DETERMINATION AND PREDICTION OF KINEMATIC VISCOSITY OF

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A THESIS SUBMITTED TO THE GRADUATE SCHOOL OF APPLIED SCIENCES OF NEAR EAST UNIVERSITY

By ORIEKE OLUCHI KALU

In Partial Fulfillment of the Requirements for the Degree of Master of Science in Mechanical Engineering

NICOSIA, 2017

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Oluchi Orieke Kalu: Determination and Prediction of Kinematic Viscosity of Biodiesel Blends.

Approval of Director of Graduate School of Applied Sciences

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To my parents

ABSTRACT

Biodiesel, can be defined as a mono-alkyl esters of vegetable oils and animals fats, which is seen as an alternative source of energy to fuels in diesel engine. In current times the use of biodiesel has become a well-known fuel which are used throughout the entire universe. Biodiesel is seen as an alternative fuel for diesel engines but encounters certain challenging problems, one problem which is most significant is the degree of unsaturation of the fatty acid compounds since they are sometimes derived from vegetable oils and fats. However, the major important property of biodiesel and conventional diesel fuel obtained from petroleum is the viscosity which is important in lubricant and can have an effect on the temperature, pressure, composition, chain length and the degree of saturation. The biodiesel property are known to have greater values of kinetic viscosity than those of euro diesel summer, euro diesel winter, kerosene, diesel fuel and most especially benzene which are basically insignificant. The biodiesel properties was measured at temperature ranging from 20°C to 140°C is presented. An absolute approach was been made using the response to surface methodology (RSM) equation which was used to predict the properties of certain blends of biodiesel with kerosene disel fuel benzene and euro diesel and the calculations in referance to the correlation of the experimental data.

Keywords: Biodiesel; Diesel Fuel; Kerosene; Euro Diesel; Kinematic Viscosity; RSM

ÖZET

Biyodizel, dizel motorlarda yakıtlara alternatif enerji kaynağı olarak görülen bitkisel yağların ve hayvan yağlarının mono-alkil esterleri olarak tanımlanabilir. Günümüzde, biyodizel kullanımı tüm evren boyunca kullanılan iyi bilinen bir yakıt haline gelmiştir.Biyodizel, dizel motorlar için alternatif bir yakıt olarak görülmekle birlikte bazı zorlu sorunlarla karşılaşmaktadır, en önemli olan bir sorun, bazen bitkisel yağlardan ve yağlardan türetildiği için yağlı asit bileşiklerinin doymamışlığının derecesidür.Bununla birlikte, biyodizelin ve petrolden elde edilen konvansiyonel dizel yakıtın en önemli özelliği, yağlayıcıda önemli olan ve sıcaklık, basınç, kompozisyon, zincir uzunluğu ve doygunluğun derecesi üzerinde etkili olabilen viskozitedir.Biyodizel mülkünün, euro dizel yaz, euro dizel kış, gazyağı, dizel yakıtı ve özellikle de benzen'den daha büyük kinetik viskozite değerlerine sahip olduğu biliniyor ki bunlar esas olarak önemsizdir. Biyodizel özellikleri, 20°C ila 140°C arasında değişen sıcaklıklarda ölçüldü.Kerosen disel yakıt benzen ve euro dizel ile bazı biyodizel karışımlarının özelliklerini ve deney verilerinin korelasyonuna referansla hesaplanan hesaplamalarda kullanılan yüzey metodolojisine (RSM) denklemi kullanılarak mutlak bir yaklaşım yapılmıştır.

Anahtar Kelimeler: Biyodizel; Dizel yakıt; gazyağı; euro dizel; Kinematik viskozite; RSM

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LIST OF SYMBOLS USED

LIST OF QUANTITIES

- U Kinematic Viscosity
- Uz Viscosity in flow direction
- Ur Viscosity in radian direction
- Ue Viscosity in radian direction
- P Flow pressure
- μ Dynamics viscosity
- g Acceleration due to gravity
- **U** Kinematic Viscosity
- Q Volume flow rate
- ε Strain rate
- σ Shear stress on the fluid element
- τ Alternative form of shear stress
- t shear time
- A Area
- r Radian length
- V Volume
- z Length in flow direction
- D Capillary diameter
- dv Changing in velocity
- dx Changing in separation height
- F Force
- L Length of viscometer

- G Universal gas constant
- H Capillary height

LIST OF ABBREVIATIONS

WFCC: World Fuel Charter Committee

ASTM: American Society for Testing and Materials

GHG: Greenhouse Gasses

CGS: Centimeter-gram-second

DIN: German Institute for Standardization

EN: European Standard

FAME: Fatty Acid Methyl Ester

ISO: International Standard Organization

KE: Kinematic Energy

BD: Biodiesel

DF: Diesel Fuel

B: Benzene

K: Kerosene

ED: Euro diesel

WFCC: World Fuel Charter Committee

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BD:

Biodiesel

DF:

Diesel Fuel

B:

Benzene

K:

Kerosene

EDS:

Euro diesel summer

EDW:

Euro diesel winter

WFCC:

World Fuel Charter Committee

CHAPTER 1 INTRODUCTION

1.1 General View

Due to the high increase in industrial development and motorization of the world complications to the high increase in demand of petroleum-based fuel (Srivastava, and Prasad, 2000). An increase in the consumption of conservative fuels in addition to the ecological concerns have brought about the crucial need for substitute in fuels. Understanding the facts that that oil reservoir where learned during the current ages, because the non-renewable fuel may not meet up with the world energy requirement during an elongated period of time. Petroleum fuels are now been replaced with fuels from renewable source such as vegetable oils, canola oil, etc. Insight of its comparable characteristics with fossil fuels (Ramadhas et al., 2004).

