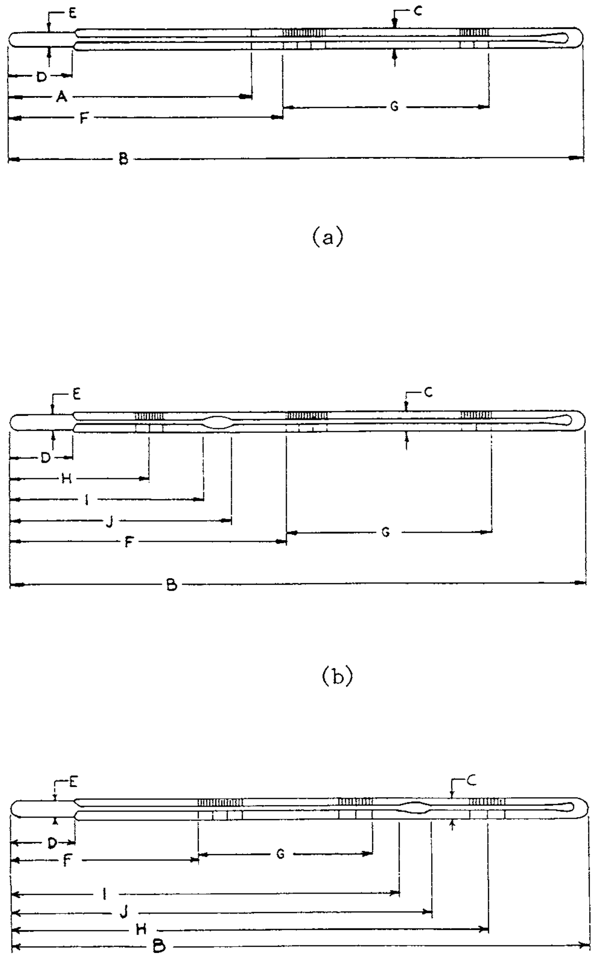
A2.2.2.1 The interval for ice-point recalibration shall be no longer than six months (see NIST GMP 11). For new thermometers, monthly checking for the first six months is recommended. A change of one or more scale divisions in the ice point means that the thermometer may have been overheated or damaged, and it may be out of calibration. Such thermometers shall be removed from service until inspected, or recalibrated, or both. A complete recalibration of the thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to this design thermometer (see NIST Special Publication 819). Any change in ice-point correction shall be added to the other corrections of the original Report of Calibration.

A2.2.2.2 Other thermometric devices, if used, will also require periodic recalibration. Keep records of all recalibration.

A2.2.3 *Procedure for Ice-point Recalibration of Liquid-in-glass Thermometers.*

A2.2.3.1 Unless otherwise listed on the certificate of calibration, the recalibration of calibrated kinematic viscosity thermometers requires that the ice-point reading shall be taken within 60 min after being at test temperature for not less than 3 min.

A2.2.3.2 Select clear pieces of ice, preferably made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the



(a)

(b)

(c)

FIG. A2.1 Thermometer Designs

crushed ice and add sufficient water to form a slush, but not enough to float the ice. As the ice melts, drain off some of the water and add more crushed ice. Insert the thermometer, and pack the ice gently about the stem, to a depth approximately one scale division below the 0°C graduation.

A2.2.3.3 After at least 3 min have elapsed, tap the thermometer gently and repeatedly at right angles to its axis while making observations. Successive readings taken at least 1 min apart shall agree within 0.005°C.

A2.2.3.4 Record the ice-point readings and determine the thermometer correction at this temperature from the mean reading. If the correction is found to be higher or lower than that corresponding to a previous calibration, change the correction at all other temperatures by the same value.

A2.2.3.5 During the procedure, apply the following conditions:

(*1*) The thermometer shall be supported vertically.

(*2*) View the thermometer with an optical aid that gives a magnification of approximately five and also eliminates parallax.

(*3*) Express the ice-point reading to the nearest 0.005°C.

A2.2.4 When in use, immerse the thermometric device to the same depth as when it was fully calibrated. For example, if a liquid-in-glass thermometer was calibrated at the normal total immersion condition, it shall be immersed to the top of the mercury column with the remainder of the stem and the expansion volume at the uppermost end exposed to room temperature and pressure. In practice, this means that the top of the mercury column shall be within a length equivalent to four scale divisions of the surface of the medium whose temperature is being measured.

A2.2.4.1 If this condition cannot be met, then an extra correction may be necessary.

A3. TIMER ACCURACY

A3.1 Regularly check timers for accuracy and maintain records of such checks.

A3.1.1 Time signals as broadcast by the National Institute of Standards and Technology are a convenient and primary standard reference for calibrating timing devices. The following can be used to an accuracy of 0.1 s:

WWV	Fort Collins, CO	2.5, 5, 10, 15, 20 MHz
WWVH	Kauai, HI	2.5, 5, 10, 15, MHz
CHU	Ottawa, Canada	3.33, 7.335, 14.67 MHz

A3.1.2 Radio broadcast of voice and audio on a telephone line at phone 303-499-7111. Additional time services are available from the National Institute of Standards and Technology.

A4. CALCULATION OF ACCEPTABLE TOLERANCE ZONE (BAND) TO DETERMINE CONFORMANCE WITH A CERTIFIED REFERENCE MATERIAL

A4.1 Determine the standard deviation for site uncertainty, σ_{site}, from a laboratory quality control program.

A4.1.1 If the standard deviation for site uncertainty, σ_{site}, is not known, use the value 0.19%.

A4.2 Determine the combined extended uncertainty (CEU) of the accepted reference value (ARV) of the certified reference material (CRM) from the supplier's label or included documentation.

A4.3 Calculate the standard error of the accepted reference value (SEARV) by dividing the CEU by the coverage factor, *k*, listed on the supplier's label or included documentation.

A4.3.1 If the coverage factor, *k*, is not known, use the value 2.

A4.4 Construct the acceptable tolerance zone:

$$TZ = \pm 1.44 \sqrt{\sigma_{site}^2 + SE_{ARV}^2}$$

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 445–06) that may impact the use of this standard.

(*1*) Change determinability requirements for materials of unknown precision in 11.2.4 as a temporary measure pending revision of the Precision and Bias section.

Designation: D 2500 – 09

An American National Standard
British Standard 4458

Designation: 219/82

Standard Test Method for Cloud Point of Petroleum Products[1]

This standard is issued under the fixed designation D 2500; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method covers only petroleum products and biodiesel fuels that are transparent in layers 40 mm in thickness, and with a cloud point below 49°C.

NOTE 1—The interlaboratory program consisted of petroleum products of Test Method D 1500 color of 3.5 and lower. The precisions stated in this test method may not apply to samples with ASTM color higher than 3.5.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standard:*[3]

Specifications for IP Standard Thermometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *biodiesel*, *n*—a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.

3.1.1.1 *Discussion*—Biodiesel is typically produced by a reaction of vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-esters and glycerin. The fuel typically may contain up to 14 different types of fatty acids that are chemically transformed into fatty acid methyl esters (FAME).

3.1.2 *biodiesel blend*, *n*—a blend of biodiesel fuel with petroleum-based diesel fuel designated BXX, where XX is the volume % of biodiesel.

3.1.3 *cloud point*, *n—in petroleum products and biodiesel fuels,* the temperature of a liquid specimen when the smallest observable cluster of hydrocarbon crystals first occurs upon cooling under prescribed conditions.

3.1.3.1 *Discussion*—To many observers, the cluster of wax crystals looks like a patch of whitish or milky cloud, hence the name of the test method. The cloud appears when the temperature of the specimen is low enough to cause wax crystals to form. For many specimens, the crystals first form at the lower circumferential wall of the test jar where the temperature is lowest. The size and position of the cloud or cluster at the cloud point varies depending on the nature of the specimen. Some samples will form large, easily observable, clusters, while others are barely perceptible.

3.1.3.2 *Discussion*—Upon cooling to temperatures lower than the cloud point, clusters of crystals will grow in multiple directions; for example, around the lower circumference of the test jar, towards the center of the jar, or vertically upwards. The crystals can develop into a ring of cloud along the bottom circumference, followed by extensive crystallization across the bottom of the test jar as temperature decreases. Nevertheless, the cloud point is defined as the temperature at which the crystals first appear, not when an entire ring or full layer of wax has been formed at the bottom of the test jar.

3.1.3.3 *Discussion*—In general, it is easier to detect the cloud point of samples with large clusters that form quickly, such as paraffinic samples. The contrast between the opacity of the cluster and the liquid is also sharper. In addition, small brightly-reflective spots can sometimes be observed inside the cluster when the specimen is well illuminated. For other more difficult samples, such as naphthenic, hydrocracked, and those samples whose cold flow behavior have been chemically

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved April 15, 2009. Published April 2009. Originally approved in 1966. Last previous edition approved in 2005 as D 2500–05.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

***A Summary of Changes section appears at the end of this standard.**

altered, the appearance of the first cloud can be less distinct. The rate of crystal growth is slow, the opacity contrast is weak, and the boundary of the cluster is more diffuse. As the temperature of these specimens decrease below the cloud point, the diffuse cluster will increase in size and can form a general haze throughout. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature of the specimen decreases, can also be caused by traces of water in the specimen instead of crystal formation (see Note 3). With these difficult samples, drying the sample prior to testing can eliminate this type of interference.