The production of biodiesel is through a process known as transesterification, biodiesel is obtained through a mixture of alkyl esters derived from vegetable oils, animal fats or greases through a transesterification reaction, factors put into consideration in other to avoid certain mischief, these factors includes the transestification of vegetable oil, The biodiesel products are been considered a relative substitute to fuels in the diesel engines, certain property such as kinematic viscosity, has a greater value than 10% of biodiesel which are presently used as fuels for diesel engines. In current times the use of biodiesel has become a well-known fuel which are used throughout the entire universe. Biodiesel is seen as an alternative fuel for diesel engines but encounters certain challenging problems, one problem which is most significant is the degree of unsaturation of the fatty acid compounds since they are sometimes derived from vegetable oils and fats. Biodiesel has a very unique feature and qualities and are known for they environmental friendliness. But one basic benefits is that it blends easily with other forms of energy resources and oils, which is safe in diesel engine. Due to its lubricating property, biodiesel aids to increase the chances of the lifetime of the diesel engine one basic significant disadvantage of biodiesel is that it is relatively expensive more than those of diesel fuel. Advantages of biodiesel is that is eco-friendly, conservational quality, safe energy, reduced growth and social amenities related to the use of petroleum fuels, particularly based on oil-importing countries unlike diesel fuel with limitations of biodiesel is that it has an has an effect on the rubber hose in certain engines biodegradable which are necessary in the

reduction of compounds of CO₂, SO₂, some volatile organic compounds, unburned hydrocarbon and particulate maters of emissions.

During the production of biodiesel, basic raw materials such as waste cooking oil, animal oils, and waste grease along with methanol are used and the by-product is known as glycerin. Biodiesel consists certain fatty acid of methyl ester (FAME) chains. In general, biodiesel possess certain comparable properties with diesel such properties which includes specific gravity and heat combustion (Naik et al., February 2010). However, biodiesel having a high viscosity can have an influence on the performance of the engine, therefore, viscosity of fuel play and significant role (Ferreira, 2009), the viscosity of fuel is an important transport properties that can affect the injection system, mostly in temperature that are relatively low when the viscosity is said to increase. The biodiesel content with diesel fuel shows related viscosity temperature dependence. The blend with diesel fuel having 5-10% of biodiesel are presently in use and are used as fuel for diesel engines.

The viscosity of a fuel is one of the most significant properties which influences the performance of fuel injection system. The Fatty Acid Methyl Ester (FAME) are been consider as possible replacement or blend for convectional diesel fuel and are produce according to the required standards. Generally the FAME has enhanced the lubricating properties; on the other hand, the higher the viscosity level which tends to form a large droplet on the injection which may cause a poor combustion increasing the exhaust smoke under a certain operating condition. ASTM D 975 requires kinematic viscosity which varies from 1.9 minimum up until 4.1 mm²/s at 40°C and World Fuel Charter Committee (WFCC) requires 2.0 - 5.0 mm²/s (Washington, 2009). Certain major facts have been stated that biodiesel with benzene and kerosene blends are not in the recommendation as those of fuels for the transportation purpose due to its high level of toxicity of aromatic compounds although some data values are obtained which are intended for the use in the assessment purpose for an improved understanding of the excellence in the viscosity of data correlation of biodiesel with benzene and biodiesel with kerosene blends are of better understanding regarding its s composition and component structure, compared to those of biodiesel with diesel fuel blends and biodiesel with euro diesel blends; these blends has no polar molecules and are said to be miscible all through the composition with biodiesel.

Understanding the essential properties of biodiesel with diesel fuel blends and biodiesel with euro diesel blends of different degrees at various temperature range using diesel fuel obtained from K.Th.G. Eco-logical Fuels Co. Ltd in Cyprus, Greek which has been investigated. A lot of studies referring to the blend of biodiesel with diesel fuel (Nita et al., 2011; Moser, 2014)

not all studies specifies the type of biodiesel, there chemical composition and average molecular weight used during experimental procedures, some facts have been achieved in this study; blends of biodiesel with benzene and kerosene have also been studied, blends of biodiesel with benzene was less studied (Vural et al., 2008).

1.2 Thesis Aim

The goal of this work is to determine experimentally the proprieties of five different forms of fuel blend samples of biodiesel with diesel fuel, biodiesel with benzene, biodiesel with euro diesel summer, biodiesel with euro diesel winter, and biodiesel with kerosene from Cyprus with their temperature relationships from 20°C up to 140°C. Additionally, to show the effect of temperature and volume of fraction of biodiesel on viscosity, and creating an empirical solution of each binary fuel blend. Mixing composition percentage will also be given.

1.3 Thesis Overview

The rest of this thesis is divided into 5 chapters, which are structured as follows.

Chapter 1 is an introduction about the thesis. In this chapter, a definition of the thesis is presented; we set the aims, the general overview of the thesis is discussed.

Chapter 2 introduces the literature review of biodiesel in several aspects presented. The properties of biodiesel and the theory of the capillary viscometer.

Chapter 3 is a detailed and general explanation on biodiesel property and samples used, Such as kinematic viscosity. Also they measurement procedure and experimental setup.