3.1.3.4 *Discussion*—The purpose of the cloud point method is to detect the presence of the wax crystals in the specimen; however trace amounts of water and inorganic compounds may also be present. The intent of the cloud point method is to capture the temperature at which the liquids in the specimen begin to change from a single liquid phase to a two-phase system containing solid and liquid. It is not the intent of this test method to monitor the phase transition of the trace components, such as water.

4. Summary of Test Method

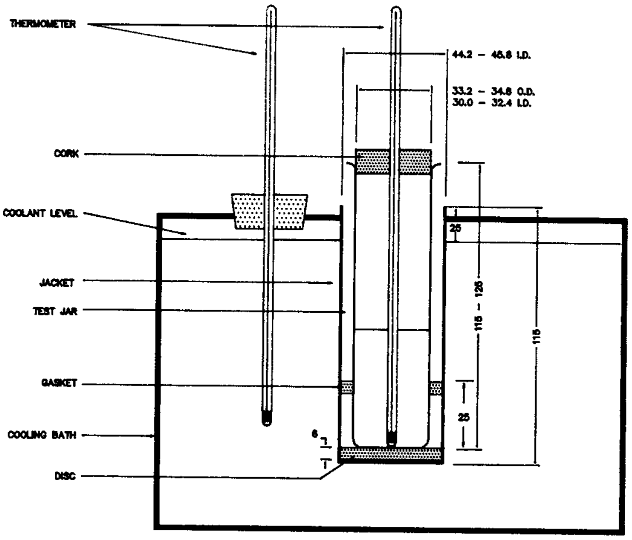
4.1 The specimen is cooled at a specified rate and examined periodically. The temperature at which a cloud is first observed at the bottom of the test jar is recorded as the cloud point.

5. Significance and Use

5.1 For petroleum products and biodiesel fuels, cloud point of a petroleum product is an index of the lowest temperature of their utility for certain applications.

6. Apparatus (see Fig. 1)

6.1 *Test Jar*, clear, cylindrical glass, flat bottom, 33.2 to 34.8-mm outside diameter and 115 and 125-mm height. The inside diameter of the jar may range from 30 to 32.4 mm within the constraint that the wall thickness be no greater than 1.6 mm. The jar should be marked with a line to indicate sample height 54 ± 3 mm above the inside bottom.



NOTE—All dimensions are in millimetres.

FIG. 1 Apparatus for Cloud Point Test

6.2 *Thermometers*, having ranges shown below and conforming to the requirements as prescribed in Specification E 1 or Specifications for IP Standard Thermometers.

		Thermometer Number	
Thermometer	Temperature Range	ASTM	IP
High cloud and pour	–38 to +50°C	5C	1C
Low cloud and pour	–80 to +20°C	6C	2C

6.3 *Cork*, to fit the test jar, bored centrally for the test thermometer.

6.4 *Jacket*, metal or glass, watertight, cylindrical, flat bottom, about 115 mm in depth, with an inside diameter of 44.2 to 45.8 mm. It shall be supported free of excessive vibration and firmly in a vertical position in the cooling bath of 6.7 so that not more than 25 mm projects out of the cooling medium.

6.5 *Disk*, cork or felt, 6-mm thick to fit loosely inside the jacket.

6.6 *Gasket*, ring form, about 5 mm in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket may be made of rubber, leather, or other material that is elastic enough to cling to the test jar and hard enough to hold its shape. Its purpose is to prevent the test jar from touching the jacket.

6.7 *Bath or Baths*, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable cooling mixtures. Cooling mixtures commonly used for bath temperatures shown are in Table 1.

7. Reagents and Materials

7.1 *Acetone*—Technical grade acetone is suitable for the cooling bath, provided it does not leave a residue on drying. (**Warning—**Extremely flammable.)

7.2 *Carbon Dioxide (Solid) or Dry Ice*—A commercial grade of dry ice is suitable for use in the cooling bath.

7.3 *Petroleum Naphtha*—A commercial or technical grade of petroleum naphtha is suitable for the cooling bath. (**Warning—**Combustible. Vapor harmful.)

7.4 *Sodium Chloride Crystals*—Commercial or technical grade sodium chloride is suitable.

7.5 *Sodium Sulfate*—A reagent grade of anhydrous sodium sulfate should be used when required (see Note 3).

TABLE 1 Cooling Mixtures and Bath Temperatures

	Bath Temperature
Ice and water	0 ± 1.5°C
Crushed ice and sodium chloride crystals, or acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	–18 ± 1.5°C
Acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	–33 ± 1.5°C
Acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	–51 ± 1.5°C
Acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	–69 ± 1. 5°C

8. Procedure

8.1 Bring the sample to be tested to a temperature at least 14°C above the expected cloud point. Remove any moisture present by a method such as filtration through dry lintless filter paper until the oil is perfectly clear, but make such filtration at a temperature of at least 14°C above the approximate cloud point.

8.2 Pour the sample into the test jar to the level mark.

8.3 Close the test jar tightly by the cork carrying the test thermometer. Use the high cloud and pour thermometer if the expected cloud point is above −36°C and the low cloud and pour thermometer if the expected cloud point is below −36°C. Adjust the position of the cork and the thermometer so that the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is resting on the bottom of the jar.

NOTE 2—Liquid column separation of thermometers occasionally occurs and may escape detection. Thermometers should be checked periodically and used only if their ice points are 0 ± 1°C, when the thermometer is immersed to the immersion line in an ice bath, and when the emergent column temperature does not differ significantly from 21°C. Alternatively, immerse the thermometer to a reading and correct for the resultant cooler stem temperature.

8.4 See that the disk, gasket, and the inside of the jacket are clean and dry. Place the disk in the bottom of the jacket. The disk and jacket shall have been placed in the cooling medium a minimum of 10 min before the test jar is inserted. The use of a jacket cover while the empty jacket is cooling is permitted. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

NOTE 3—Failure to keep the disk, gasket, and the inside of the jacket clean and dry may lead to frost formation, which may cause erroneous results.

8.5 Maintain the temperature of the cooling bath at 0 ± 1.5°C.

8.6 At each test thermometer reading that is a multiple of 1°C, remove the test jar from the jacket quickly but without disturbing the specimen, inspect for cloud, and replace in the jacket. This complete operation shall require not more than 3 s. If the oil does not show a cloud when it has been cooled to 9°C, transfer the test jar to a jacket in a second bath maintained at a temperature of −18 ± 1.5°C (see Table 2). Do not transfer the jacket. If the specimen does not show a cloud when it has been cooled to −6°C, transfer the test jar to a jacket in a third bath maintained at a temperature of −33± 1.5°C. For the determination of very low cloud points, additional baths are required, each bath to be maintained in accordance with Table 2. In each case, transfer the jar to the next bath, if the specimen does not exhibit cloud point and the temperature of the specimen reaches the lowest specimen temperature in the range identified for the current bath in use, based on the ranges stated in Table 2.

TABLE 2 Bath and Sample Temperature Ranges

Bath	Bath Temperature Setting, °C	Sample Temperature Range, °C
1	0 ± 1.5	Start to 9
2	−18 ± 1.5	9 to −6
3	−33 ± 1.5	−6 to −24
4	−51 ± 1.5	−24 to −42
5	−69 ± 1.5	−42 to −60

8.7 Report the cloud point, to the nearest 1°C, at which any cloud is observed at the bottom of the test jar, which is confirmed by continued cooling.

NOTE 4—A wax cloud or haze is always noted first at the bottom of the test jar where the temperature is lowest. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature is lowered, is usually due to traces of water in the oil. Generally this water haze will not interfere with the determination of the wax cloud point. In most cases of interference, filtration through dry lintless filter papers, such as described in 8.1, is sufficient. In the case of diesel fuels, however, if the haze is very dense, a fresh portion of the sample should be dried by shaking 100 mL with 5 g of anhydrous sodium sulfate for at least 5 min and then filtering through dry lintless filter paper. Given sufficient contact time, this procedure will remove or sufficiently reduce the water haze so that the wax cloud can be readily discerned. Drying and filtering should be done always at a temperature at least 14°C above the approximate cloud point but otherwise not in excess of 49°C.

9. Report

9.1 Report the temperature recorded in 8.7 as the cloud point, Test Method D 2500.

10. Precision and Bias

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 2°C only in 1 case in 20.

10.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 4°C only in 1 case in 20.

10.1.3 The precision statements were derived from a 1990 interlaboratory cooperative test program.[4] Participants analyzed 13 sample sets comprised of various distillate fuels and lubricating oils with temperature range from -1 to -37°C. Eight laboratories participated with the manual D 2500/IP219 test method. Information on the type of samples and their average cloud points are in the research report.

10.2 *Bias*—The procedure in this test method has no bias, because the value of cloud point can be defined only in terms of a test method.

10.3 *Precision for Biodiesel Products*[5]—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.3.1 *Repeatability for Blends of Biodiesel in Diesel*—The difference between successive test results obtained by the same operator, using the same apparatus, under constant operating

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1444.

[5] Supporting data (the results of the 2001 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1524.

conditions, on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 2°C only in 1 case in 20.

10.3.2 *Reproducibility for Blends of Biodiesel in Diesel*—The difference between two single and independent test results obtained by different operators, working in different laboratories, on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 3°C only in 1 case in 20.