Chapter 4 is a detailed explanation on the results obtain from the blends of biodiesel with kerosene, diesel fuel, kerosene and euro diesel summer, euro diesel winter, and the accuracy of two different equations recommended to correlate and predict these properties was also evaluated.

Chapter 5 is a conclusion and recommendation on the behaviour of the biodiesel property with fuel blends.

CHAPTER 2 RELATED RESEARCH

2.1 Literature Review

The entire biosphere encounters the crises of reduction in fossil fuels in regards to the problem of ecological degradation. A quick reduction in fossil fuel reserves with a rise in the request and doubt in supply of fossil fuel, where also a quick increase of the petroleum values, which has stirred up a great research for the search of other alternatives to fossil fuels. An respect to these search an urgent need to discover new alternatives, which are more are likely to reduce our reliance on the importation of oil which would help to provide protection on the environment for a sustainable development. Several substitute to fuel are now being in the existence recently discovered as a possible replacements for a current raise in pollutant for diesel fuel are obtained from a marketable resource. The biodiesel products are referred to as a more energy efficient ecologically welcoming choice in current period in other to full fill a great energy need required for the future. Biodiesel is seen as a renewable diesel substitute which is derived from conjoining together chemically, to any natural oils and fats with alcohols. In the past 15 years, the biodiesel which has a progressively in the study phase has greater scale in the production in several industrialized nations. In areas like the India context, oils which are non-edible are used as a preferred feedstock and in several field trails have also been made for the production of biodiesel (Giunta, 1996; Campen, 1990). There are some papers in the literatures on the properties of biodiesel blended with petro diesel or with another kind of biodiesel at low temperatures. The report stated by the kinematic viscosity is an utmost significant importance in the transport properties of fuel, which has influenced particularly in the injection system mostly at a very low temperature when the viscosity increases. We have two methods in the predictions of biodiesel with diesel fuel and biodiesel blends of viscosity which are accessible in these works. Blends of 5 - 10% of biodiesel with diesel fuel are presently commercialised in the fuel for diesel engines.

The molecular similarities of biodiesel to diesel fuel compounds has an alternative fuel in satisfying the requirements of diesel engine. Engine fuel for biodiesel which produces a lesser contaminating kind of specie that lacks any essential need for emission control equipment. Characteristics of biodiesel are of close to that of diesel fuel which are thus to become strong alternative on the replacements in replacements on diesel fuel. In the conservation of

triglycerides into methyl or ethyl in transestification procedure causes a reduction of the molecular weight to at least a one-third of that trilycerid which drops the viscosity by the factor of about eight which will increase the explosive nature marginally. The biodiesel has a viscosity close by to that of a diesel fuel. The esters comprise of 10 to 11% oxygen by weight, these might help to hearten the additional ignition than those of the hydrocarbon which are centred on diesel fuel engines. The biodiesel has a lesser volumetric heating values about 12% than that of diesel fuel which abstains a higher centane number and flash point.

Equally in the continuance numerous study, one the essential properties of biodiesel blends (Nita et al., 2011), our aim in these project work is to present new experimental statistics on the efficient methods in other to assess the exactness of some certain equation for viscosity in the correlation of data or prediction from unadulterated component assets. The purpose of the equation for biodiesel blends, response to surface methodology equations, Grunberg–Nissan eqaution or Wilke equation in the petroleum domain and McAllister equation in the conventional thermodynamics in the molecular mixtures may be used.

Subsequently the information, facts and properties between biodiesel with the blends of diesel fuel at various degree at several temperatures which have been investigated biodiesel. There are so many studies on biodiesel with diesel blends (Nita et al., 2011) preferably not all specifies the type of the biodiesel kind used, the chemical component and the mediocre of the molecular weight, and finally facts are been achieved in these study. Nevertheless the attempts to carry out the process, it is unavoidable to repeat the mistakes that might have been overlooked one way or another.

2.2 Bbiodiesel Properties

2.2.1 Kinematic Viscosity

Kinematic viscosity is a unique important characteristics towards the possessions of all liquids. As soon as the liquid flows, which has an internal resistance to the flow which mainly depends on the interaction forces of molecules. The kinematic viscosity also can be labelled as drag force in addition remains measured by fractional assets of the liquid, it also measures resistance to flow or shear (Tushar et al., 2007). Kinematic viscosity measures surrender to a fluid which either can be deformed either through the shear stress or tensile stress. Nowadays terms (for fluids only), viscosity is said to "thickness" or "internal friction". The fluid flow plays a very important part in the processing of materials.

Three major combination factors are govern the kinematic viscosity, such as:

- a. Intermolecular force: When the bonds of the molecules are stronger, the more viscous the fluid becomes.
- b. Molecular size: when the molecules becomes smaller the flow past on one another are easier than the larger molecules.
- c. Molecular shape: these properties are quite complicated. Occasionally, the linear molecular flow past one another than those of the branched molecules. Additional, in most cases linear molecules are easier to get stocked at the upper one than those branched molecules, the intermolecular bonding increases amid between the linear molecules.