NOTE 5—The precision for blends of biodiesel in diesel samples comprised cloud points from about −2 to +10°C.

10.3.3 The precision statements were derived from a 2001 interlaboratory cooperative test program.[5] Participants analyzed eleven sample sets comprised of different blends of two petroleum distillate fuels, diesel and kerosene, with various biodiesel fuels with temperature range from +10 to −45°C. Ten laboratories participated with the manual D 2500/IP219 test method. Information on the type of samples and their average cloud points are in the research report.

NOTE 6—One of the outcomes of the interlaboratory study was the selection of the sample types, since the ones used in the study contributed to a difficulty in determining the precision statement. Kerosene is a sufficiently different fuel type from biodiesel to cause some slight separation of phases upon cooling when in B20 blends. Also, the particular kerosene sample used was atypical, which complicated the study further. Therefore, data from the blends of kerosene in biodiesel were not used in the precision statement. In addition, the diesel fuel used in the research report was high cloud point material. Due to the cloud point of the base diesel material, this temperature range in the precision statement was limited.

NOTE 7—A future interlaboratory cloud study will be done to include a wider range of base biodiesel fuels with various distillate blend stocks.

10.4 *Bias for Biodiesel Products*[5]—The procedure in this test method has no bias, because the value of cloud point can be defined only in terms of a test method.

11. Keywords

11.1 cloud point; petroleum products; wax crystals

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 2500–05) that may impact the use of this standard.

(*1*) Revised 6.7.
(*2*) Revised 7.
(*3*) Added Table 1.
(*4*) Deleted original Note 2.

Designation: D 97 – 05

An American National Standard

Designation: 15/95

Standard Test Method for Pour Point of Petroleum Products[1]

This standard is issued under the fixed designation D 97; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method is intended for use on any petroleum product.[2] A procedure suitable for black specimens, cylinder stock, and nondistillate fuel oil is described in 8.8. A procedure for testing the fluidity of a residual fuel oil at a specified temperature is described in Appendix X1.

1.2 Several ASTM test methods offering alternative procedures for determining pour points using automatic apparatus are available. None of them share the same designation number as Test Method D 97. When an automatic instrument is used, the ASTM test method designation number specific to the technique shall be reported with the results. A procedure for testing the pour point of crude oils is described in Test Method D 5853.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 117 Guide for Sampling, Test Methods, and Specifications for Electrical Insulating Oils of Petroleum Origin
D 396 Specification for Fuel Oils
D 1659 Test Method for Maximum Fluidity Temperature of Residual Fuel Oil[4]
D 2500 Test Method for Cloud Point of Petroleum Products
D 3245 Test Method for Pumpability of Industrial Fuel Oils
D 5853 Test Method for Pour Point of Crude Oils
E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standards:*

Specifications for IP Standard Thermometers [5]

3. Terminology

3.1 *Definitions:*

3.1.1 *black oil, n*—lubricant containing asphaltic materials. Black oils are used in heavy-duty equipment applications, such as mining and quarrying, where extra adhesiveness is desired.

3.1.2 *cylinder stock, n*—lubricant for independently lubricated engine cylinders, such as those of steam engines and air compressors. Cylinder stock are also used for lubrication of valves and other elements in the cylinder area.

3.1.3 *pour point, n—in petroleum products*, the lowest temperature at which movement of the test specimen is observed under prescribed conditions of test.

3.1.4 *residual fuel, n*—a liquid fuel containing bottoms remaining from crude distillation or thermal cracking; sometimes referred to as heavy fuel oil.

3.1.4.1 *Discussion*—Residual fuels comprise Grades 4, 5, and 6 fuel oils, as defined in Specification D 396.

4. Summary of Test Method

4.1 After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which movement of the specimen is observed is recorded as the pour point.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved June 1, 2005. Published July 2005. Originally approved in 1927, replacing D 47. Last previous edition approved in 2004 as D 97–04.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP Standard in 1965.

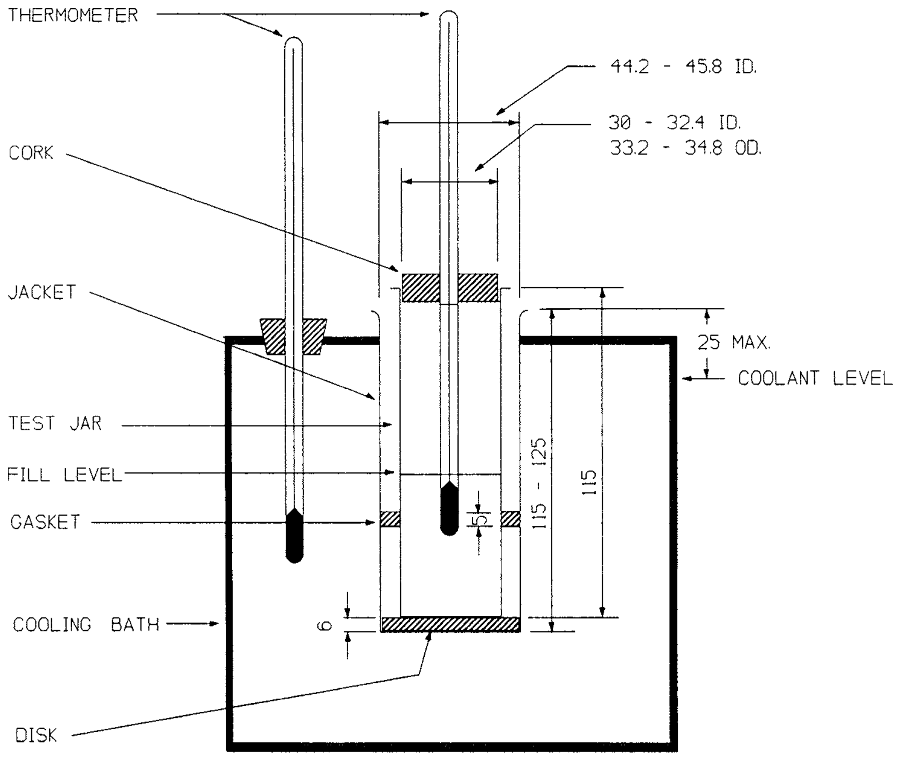
[2] Statements defining this test and its significance when applied to electrical insulating oils of mineral origin will be found in Guide D 117.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[4] Withdrawn.

[5] Methods for Analysis and Testing, *IP Standards for Petroleum and its Products*, Part I, Vol 2.

***A Summary of Changes section appears at the end of this standard.**



NOTE—Dimensions are in millimetres (not to scale).

FIG. 1 Apparatus for Pour Point Test

5. Significance and Use

5.1 The pour point of a petroleum specimen is an index of the lowest temperature of its utility for certain applications.

6. Apparatus

6.1 *Test Jar*, cylindrical, of clear glass, flat bottom, 33.2 to 34.8-mm outside diameter, and 115 to 125 mm in height. The inside diameter of the jar can range from 30.0 to 32.4 mm, within the constraint that the wall thickness be no greater than 1.6 mm. The jar shall have a line to indicate a sample height 54 ± 3 mm above the inside bottom. See Fig. 1.

6.2 *Thermometers*, having the following ranges and conforming to the requirements prescribed in Specification E 1 for thermometers:

Thermometer	Temperature Range	Thermometer Number ASTM	IP
High cloud and pour	−38 to +50°C	5C	1C
Low cloud and pour	−80 to +20°C	6C	2C
Melting point	+32 to +127°C	61C	63C

6.2.1 Since separation of liquid column thermometers occasionally occurs and may escape detection, thermometers should be checked immediately prior to the test and used only if they prove accurate within ±1°C (for example ice point).

6.3 *Cork*, to fit the test jar, bored centrally for the test thermometer.

6.4 *Jacket*, watertight, cylindrical, metal, flat-bottomed, 115 ± 3-mm depth, with inside diameter of 44.2 to 45.8 mm. It shall be supported in a vertical position in the cooling bath (see 6.7) so that not more than 25 mm projects out of the cooling medium, and shall be capable of being cleaned.

6.5 *Disk*, cork or felt, 6 mm thick to fit loosely inside the jacket.

6.6 *Gasket*, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket may be made of rubber, leather, or other material that is elastic enough to cling to the test jar and hard enough to hold its shape. Its purpose is to prevent the test jar from touching the jacket.

6.7 *Bath or Baths*, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be obtained by refrigeration if available, otherwise by suitable freezing mixtures. Freezing mixtures commonly used for temperatures down to those shown are as follows:

	For Temperatures Down
Ice and water	9°C
Crushed ice and sodium chloride crystals	−12°C
Crushed ice and calcium chloride crystals	−27°C
Acetone or petroleum naphtha (see Section 6) chilled in a covered metal beaker with an ice-salt mixture to −12°C then with enough solid carbon dioxide to give the desired temperature.	−57°C

7. Reagents and Materials

7.1 The following solvents of technical grade are appropriate for low-temperature bath media.

7.1.1 *Acetone*, (**Warning**—Extremely flammable).

7.1.2 *Alcohol, Ethanol* (**Warning**—Flammable).

7.1.3 *Alcohol, Methanol* (**Warning**—Flammable. Vapor harmful).

7.1.4 *Petroleum Naphtha*, (**Warning**—Combustible. Vapor harmful).

7.1.5 *Solid Carbon Dioxide*, (**Warning**—Extremely cold −78.5°C).

8. Procedure

8.1 Pour the specimen into the test jar to the level mark. When necessary, heat the specimen in a water bath until it is just sufficiently fluid to pour into the test jar.

NOTE 1—It is known that some materials, when heated to a temperature higher than 45°C during the preceding 24 h, do not yield the same pour point results as when they are kept at room temperature for 24 h prior to testing. Examples of materials which are known to show sensitivity to thermal history are residual fuels, black oils, and cylinder stocks.

8.1.1 Samples of residual fuels, black oils, and cylinder stocks which have been heated to a temperature higher than 45°C during the preceding 24 h, or when the thermal history of these sample types is not known, shall be kept at room temperature for 24 h before testing. Samples which are known by the operator not to be sensitive to thermal history need not be kept at room temperature for 24 h before testing.

8.1.2 Experimental evidence supporting elimination of the 24-h waiting period for some sample types is contained in a research report.[6]

8.2 Close the test jar with the cork carrying the high-pour thermometer (5.2). In the case of pour points above 36°C, use a higher range thermometer such as IP 63C or ASTM 61C. Adjust the position of the cork and thermometer so the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is immersed so the beginning of the capillary is 3 mm below the surface of the specimen.

8.3 For the measurement of pour point, subject the specimen in the test jar to the following preliminary treatment:

8.3.1 *Specimens Having Pour Points Above −33°C*—Heat the specimen without stirring to 9°C above the expected pour point, but to at least 45°C, in a bath maintained at 12°C above the expected pour point, but at least 48°C. Transfer the test jar to a water bath maintained at 24°C and commence observations for pour point.

8.3.2 *Specimens Having Pour Points of −33°C and Below*—Heat the specimen without stirring to 45°C in a bath maintained at 48°C and cool to 15°C in a water bath maintained at 6°C. Remove the high cloud and pour thermometer, and place the low cloud and pour thermometer in position.

8.4 See that the disk, gasket, and the inside of the jacket are clean and dry. Place the disk in the bottom of the jacket. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

8.5 After the specimen has cooled to allow the formation of paraffin wax crystals, take great care not to disturb the mass of specimen nor permit the thermometer to shift in the specimen; any disturbance of the spongy network of wax crystals will lead to low and erroneous results.

8.6 Pour points are expressed in integers that are positive or negative multiples of 3°C. Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the expected pour point (estimated as a multiple of 3°C). At each test thermometer reading that is a multiple of 3°C below the starting temperature remove the test jar from the jacket. To remove condensed moisture that limits visibility wipe the surface with a clean cloth moistened in alcohol (ethanol or methanol). Tilt the jar just enough to ascertain whether there is a movement of the specimen in the test jar. The complete operation of removal, wiping, and replacement shall require not more than 3 s.

8.6.1 If the specimen has not ceased to flow when its temperature has reached 27°C, transfer the test jar to the next lower temperature bath in accordance with the following schedule:

Specimen is at +27°C, move to 0°C bath
Specimen is at +9°C, move to −18°C bath
Specimen is at −6°C, move to −33°C bath
Specimen is at −24°C, move to −51°C bath
Specimen is at −42°C, move to −69°C bath

8.6.2 As soon as the specimen in the jar does not flow when tilted, hold the jar in a horizontal position for 5 s, as noted by an accurate timing device and observe carefully. If the specimen shows any movement, replace the test jar immediately in the jacket and repeat a test for flow at the next temperature, 3°C lower.

8.7 Continue in this manner until a point is reached at which the specimen shows no movement when the test jar is held in a horizontal position for 5 s. Record the observed reading of the test thermometer.

8.8 For black specimen, cylinder stock, and nondistillate fuel specimen, the result obtained by the procedure described in 8.1 through 8.7 is the upper (maximum) pour point. If required, determine the lower (minimum) pour point by heating the sample while stirring, to 105°C, pouring it into the jar, and determining the pour point as described in 8.4 through 8.7.

8.9 Some specifications allow for a pass/fail test or have pour point limits at temperatures not divisible by 3°C. In these cases, it is acceptable practice to conduct the pour point measurement according to the following schedule: Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the specification pour point. Continue observations at 3°C intervals as described in 8.6 and 8.7 until the specification temperature is reached. Report the sample as passing or failing the specification limit.

9. Calculation and Report

9.1 Add 3°C to the temperature recorded in 8.7 and report the result as the Pour Point, ASTM D 97. For black oil, and so forth, add 3°C to the temperature recorded in 8.7 and report the

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1377.

result as Upper Pour Point, ASTM D 97, or Lower Pour Point, ASTM D 97, as required.

10. Precision and Bias

10.1 *Lubricating Oil and Distillate and Residual Fuel Oil.* [7]

10.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 3°C only in one case in twenty. Differences greater than this should be considered suspect.

10.1.2 *Reproducibility*—The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 6°C only in one case in twenty. Differences greater than this should be considered suspect.

10.2 *Bias*—There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

10.3 The precision statements were prepared with data on ten new (unused) mineral oil-based lubricants and sixteen assorted fuel oils tested by twelve cooperators. The mineral oil-based lubricants had pour points ranging from −48 to −6°C while the fuel oils had pour points ranging from −33 to +51°C. The following precision data were obtained:

	Mineral Oil Lubricants	Fuel Oils
95 % Confidence		
Repeatability, °C	2.87	2.52
Reproducibility, °C	6.43	6.59

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[7] The cloud point procedure formerly part of this test method now appears as Test Method D 2500.

APPENDIX

(Nonmandatory Information)

X1. TEST FOR FLUIDITY OF A RESIDUAL FUEL OIL AT A SPECIFIED TEMPERATURE

X1.1 General

X1.1.1 The low-temperature flow properties of a waxy fuel oil depend on handling and storage conditions. Thus, they may not be truly indicated by pour point. The pour point test does not indicate what happens when an oil has a considerable head of pressure behind it, such as when gravitating from a storage tank or being pumped along a pipeline. Failure to flow at the pour point is normally attributed to the separation of wax from the fuel; however, it can also be due to the effect of viscosity in the case of very viscous fuel oils. In addition pour points of residual fuels are influenced by the previous thermal history of the specimens. A loosely knit wax structure built up on cooling of the oil can be normally broken by the application of relatively little pressure.

X1.1.2 The usefulness of the pour point test in relation to residual fuel oils is open to question, and the tendency to regard the pour point as the limiting temperature at which a fuel will flow can be misleading. The problem of accurately specifying the handling behavior of fuel oil is important, and because of the technical limitations of the pour point test, various pumpability tests have been devised to assess the low-temperature flow characteristics of heavy residual fuel oils. Test Method D 3245 is one such method. However, most alternative methods tend to be time-consuming and as such do not find ready acceptance as routine control tests for determining low-temperature flow properties. One method which is relatively quick and easy to perform and has found limited acceptance as a "go-no-go" method is based on the appendix method to the former Test Method D 1659–65. The method is described as follows.

X1.2 Scope

X1.2.1 This method covers the determination of the fluidity of a residual fuel oil at a specified temperature in an as-received condition.

X1.3 Definition

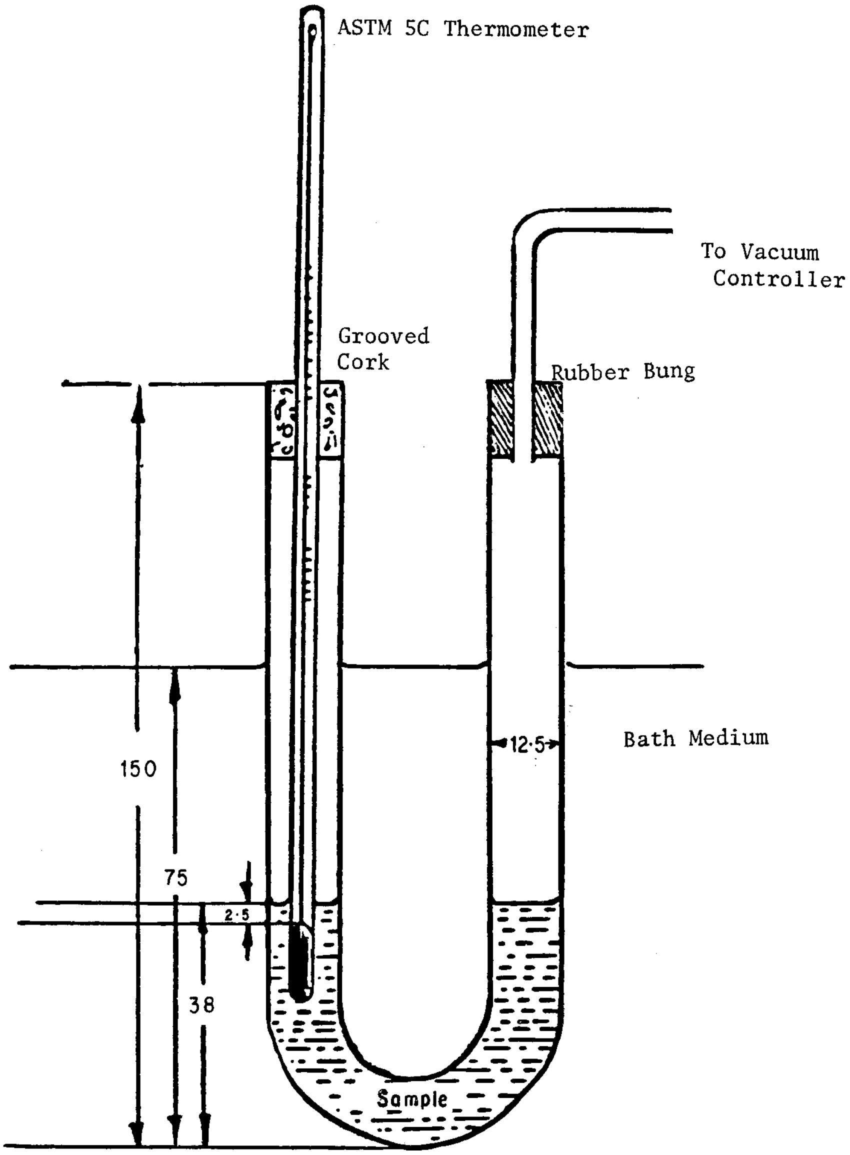
X1.3.1 *fluidity temperature*—the sample when tested in an as-received condition is considered "fluid at the temperature of the test" if it will flow 2 mm in 1 min in a 12.5 mm U-tube under a maximum pressure of 152 mm of mercury.