2.2.2 Viscosity Coefficients

They are two way by which the Viscosity coefficients that are conveyed such as:

- Dynamic viscosity, or an absolute viscosity, which is the more usual one (typical units Pa·s, Poise, P);
- Kinematic viscosity is a dynamic viscosity which stands to be divided by the density (typical unit's m²/s, Stokes, St).
- I. Dynamic viscosity: The SI physical unit of dynamic viscosity remains the Pascalsecond (Pa·s), equivalent to N·s/m², or kg/ (m·s). It is the tangential force per unit area required to slide one layer (A) against another layer (B) as shown in Figure 2.1. for an example, If a fluid with a viscosity of one Pa·s is placed between two plates, and one plate is pushed sideways with a shear stress of one Pascal, it moves a distance equal to the thickness of the layer between

Due to the viscosity of the fluid is clear in the measure on exactly by what means resistance to the fluid flow, mathematically, it is labelled as:

Shear stress = μ (shear rate or strain)

Where µ is known as the dynamic viscosity

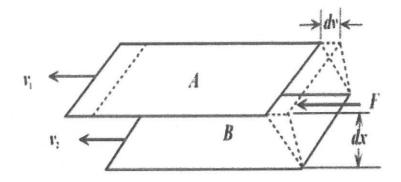


Figure 2.1: Shear of the fluid film

Where, (σ) shear stress and (ε) is strain rate, therefore the expression becomes:

$$\sigma = \mu \varepsilon$$
 (2.1)

The strain rate is generally expressed as

$$\varepsilon = \frac{1}{r} \frac{dx}{dt} = \frac{v}{r} \tag{2.2}$$

Where,

x = length

t = time

dx/dt = velocity(v),

Therfore the dynamic viscosity can be written as;

$$\mu\sigma\frac{x}{v} \tag{2.3}$$

Also by for a Newtonian fluid, Relationship between shear stress and the deformation is linearly proportional with proportionality constant as μ , where σ could also be replace by τ .

$$\sigma \alpha \left(\frac{dv}{dx}\right)^n = \mu \frac{dv}{dx} \qquad , if \ n = 1$$
 (2.4)

The centipoise (cP) is utmost suitable unite to the report the absolute or dynamic viscosity of liquids. It is 1/1000 of Poise. "Poise is the short form of Poiseuille named after a French physician, Jean Louis Poiseuille (1799-1869)". Other units are:

- SI system: Ns/m^2 , Pa.s or kg/m.s where N is Newton and Pa is Pascal, and 1Pa.s = 1 $N.s/m^2 = 1$ kg/m.s
- Metric system: CGS (centimeter-gram-second) as g/cm.s, dyne.s/cm² or poise (P)
 where, 1 poise = dyne.s/cm² = g/cm.s = 1/10 Pa.s

- British unit system: lb/ft.s or lbf.s/ft².
- II. Kinematic viscosity: Several situations, concerned with ratio of internal force to viscous force (i.e. the Reynolds number, $Re = \frac{VD}{v}$), which are characterized by the fluid density ρ . By understanding the concept density at required temperature and pressure, kinematic viscosity defined as follows:

$$v = \frac{\mu}{\rho} \tag{2.5}$$

Where ρ is density of the fluid.

For kinematic viscosity the SI unit is m²/s and density in kg/m³,

The physicial properties of knematic viscosity is refferred to as the stokes (St), which was named after a man by Geoorge Gabrial Stokes.which are at times expressed in cntiStrokes (cSt). In the U.S. the stokes are used as a singular form (Tushar et al., 2007).

2.2.3 Factors Affecting Viscosity

Generally, there are few factors which affects the viscosity in the Newtonian fluid such as temperature, pressure and in the case which involves the solution and mixture, by composition. Additionally, biodiesel as a Newtonian fuel is affected by some factors such as the chain length and degree of saturation. Viscosity is inversely proportional to temperature. Viscosity increases as temperature decreases also, viscosity decreases as the temperature increases, and finially the chain length (number of carbon atoms) in viscosity increases with and with increasing degree of saturation.

A viscometer which also can be called viscometer, it is a device used in the measurement of viscosity of a fluid or viscosity of Newtonian fluids. Viscometers can be largely categorized into seven classes as stated below;

- High temperature, high shear rate viscometer
- Orifices viscometer
- Ultrasonic viscometer
- Falling ball viscometer
- Capillary viscometer
- Vibrational viscometer
- Rotational viscometer

The commonly used instrument is the glass capillary for the measurement of kinematic viscosity.

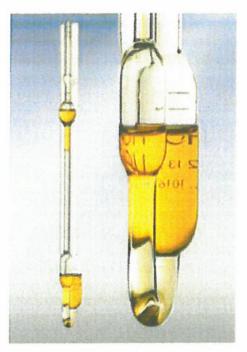


Figure 2.2: Capillary viscometer

2.2.4 Capillary Viscometer

The capillary viscometer is compressed of cylinder capillary tube of a minor diameter. In the capillary viscometer, liquid is enforced into the capillary tube through the process of imposing a pressure drop.

In the inner diameter of a capillary viscometer tube, during the viscosity measurement the velocity drop required to build up the form the laminar tube flow within measurement of the capillary under an idealized circumstances such as;

- Wall to adhere of the liquid
- Newtonian flow behaviour of liquid
- The stationary flow condition
- The laminar, isothermal flow condition
- The pressure independence of viscosity

• Incompressibility of the liquid

Negligence in the flow have an impacts on access and exist of the capillary for a satisfactory length, Figure 2.3 shows liquid flow in coaxial layer which is towards the pressure drop in the capillary. The flow occurs in a parabolic velocity.