X1.4 Summary of Test Method

X1.4.1 A sample of fuel in its as-received condition is cooled at the specified temperature for 30 min in the standard U-tube and is tested for movement under prescribed pressure conditions.

X1.5 Significance and Use

X1.5.1 This method may be used as a "go-no-go" procedure for operational situations where it is necessary to ascertain the fluidity of a residual oil under prescribed conditions in an as-received condition. The conditions of this method simulate those of a pumping situation where the oil is expected to flow through a 12-mm pipe under slight pressure at a specified temperature. Fluidity, like Test Method D 97, is used to define cold flow properties. It differs from D 97, however, in that (*1*) it is restricted to residual fuel oil and (*2*) a prescribed pressure is applied to the sample. The latter represents an attempt to overcome the technical limitations of the Pour Point Method where gravity-induced flow is the criterion. Test Method



NOTE—All dimensions are in millimetres

FIG. X1.1 Disposition of U-tube in Fluidity Temperature Test Bath

D 3245, represents another method for predicting field performance in cold flow conditions. Test Method D 3245, however, does have limitations and may not be suitable for use with very waxy fuel oils which solidify so rapidly in the chilling bath that a reading cannot be obtained under the conditions of the test. It is also a time-consuming test and therefore not suitable for routine control testing.

X1.6 Apparatus

X1.6.1 *Glass U-Tubes*, 150 mm high, having a uniform internal diameter of 12.5 ± 1 mm and a radius of curvature, measured to the outside curve of the tube of 35 mm (Fig. X1.1).

X1.6.2 *Thermometers*—Thermometers having a range from −38 to +50°C and conforming to the requirements of Thermometer 5C as prescribed in Specification E 1, shall be used for insertion in the glass U-tubes and for measuring the temperatures of the baths.

X1.6.3 *Fluidity Temperature Test Bath,*[8]

consists of a reservoir, a stirrer, and a motor and pump to circulate coolant through the coils of the tubing placed in the bottom of the test bath and passing through the cold bath. The flow of coolant through these coils can be controlled by a thermostat and a solenoid valve. It is possible that, where justified by the quantity of work, more than one such bath could be utilized to permit concurrent testing at more than one temperature (Fig. X1.2).

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[8] A kinematic viscosity bath is usually satisfactory.

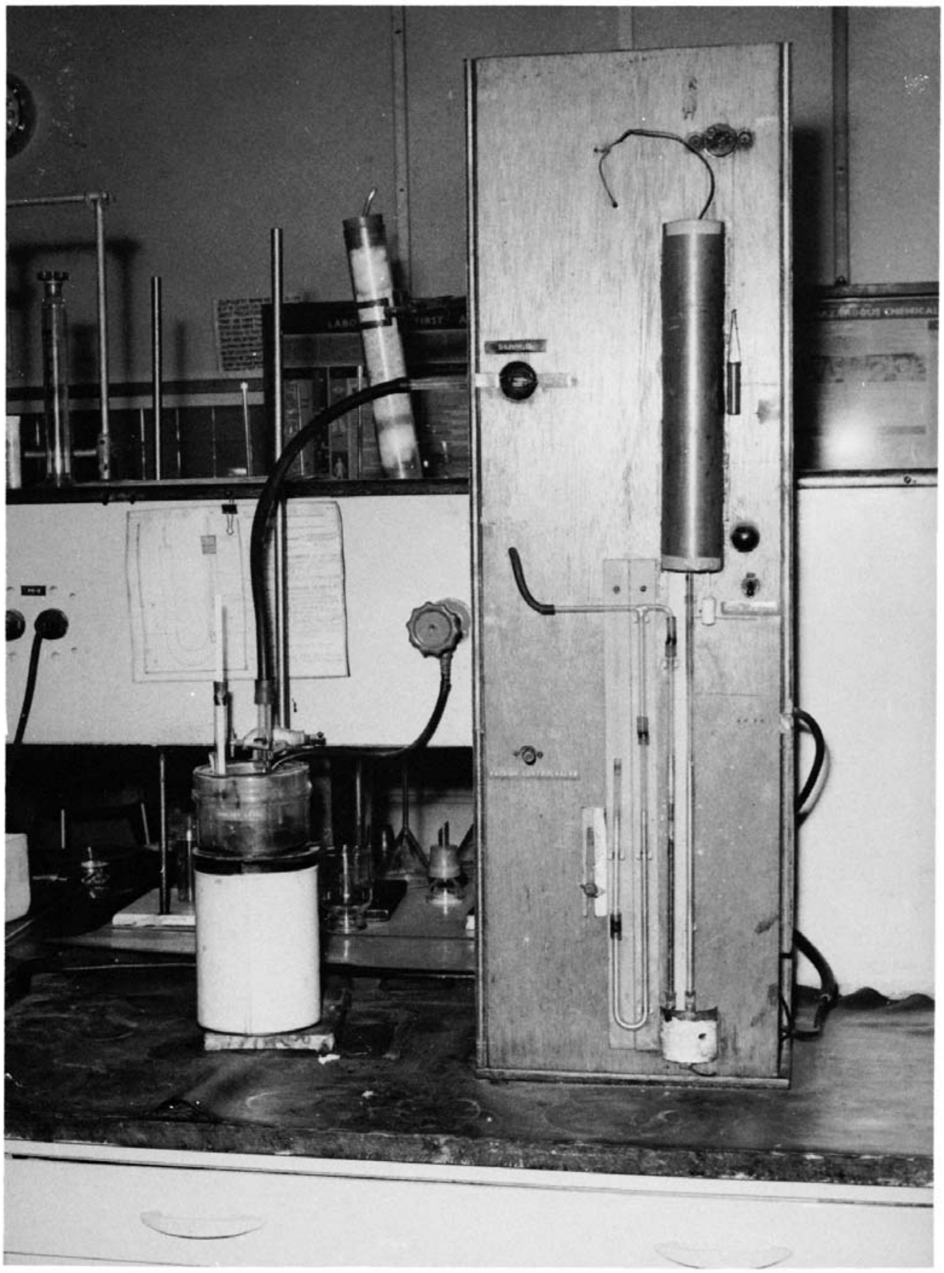

FIG. X1.2 Fluidity Temperature Apparatus

X1.6.4 *Mercury Manometer* calibrated in 10-mm divisions with a distinguishing mark at 152 mm (equivalent to 20.3 kPa).

X1.6.5 *Automatic Vacuum Controller*[9] *(as shown in Fig. X1.3 and Fig. X1.4)*—A device that gradually increased the vacuum applied to one end of the U-tube at the specified rate of 10 mm/4S.

X1.7 Preparation of Apparatus

X1.7.1 Adjust the automatic vacuum controller as follows: close the stopcock on the tube connecting the automatic vacuum controller to the fluidity tester. A pinchcock on the rubber tube will serve as well as a stopcock. Wind the thread attached to the steel rod around the pulley on the synchronous motor until the end of the rod is about 15 mm above the zero level of the mercury in the control manometer. Turn on the power switch. The thread will begin to unwind, lowering the steel rod. When the rod contacts the mercury, the relay will

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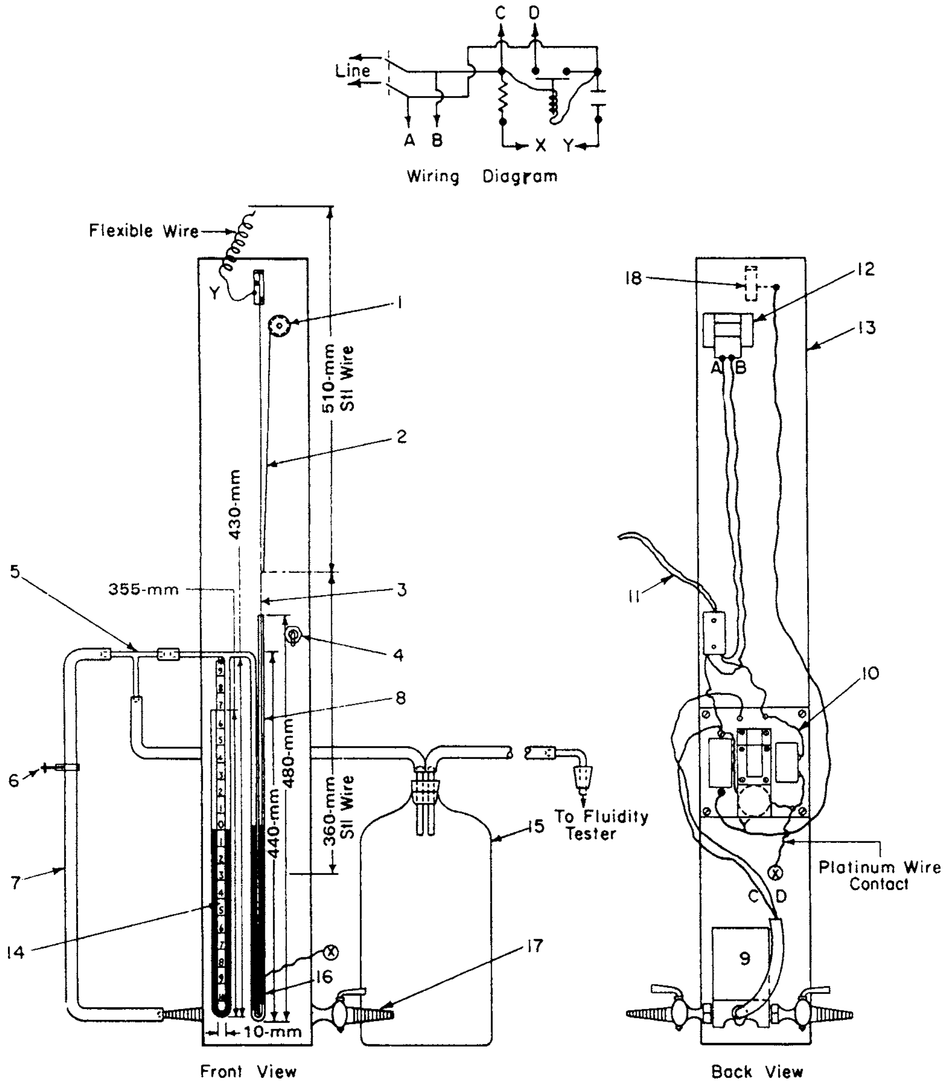
[9] This apparatus may be shop fabricated. Details of special parts are indicated in Figs. X1.3 and X1.4. Alternatively the apparatus can be purchased.



1—26-mm diameter face pulley
2—Thread
3—Steel rod
4—Switch-DPST
5—Tee, 90-mm long
6—Needle valve
7—Rubber or plastic tubing
8—6-mm heat-resistant glass tube
9—Solenoid valve
10—Electric relay
11—Electric cord to outlet
12—Synchronous motor
13—Plywood of approximately 10-mm thickness
14—Millimeter scale
15—4-L bottle
16—0.5-mm heat-resistant glass capillary
17—To vacuum line
18—Rod holder

FIG. X1.3 Assembly Automatic Vacuum Controller Apparatus

open the solenoid valve in the vacuum line and air will be pumped from the system at a rate limited by the needle valve. Adjust this needle valve until the descending mercury in the control manometer just leads the rod, reducing the relay operation to a minimum. When properly adjusted, the pulsations caused by the opening and closing of the solenoid valve should not exceed ±1 mm. In this manner the pressure in the system will be reduced gradually at a rate governed by the descent of the steel rod.

X1.8 Procedure

X1.8.1 Pour the sample as received into a thoroughly cleaned and dry standard fluidity U-tube, without contacting the upper walls of the tube, until the vertical height of the

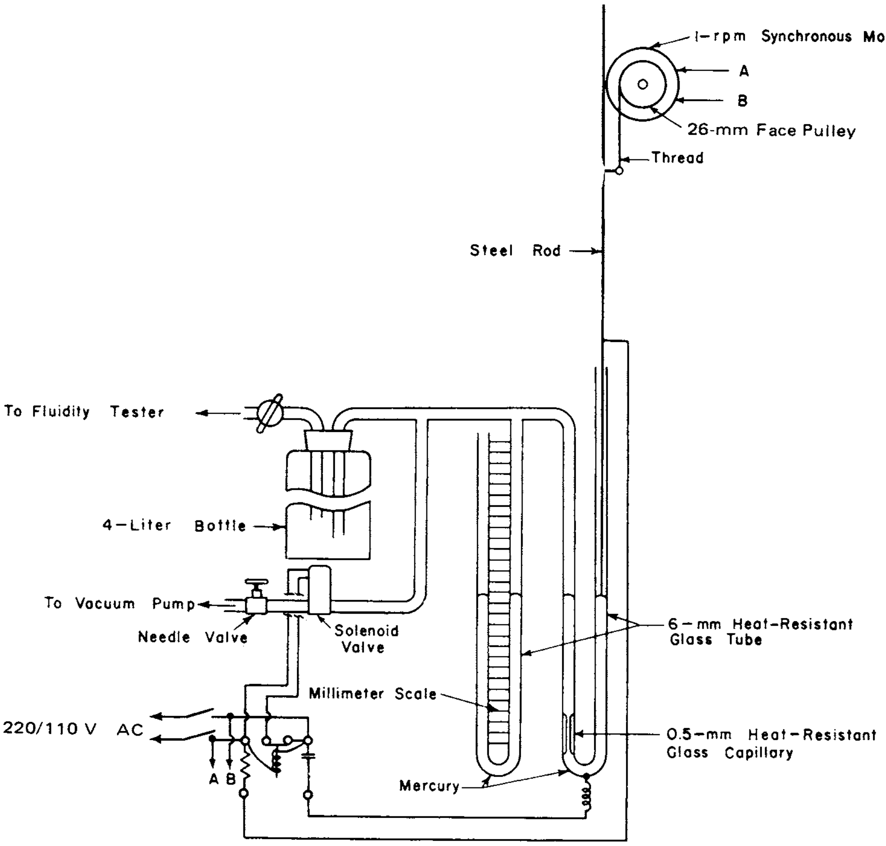


FIG. X1.4 Detail of Automatic Vacuum Controller

sample in the U-tube is 38 mm. Insert in one leg of each U-tube an ASTM Thermometer 5C in a cork that has been grooved to permit the passage of air. The thermometer must be placed in the center of the tube and its bulb immersed so that the beginning of the capillary is 3 mm below the surface of the specimen.

X1.8.2 Fix the tube in the bath set at the specific temperature, immersed to a depth of approximately 75 mm. Control the bath and sample temperatures within ±1°C and ±0.5°C, respectively, of the specified temperature of the test.

X1.8.3 Maintain the sample at the specified temperature for 30 min ± 30 s, with the U-tube connected to the automatic vacuum controller, and the stopcock or pinch-clamp open. Wind the thread on the pulley attached to the synchronous motor. Turn the power switch to the ON position. Apply suction automatically to the U-tube at the prescribed rate. Observe any movement of the specimen during a one-minute interval which is the time required to apply 152-mm Hg vacuum to the specimen in the U-tube. Immediately disconnect the U-tube from the automatic vacuum controller, turn off the power switch and rewind the thread. If the specimen has moved 2 mm or more during the time (1 min) the suction was applied, the specimen is considered fluid at the temperature of the test.

X1.9 Report

X1.9.1 Report the fluidity of the sample at a specified temperature as follows:

X1.9.1.1 If the sample fulfills the conditions of flow, as defined in X1.3.1, report fluidity: "Fluid at (temperature of test)" or fluidity at (temperature of test): "Pass."

X1.9.1.2 If the sample does not fulfill the conditions of flow, as defined in X1.3.1, report fluidity: "Not fluid at (temperature of test)" or fluidity at (temperature of test): "Fail."

X1.10 Precision and Bias

X1.10.1 As in the case of pass-fail data, no statement is made about either the precision or the bias of this method for measuring the fluidity of a residual fuel specimen since the result merely states whether there is conformance to the criteria for success specified in the procedure.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 97–04) that may impact the use of this standard.

(*1*) Added Test Method D 5853 to the Scope and Referenced Documents sections.