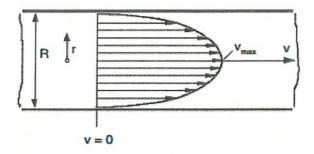


Figure 2.3: Velocity profile with laminar tube flow

There are various types of capillary viscometers which are been. The most suitable type is the glass capillary viscometer for the measurement of viscosity in Newtonian liquids. Generally the kinematic viscosity is used in measuring viscometer.

2.2.5 Theory of Capillary Viscometer

The capillary viscometer is used in the measurement calculation of viscosity which is based on Poiseuille's equation of a Newtonian fluid (Tushar et al., 2007). Figure 2.4 shows a developed laminar flow through a straight vertical tube of circular cross section.

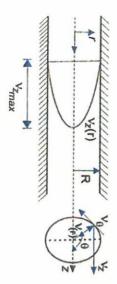


Figure 2.4: Hagen -poiseuille flow

If z-axis is taken as the axis of the tube along which all the fluid particle travels and considering rotational symmetry to make the flow two-dimensional axisymmetry, then,

$$v_z \neq 0, v_r = 0, v_\theta = 0$$
 (2.6)

From continuity equation,

$$\frac{\partial v_r}{\partial r} + \frac{v_r}{r} + \frac{\partial v_z}{\partial z} = 0 \tag{2.7}$$

For rotational symmetry,

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

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

By inserting,

$$v_{\rm r} = 0$$
, $v_{\theta} = 0$, $\frac{\partial v_z}{\partial z} = 0$ and $\frac{\partial}{\partial \theta} (any \ quantity) = 0$ (2.10)

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

$$\frac{\partial v_z}{\partial t} = -\frac{1}{\rho} \cdot \frac{\partial p}{\partial z} + v \left(\frac{\partial^2 v_z}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial v_z}{\partial r} \right) \text{ in z direction}$$
 (2.11)

For steady flow, the governing equation becomes

$$\frac{\partial^2 v_z}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial v_z}{\partial r} = \frac{1}{\mu} \frac{dp}{dz} \tag{2.12}$$

Solving differential equation 2.12 with boundary conditions

$$0$$
; v_z is finite (2.13)

$$r = R \; ; \; v_z = 0$$
 (2.14)

Yields

$$v_z = \frac{R^2}{4\mu} \left(-\frac{dp}{dz} \right) \left(1 - \frac{r^2}{R^2} \right) \tag{2.15}$$

While

$$-\frac{dp}{dz} = \frac{\Delta p}{L} \tag{2.16}$$

The viscosity of distribution is noted across the capillary is said to be parabolic. Overall flow rate (Q) are achieved through the integration of the following expression.

$$Q = \int_0^R 2\pi v_z \, r \, dr \tag{2.17}$$

Inserting 2.15 & 2.16 into 2.17, we obtain

$$Q = \pi \frac{R^4}{8\mu} \left(\frac{\Delta p}{L}\right) \tag{2.18}$$

The above equation is recognised as the Poiseuille's equation for the calculation of viscosity during the use of capillary viscometer.

$$Q = \frac{V}{t} \tag{2.19}$$

Where:

Q is overall flow rate

V is volume

t is time

$$v = \frac{\mu}{\rho} \tag{2.20}$$

In the capillary viscometer in the case of a vertical arrangement, hydrostatic pressure, ρgh , which be governed by on the height, h, in the liquid. Consequently, pressure differs, Δp , in terms of the hydrostatic pressure which stays governed by the given equation below;

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

$$v = \frac{\pi g H R^4}{8LV} \cdot t \tag{2.21}$$

k, constant for viscometer

$$k = \frac{\pi g H R^4}{8LV} \tag{2.22}$$

Then,

$$v = kt \tag{2.23}$$

Equation 2.23 is similar to ASTM kinematic viscosity equation with an exception of the correction factor.

$$\upsilon = \left(\frac{10\pi g D4Ht}{128VL}\right) - \frac{E}{t2} \tag{2.24}$$

Where E is the correction factor.

2.3 Response Surface Methodology (RSM)

The Response to surface methodology (RSM) is an assembly of a numerical or exact or calculated method valuables used in developed, refining, and enhancing processes (Myers, and Montgomery, 2002). RSM method are seen as one of the most expensive methods which

are specific conditions are of several inputs variable potentially consequence of most performance of quality or measure features of characteristics of these process. The input variables are known as independent variables, and are themed in the control of scientist or engineers. RSM method entails experimental approach in the explore of space in the process or independent variables, in other to develop an appropriate approximate model for the empirical statistics between the yield and process variables, including the optimizing methods in finding the values in the process variables which produce a desired value of response. In other to develop an appropriate approximation of the model, a statistical modelling was developed, between the model y and independent variables $\xi_1, \xi_2,...,\xi_k$.

The general relationship is given as;

$$y = f(\xi 1, \xi 2, \dots, \xi k) + \varepsilon \tag{2.26}$$

Through a cautious design analysis in experiment, the aim is to improve on the response (output variable) influenced by numerous a self-governing variables (input variables). Experimental analysis of series of test records, are known as run, whereby changes are prepared in the output variables which helps to recognise the change of the out response. RSM method was established in other to models the experimental response (Box, and Draper, 1987), which migrates into the model of numerical experiments. During experimental measurement errors can be due to inaccuracy of the results, however in computer experiments, inaccuracies or discrete illustration of constant physical phenomena (Myers, and Montgomery, 2002) maybe due to arithmetic noise, which as a product in an imperfect conjunction in iterative procedures. The mistakes in RSM method, are assumed to be random.