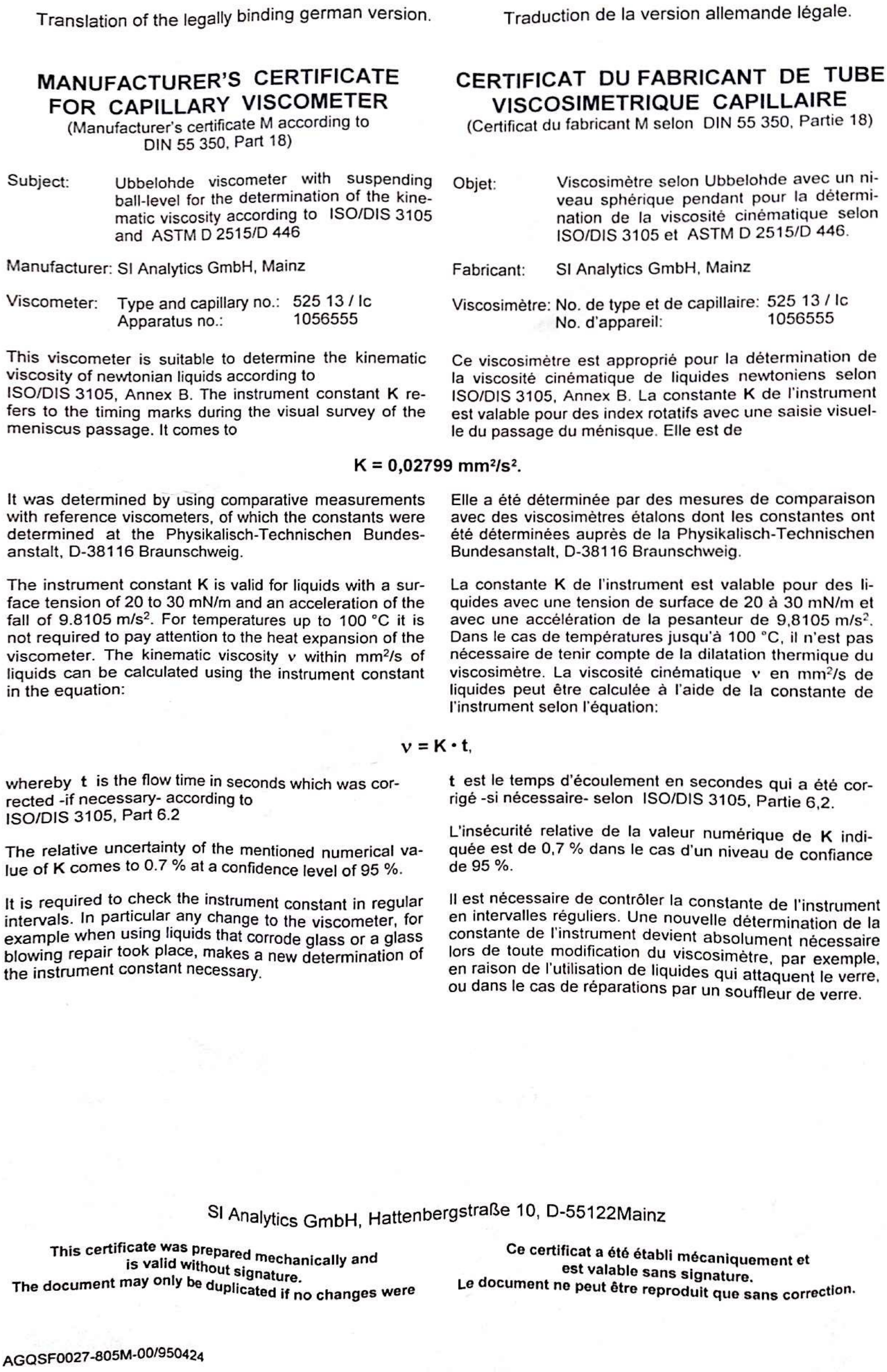
APPENDIX 2.

SOME INTERNATIONAL STANDARDS OF BIODIESEL

Parameters	Austria (ON)	Czech republic (CSN)	France (general official)	Germany (DIN)	Italy (UNI)	USA (ASTM)
Density at 15 ◦C g/cm3	0.85–0.89	0.87–0.89	0.87–0.89	0.875–0.89	0.86–0.90	-
Viscosity at 40 mm2/s	3.5–5.0	3.5–5.0	3.5–5.0	3.5–5.0	3.5–5.0	1.9–6.0
Flash point (◦C)	100	110	100	110	100	130
Pour point (◦C)	-	-	-10		0 to -5	-
Cetane number	≥49	≥48	≥49	≥49		≥47
Conradson carbon residue (%)	0.05	0.05	-	0.05	-	0.05
Iodine number	≤120	-	≤115	≤115		-
Methanol/ethanol (mass%)	≤0.2	-	≤0.1	≤0.3	≤0.2	-
Ester content (mass%)	-	-	≥96.5		≥98	-
Monoglyceride (mass%)	-	-	≤0.8	≤0.8	≤0.8	-
Diglyceride (mass%)	-	-	≤0.2	≤0.4	≤0.2	-
Triglyceride (mass%)	-	-	≤0.2	≤0.4	≤0.1	-
Free glyceride (mass%)	≤0.02	≤0.02	≤0.02	≤0.02	≤0.05	≤0.02
Total glycerol (mass%)	≤0.24	≤0.24	≤0.25	≤0.25	-	≤0.24

APPENDIX 3.

MANUFACTURER'S CERTIFICATE FOR CAPILLARY VISCOMETER



Translation of the legally binding german version.

Traduction de la version allemande légale.

MANUFACTURER'S CERTIFICATE FOR CAPILLARY VISCOMETER
(Manufacturer's certificate M according to DIN 55 350, Part 18)

CERTIFICAT DU FABRICANT DE TUBE VISCOSIMETRIQUE CAPILLAIRE
(Certificat du fabricant M selon DIN 55 350, Partie 18)

Subject: Ubbelohde viscometer with suspending ball-level for the determination of the kinematic viscosity according to ISO/DIS 3105 and ASTM D 2515/D 446

Objet: Viscosimètre selon Ubbelohde avec un niveau sphérique pendant pour la détermination de la viscosité cinématique selon ISO/DIS 3105 et ASTM D 2515/D 446.

Manufacturer: SI Analytics GmbH, Mainz

Fabricant: SI Analytics GmbH, Mainz

Viscometer: Type and capillary no.: 525 13 / Ic
Apparatus no.: 1056555

Viscosimètre: No. de type et de capillaire: 525 13 / Ic
No. d'appareil: 1056555

This viscometer is suitable to determine the kinematic viscosity of newtonian liquids according to ISO/DIS 3105, Annex B. The instrument constant **K** refers to the timing marks during the visual survey of the meniscus passage. It comes to

Ce viscosimètre est approprié pour la détermination de la viscosité cinématique de liquides newtoniens selon ISO/DIS 3105, Annex B. La constante **K** de l'instrument est valable pour des index rotatifs avec une saisie visuelle du passage du ménisque. Elle est de

$$K = 0{,}02799\ \mathrm{mm^2/s^2}.$$

It was determined by using comparative measurements with reference viscometers, of which the constants were determined at the Physikalisch-Technischen Bundesanstalt, D-38116 Braunschweig.

Elle a été déterminée par des mesures de comparaison avec des viscosimètres étalons dont les constantes ont été déterminées auprès de la Physikalisch-Technischen Bundesanstalt, D-38116 Braunschweig.

The instrument constant **K** is valid for liquids with a surface tension of 20 to 30 mN/m and an acceleration of the fall of 9.8105 m/s^2. For temperatures up to 100 °C it is not required to pay attention to the heat expansion of the viscometer. The kinematic viscosity ν within mm^2/s of liquids can be calculated using the instrument constant in the equation:

La constante **K** de l'instrument est valable pour des liquides avec une tension de surface de 20 à 30 mN/m et avec une accélération de la pesanteur de 9,8105 m/s^2. Dans le cas de températures jusqu'à 100 °C, il n'est pas nécessaire de tenir compte de la dilatation thermique du viscosimètre. La viscosité cinématique ν en mm^2/s de liquides peut être calculée à l'aide de la constante de l'instrument selon l'équation:

$$\nu = K \cdot t,$$

whereby **t** is the flow time in seconds which was corrected -if necessary- according to ISO/DIS 3105, Part 6.2

t est le temps d'écoulement en secondes qui a été corrigé -si nécessaire- selon ISO/DIS 3105, Partie 6,2.

The relative uncertainty of the mentioned numerical value of **K** comes to 0.7 % at a confidence level of 95 %.

L'insécurité relative de la valeur numérique de **K** indiquée est de 0,7 % dans le cas d'un niveau de confiance de 95 %.

It is required to check the instrument constant in regular intervals. In particular any change to the viscometer, for example when using liquids that corrode glass or a glass blowing repair took place, makes a new determination of the instrument constant necessary.

Il est nécessaire de contrôler la constante de l'instrument en intervalles réguliers. Une nouvelle détermination de la constante de l'instrument devient absolument nécessaire lors de toute modification du viscosimètre, par exemple, en raison de l'utilisation de liquides qui attaquent le verre, ou dans le cas de réparations par un souffleur de verre.

SI Analytics GmbH, Hattenbergstraße 10, D-55122Mainz

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MANUFACTURER'S CERTIFICATE FOR CAPILLARY VISCOMETER

(Manufacturer's certificate M according to DIN 55 350, Part 18)

CERTIFICAT DU FABRICANT DE TUBE VISCOSIMETRIQUE CAPILLAIRE

(Certificat du fabricant M selon DIN 55 350, Partie 18)

Subject: Ubbelohde viscometer with suspending ball-level for the determination of the kinematic viscosity according to ISO/DIS 3105 and ASTM D 2515/D 446

Objet: Viscosimètre selon Ubbelohde avec un niveau sphérique pendant pour la détermination de la viscosité cinématique selon ISO/DIS 3105 et ASTM D 2515/D 446.

Manufacturer: SI Analytics GmbH, Mainz

Fabricant: SI Analytics GmbH, Mainz

Viscometer: Type and capillary no.: 525 20 / II
Apparatus no.: 1058566

Viscosimètre: No. de type et de capillaire: 525 20 / II
No. d'appareil: 1058566

This viscometer is suitable to determine the kinematic viscosity of newtonian liquids according to ISO/DIS 3105, Annex B. The instrument constant **K** refers to the timing marks during the visual survey of the meniscus passage. It comes to

Ce viscosimètre est approprié pour la détermination de la viscosité cinématique de liquides newtoniens selon ISO/DIS 3105, Annex B. La constante **K** de l'instrument est valable pour des index rotatifs avec une saisie visuelle du passage du ménisque. Elle est de

$$\mathbf{K = 0{,}08947\ mm^2/s^2.}$$

It was determined by using comparative measurements with reference viscometers, of which the constants were determined at the Physikalisch-Technischen Bundesanstalt, D-38116 Braunschweig.

Elle a été déterminée par des mesures de comparaison avec des viscosimètres étalons dont les constantes ont été déterminées auprès de la Physikalisch-Technischen Bundesanstalt, D-38116 Braunschweig.

The instrument constant **K** is valid for liquids with a surface tension of 20 to 30 mN/m and an acceleration of the fall of 9.8105 m/s^2. For temperatures up to 100 °C it is not required to pay attention to the heat expansion of the viscometer. The kinematic viscosity ν within mm^2/s of liquids can be calculated using the instrument constant in the equation:

La constante **K** de l'instrument est valable pour des liquides avec une tension de surface de 20 à 30 mN/m et avec une accélération de la pesanteur de 9,8105 m/s^2. Dans le cas de températures jusqu'à 100 °C, il n'est pas nécessaire de tenir compte de la dilatation thermique du viscosimètre. La viscosité cinématique ν en mm^2/s de liquides peut être calculée à l'aide de la constante de l'instrument selon l'équation:

$$\nu = \mathbf{K} \cdot \mathbf{t},$$

whereby **t** is the flow time in seconds which was corrected -if necessary- according to ISO/DIS 3105, Part 6.2

t est le temps d'écoulement en secondes qui a été corrigé -si nécessaire- selon ISO/DIS 3105, Partie 6,2.

The relative uncertainty of the mentioned numerical value of **K** comes to 0.7 % at a confidence level of 95 %.

L'insécurité relative de la valeur numérique de **K** indiquée est de 0,7 % dans le cas d'un niveau de confiance de 95 %.

It is required to check the instrument constant in regular intervals. In particular any change to the viscometer, for example when using liquids that corrode glass or a glass blowing repair took place, makes a new determination of the instrument constant necessary.

Il est nécessaire de contrôler la constante de l'instrument en intervalles réguliers. Une nouvelle détermination de la constante de l'instrument devient absolument nécessaire lors de toute modification du viscosimètre, par exemple, en raison de l'utilisation de liquides qui attaquent le verre, ou dans le cas de réparations par un souffleur de verre.

SI Analytics GmbH, Hattenbergstraße 10, D-55122Mainz

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APPENDIX 4.

EXPERIMENTAL DATA FOR KINEMATIC VISCOSITY AND CLOUD POINT & POUR POINT

Kinematic Viscosity

100% Used Canola Oil

Temp. ^{0}C	Times	Sec.	Ave. Time (sec.)	Capillary Tube Constant (K) (mm^2/sec^2)	Kinetic Energy Correction (y) (sec.)	Kinematic Viscosity (mm^2/sec)
20	Time 1	262.1	262.68	0.02799	0.02	7.35171345
	Time 2	262.3				
	Time 3	263.1				
	Time 4	263.2				
15	Time 1	308.3	307.70	0.02799	0.01	8.61224310
	Time 2	308.1				
	Time 3	307.2				
	Time 4	307.2				
10	Time 1	347.2	349.73	0.02799	0.01	9.78852285
	Time 2	346.3				
	Time 3	352.4				
	Time 4	353.0				
5	Time 1	424.2	422.83	0.02799	0.01	11.83459185
	Time 2	423.1				
	Time 3	421.7				
	Time 4	422.3				
0	Time 1	502.3	501.28	0.02799	0.01	14.03040735
	Time 2	501.2				
	Time 3	501.2				
	Time 4	500.4				
-3	Time 1	556.4	557.00	0.02799	0.01	15.59015010
	Time 2	557.0				
	Time 3	557.4				
	Time 4	557.2				
-5	Time 1	654.1	653.20	0.02799	0.01	18.28278810
	Time 2	651.4				
	Time 3	654.1				
	Time 4	653.2				
-7	Time 1	240.0	240.50	0.08947	0.01	21.51664030
	Time 2	241.0				
	Time 3	241.0				
	Time 4	240.0				
-8	Time 1	266.6	266.73	0.08947	0.01	23.86299105
	Time 2	265.3				
	Time 3	267.0				
	Time 4	268.0				
-9	Time 1	0.0	0	0	0	Not Determined
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-10	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				

100% Used Frying Oil						
Temp. ^{0}C	Times	Sec.	Ave. Time (sec.)	Capillary Tube Constant (K) (mm^2/sec^2)	Kinetic Energy Correction (y) (sec.)	Kinematic Viscosity (mm^2/sec)
20	Time 1	266.1	264.95	0.02799	0.02	7.41539070
	Time 2	263.5				
	Time 3	264.2				
	Time 4	266.0				
15	Time 1	311.3	311.25	0.02799	0.01	8.71160760
	Time 2	310.4				
	Time 3	312.0				
	Time 4	311.3				
14	Time 1	0.0	0	0	0	not determined
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
10	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
5	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
0	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-5	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-10	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				

50% Used Canola Oil & 50% Used Frying Oil

Temp. ^{0}C	Times	Sec.	Ave. Time (sec.)	Capillary Tube Constant (K) (mm^2/sec^2)	Kinetic Energy Correction (y) (sec.)	Kinematic Viscosity (mm^2/sec)
20	Time 1	343.2	344.33	0.02799	0.01	9.63737685
	Time 2	345.0				
	Time 3	344.1				
	Time 4	345.0				
15	Time 1	409.7	409.90	0.02799	0.01	11.47282110
	Time 2	408.9				
	Time 3	411.0				
	Time 4	410.0				
10	Time 1	479.0	479.50	0.02799	0.01	13.42092510
	Time 2	480.0				
	Time 3	479.0				
	Time 4	480.0				
8	Time 1	512.4	512.75	0.02799	0.01	14.35159260
	Time 2	514.1				
	Time 3	512.2				
	Time 4	512.3				
7	Time 1	0.0	0	0	0	not determined
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
5	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
0	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-5	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-10	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				

75% Used Canola Oil & 25% Used Frying Oil

Temp. ^{0}C	Times	Sec.	Ave. Time (sec.)	Capillary Tube Constant (K) (mm^2/sec^2)	Kinetic Energy Correction (y) (sec.)	Kinematic Viscosity (mm^2/sec)
20	Time 1	373.1	372.75	0.02799	0.01	10.43299260
	Time 2	372.2				
	Time 3	374.1				
	Time 4	371.6				
15	Time 1	441.4	440.00	0.02799	0.01	12.31532010
	Time 2	440.3				
	Time 3	439.5				
	Time 4	438.8				
10	Time 1	517.5	518.75	0.02799	0.01	14.51953260
	Time 2	519.3				
	Time 3	519.1				
	Time 4	519.1				
5	Time 1	628.2	627.75	0.02799	0.01	17.57044260
	Time 2	627.5				
	Time 3	625.9				
	Time 4	629.4				
2	Time 1	225.6	226.73	0.08947	0.01	20.28419105
	Time 2	226.1				
	Time 3	227.2				
	Time 4	228.0				
1	Time 1	0.0	0	0	0	not determined
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
0	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-5	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-10	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				

25% Used Canola Oil & 75% Used Frying Oil

Temp. ^{0}C	Times	Sec.	Ave. Time (sec.)	Capillary Tube Constant (K) (mm^2/sec^2)	Kinetic Energy Correction (y) (sec.)	Kinematic Viscosity (mm^2/sec)
20	Time 1	345.0	344.33	0.02799	0.01	9.63737685
	Time 2	343.2				
	Time 3	344.1				
	Time 4	345.0				
15	Time 1	408.3	408.25	0.02799	0.01	11.42663760
	Time 2	410.0				
	Time 3	407.4				
	Time 4	407.3				
10	Time 1	479.0	479.75	0.02799	0.01	13.42792260
	Time 2	480.0				
	Time 3	479.0				
	Time 4	481.0				
9	Time 1	0.0	0	0	0	not determined
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
5	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
0	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-5	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				
-10	Time 1	0.0	0	0	0	0
	Time 2	0.0				
	Time 3	0.0				
	Time 4	0.0				

Cloud Point and Pour Point

Samples	% UCO	Cloud Point (CP)	Ave. Cloud Point	Pour Point (PP)	Ave. Pour Point
100% UFOME	0	18.0	17.8	11.5	11.3
		17.5		11.5	
		18.0		11.0	
75% UFOME + 25% UCOME	25	14.5	14	8.0	7.7
		13.5		7.5	
		14.0		7.5	
50% UFOME + 50% UCOME	50	11.5	11.5	3.5	3.5
		11.5		3.5	
		11.5		3.5	
25% UFOME + 75% UCOME	75	5.5	5.7	-1.0	-1
		5.5		-1.0	
		6.0		-1.0	
100% UCOME	100	-1.0	-1	-10.5	-10.5
		-1.0		-10.5	
		-1.0		-10.5	