It's important to understand that RSM takes in amount of some advantages of dropping the number of experimental runs, in which it's important and necessary to provide statistically satisfactory results.

2.3.1 Applications

- The most frequent applications of RSM are in the industrial, Biologicial, Agriculture, Electronics, Medical field and many others like this. it is used where optimium response is required.
- 2. RSM is important in design formulation, improvement and the development and analysing of new specific scientific study and product.

2.4. Standards

Internationally, the production of biodiesel with any of the methods must meet the international biodiesel standard specifications, Biodiesel producers, engine designers and consumers must know these standards. It is designated by B100 the assembly of specifications in American Society for Testing and Materials (ASTM D 6751-3) or the European Union (N 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 (Srivastava, and Prasad, 2000).

The engineering sector, the quality control and specification of fuel characteristic, ASTM D 975 requires a kinematic viscosity ranges from 1.9 minimum to 4.1 maximum mm^2/s at 40° C, biodiesel per ASTM D 6751 requires $1.9 - 6.0 \text{ mm}^2/s$ at 40° C, biodiesel per EN. The unit " mm^2/s " can be replace directly by cSt.

CHAPTER 3 MATERIALS AND METHODS

3.1 Biodiesel Samples

Five different blends of biodiesel + diesel fuel (BD + DF), biodiesel + benzene (BD + B), biodiesel + kerosene (BD + K), biodiesel + euro diesel winter (BD + EDW), and biodiesel + euro diesel summer (BD + EDS) were prepared by volume fractions of 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, 0.50, 0.55, 0.60, 0.65, 0.70, 0.75, 0.80, 0.85, 0.90 and 0.95, were used. All components are completely miscible. Biodiesel are produced by different methods and a numbers of possible different routes ("Oil, Grease and Petroleum Testing Instruments," 2013). The biodiesel used for this experiment was produced in K.Th.G. Eco-logical Fuels Co. Ltd, The comparisons of the constituents of the vegetable oils and animal fats and petroleum are obtained in diesel that make the vegetable oils right for conversion to biodiesel (ASTM D 446-07, 1995).

3.2 Measurement of Viscosity

When measuring the viscosity an important significant importance that should be put into consideration is in both businesses or production sector and academia. The measurement of viscosity through a device known as viscometer, Viscosity of a fluid can be measured by several types of viscometer and rheometers which could be used. The rheometer are used in fluid that can't be well-defined just one only value of viscosity and are consequently are required toward having more than a parameter which measures and set in the case of a viscometer.

Viscosity is stated in kerbs units (KU), which stay exceptional to stormer viscometers. The ford viscosity cup aids to measure the degree of the flow of liquid. In an ideal condition it is proportionate to the kinematic viscosity. The vibrating viscometer are also been used in the measurement of viscosity. Capillary viscometers is one of the extensively used instrument for measuring the viscosity of Newtonian liquids.

3.2.1 Ubbelohde Viscometer

These type of viscometer or suspended-level viscometer is an instrument used in measurement use in of a capillary base method in the measurement of a viscosity. It is in

suggestion for high viscosity for a cellulosic polymer solution. One benefits in these kind of device stands as that the values gotten are self-governing of the entire volume. This device was invented by a German chemist Leo Ubbelohde (1877-1964) (Poonam et al., 2011). Figure 3.1 shows some details for Ubbelohde viscometer.

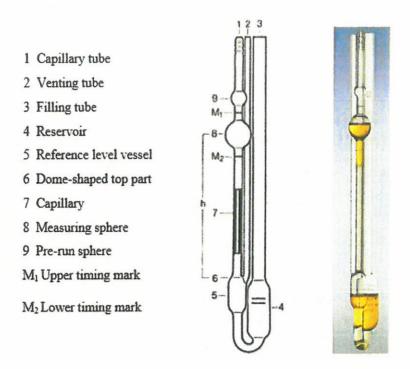


Figure 3.1: Illustrated diagram of ubbelohde viscometer

The viscometer essentially entails capillary tube (1), venting tube (2) and the filling tube (3), the capillary (7) with measuring sphere (8), the pre-run sphere (9) (or Ubbelohde Viscometers) and the reference level vessel (5). Up and down measuring sphere (8) are in print on timing marks M_1 and M_2 . These inscriptions not only describe the flow-through volume of the sample, nevertheless likewise 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).

Biodiesel sample is pulled into the reservoir through the filling tube (3). The U-tube at the bottom which have to be filled completely and ought to be unrestricted from any form of air

bubbles and particulate matter. After desired temperature should be gotten, and plug or finger is located over venting tube (2) and then sucked gently through the capillary tube (1) than measure the bulb. The suction is detached from capillary tube (1) and the plug is removed from venting tube (2).

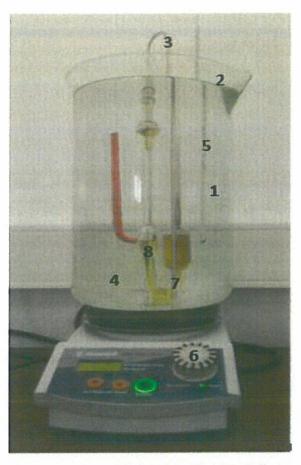
Liquid is permitted to travel back from end to end in the measuring bulb and the time it takes for the liquid to pass through the two calibrated marks should be noted and calculated (upper timing mark and lower timing mark) and the viscosity measured. Ubbelohde device the third arm which extends very close to the capillary and opens up to the atmosphere. In like manner the pressure head depends merely on a static height and is extended on the entire volume of the fluid (Poonam, and Anoop, 2011; Helwani et al., 2009).

Ubbelohde viscometer is beneficial through the purpose in the kinematic viscosity of a translucent Newtonian liquids in the ranges 0.3 to about 100,000 cSt (mm²/s). These kind of measurement procedure has an advantage of posing the same constant temperature during all experimental procedure. These unique properties is of great advantage measurements procedure and are carried out through the numbers of dissimilar forms of temperatures. The flow of the fluid is made to pass merely downwards through the fortifications of the bulb underneath the capillary, which forms a on hold at level that helps to ensure the fluid level at automatically at stationary and are coincided by means of the minor close to the capillary, in other to avoid these necessity in other to bring up the viscometer with certain volume of the fluid and the request of correction for the development of glass in line for to the varieties in the temperature (Poonam, and Anoop, 2011; Helwani et al., 2009).

Ubbelohde type viscometer has some benefits such as speed, low susceptibility to inaccuracies and an accurate result (within \pm 0.1%). It needs a slight amount of sample size (15 mL). The apparatus are way more inexpensive than those of the other models which provide same kind of an accurate result. The central concern of this viscometer is that it view the blockage (particularly, in minor capillaries), (Wilke et al.).

There are many different categories of ubbelohde viscometers covering the kinematic viscosity in the variety of even less than 0.3 to above 100,000 cSt (mm²/s). In Tables 3.1, size number of ubbelohde viscometers, their constants which corresponds to the kinematic viscosity that range are been tabulated.

3.2.2 Experimental Set-up and Methods



- 1: Water at low temperature and silicon oil at high temperature
- 2: 3000ml Standard Beaker / Oil Bath
- 3: Capillary Holder
- 4: Electromagnetic mixer
- 5: Thermometer
- 6: Electromagnetic plate
- 7: Capillary Viscometer
- 8: Biodiesel sample

Figure 3.2: Illustrated diagram of the Experimental set-up

Silicon oil (1) in a standard beaker (2) is used as oil bath. The capillary viscometer (3) is placed in its holder (3) which holds it in an upright position in the oil bath. The oil bath is heated by an electromagnetic plate (6) and its temperature is controlled by a standard thermometer (5). The methods, procedures and the use of each equipment will be illustrated below.

Table 3.1: Various Types of Ubbelohde Viscometers for Transparent Fluids

Size no:	Approximate	Kinematic	Inside Diameter	Volume,	Inside Diameter
	Constant,	Viscosity Range	of Tube, R, mm	Bulb	of Tube P,ml
	$(mm^2/s)/s$	(mm^2/s)	(±2%)	C,ml	(±5%)
				(±5%)	
0	0.001	0.3 ^A to 1	0.24	1.0	6.0
0C	0.003	0.6 to 3	0.36	2.0	6.0
0B	0.005	1 to 5	0.46	3.0	6.0
1	0.01	2 to 10	0.58	4.0	6.0
1C	0.03	6 to 30	0.78	4.0	6.0
1B	0.05	10 to 50	0.88	4.0	6.0
2	0.1	20 to 100	1.03	4.0	60
2C	0.3	60 to 300	1.36	4.0	6.0
2B	0.5	100 to 500	1.55	4.0	6.0
3	1.0	200 to 1000	1.83	4.0	6.0
3C	3.0	600 to 3000	2.43	4.0	6.0
3B	5.0	1000 to 5000	2.75	4.0	6.5
4	10	2000 to 10,000	3.27	4.0	7.0
4C	30	6000 to 30,000	4.32	4.0	8.0
4B	50	10,000to50,000	5.20	5.0	8.5
5	100	20,000to100,000	6.25	5.0	10.0

A300-s least flow time; 200-s least flow time for all other units

The ubbelohde viscometer (ASTM) was selected due to its wide known application and accuracy. It enables transparent and high temperature measurement. Two viscometers of size According to their kinematic viscosity range, two viscometer of size Oc and I were chosen in this work for measuring kinematic viscosity, they are both calibrated with constants for manual measurements (Appendix 3 shows the technical specifications of the viscometers). The manufacturer has done their calibrations and their constant k is given in Table 3.1 for manual measurements.

3.2.3 Measurement of the Flow Time of the Ubbelohde Viscometer

In the case flow time, is measured by the use of an operator known as the stop watch. The manufacture of Glass viscometers was meant for the purpose which must have an annular measurement mark burnt to an above and below the measurement sphere. One major disadvantage of these are clear:

- Generally observations in errors are made or the dissimilarities of the feedback time of
 the machinist is used must start and finish at the timing which will lead to an increase
 in the reproducibility of doubts and, below certain conditions, to a systematic error
 which can be made.
- Another factor of this is for a cloudy materials the meniscus may not be seen. One has alternative to Reverse-Flow Viscometers with their more difficult behaviour and reduced exactness.

For manual measurement the flow time, the liquid is sucked into a measurement sphere through the application of the vacuum in the capillary tube. When viscometers is in use with feeder sphere, latter must be poured up to at least up to its half

The pre run sphere without viscometers must be filled until the liquid meniscus should be approximately 20mm above and below the annular mark. The Ubbelohde viscometers are used, fingertip should be used to close the venting tube prior to the start of sucking in. In completion to the filling process the suction hose should be removed from the capillary tube and, the venting tube should be released in the case of the ubbelohbe viscometer.

Once the sample to be measured is viscous its recommendable to retain the capillary tube should be closed after the release of the venting tube till the runs empty and the suspended level to be built up.

In a volatile substance are measured suggestions should be made to perform of the filling measurement sphere in the application of an over pressured filling tube, when no bubbles occurs only in fluids. Ubbelohde viscometers in the closing and opening of the venting tube should be done analogously.

The lower vertex of the meniscus sinks from the top edge in the upper annular mark below until the top edge of the down annular mark. It should be taken into consideration that the using the stop watch should be used in timing must have a dissolution of at minimum be in the range of 0.1s. During the passage of the meniscus detection should be made at an eye level to observe the annual mark.

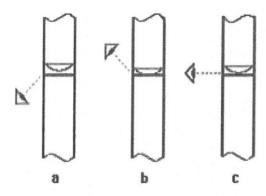


Figure 3.3: Manual measurement of the meniscus (a) and (b) incorrect detection, (c) correct detection of the meniscus passage

3.2.4 Temperature Measurement

Temperature measurement is measured with a standardized calibrated mercury thermometer used to control the temperature in the beaker bath of water at low temperature using water and at high temperature using silicon oil.

According to the ASTM D445 standards it is important to maintain a constant temperature to avoid or reduce unnecessary errors. To ensure that the temperature in the oil bath is uniform, a metal like rod is used to stir the silicon oil to a uniform temperature two thermometers are then used, one for fixed control and the other or varying control.

3.2.5 Accessories

In other to achieve a more conservative and an effective measurement of kinematic viscosity, additional accessories were used this includes;

- 1. Glass pipette: used for transporting a measures volume of biodiesel sample into the viscometer and cleaning the viscometer after changing the samples.
- 2. Vacuumed Syringe: used for suction process during measurement.
- 3. Stop watch: is used for accurate measurement for time required by the standard procedure.
- 4. Beaker Insulator: used to prevent heat loss to the environment at relatively high elevated temperature.
- 5. Viscometer Holder: are used to keep the ubbelohde capillary viscometer vertically upright in the oil bath.

3.2.6 Electromagnetic Hot Plate and Stirrer

An electromagnetic stirrer or magnetic mixer are used in the laboratory as a device that services as a rotating magnetic field which meant have an effect a stir bar ("flea") submerged into the liquid which helps to spin it very quickly, which stirs it up. The rotating field meant be made also through a rotating magnetic or set of stationary electromagnetics which are placed below the vessel with the liquid. For the purpose safe heating and mixing, the Hiedolph MR Hei-tec electromagnetic heater and stirrer was used. It is made of aluminium, thus making it to offer a fast heating times and the water-thin ceramic coating makes the heating plate both chemically and scratch resistant. Figure 3.4 gives a sample of the used plate.



Figure 3.4: Heidolph MR Hei-Tec

3.2.7 Silicon Oil

A silicon oil is a fluid that is polymerized by siloxane having an organic having side chains. The greatest significant associate is polydimethylsiloxane. These kind of polymers have a profitable importance since it is relatively of great thermal stability with their lubricating properties.

Due to the high temperature range requirement, impossible to use water as an appropriate temperature bath at 70°C to 140°C. A wacker silicone fluid AK oil was used. Wacker silicone fluid AK are dimethyl polyslloxane of whose un-branched chains which are prepared to an substitute silicon and oxygen atoms, free valences of the silicon being saturated by methyl group. However carbons chains of an organic compound should display a little confrontation to certain exterior effects, the stability of inorganic Si-O linkage is of many ways and in like manner the chemical inertness of silicate.

Table 3.2: Properties of Silicone Oil

Kinematic	Viscosity	Coefficient of	Thermal	Flash point	Pour point DIN	Volatility
viscosity	temperature	Thermal	conductivity at	ISO 2592 acc.	51794	(%)
at 25 °C	coefficient ²	expansion at 0 -	50 °C	to Cleveland	(°C)	(70)
		150°C	(W · K-1 · m-1)	(°C)		
(mm ² s- ¹)		cm ³ .10 ⁴ /cm ³ °C				
				200	50	-15
350	0.595	9.25	0.15	> 300	-50	< 1.5

A The tolerance for up to 50 mm²/s is \pm 10 %, for higher viscosity fluids \pm 5 %

^B Viscosity-temperature coefficient: $1 - \frac{kinematic\ viscosity\ at\ 99\ ^{\circ}C}{kinematic\ viscosity\ at\ 38\ ^{\circ}C}$

3.3 Methodology

The following steps below are essential for measuring the kinematic viscosity of the biodiesel blend samples.

- 1. The first step that must be put into consideration is that the viscometer must be cleaned with the concentration of about 15% H₂O₂ and 15% HCL Afterwards, the viscometer is than rinsed with a more appropriate solvent (Acetone was chosen for these experiment). The viscometer should be completely dried off and dirt free before been placed to use either done manually for measurements.
- 2. The samples were filtered to clean it from probability of lint, apart from the solid material in the liquid sample, please filter in a glass filter through a fine mesh screen.
- 3. Ensure enough the volume of each samples is introduced viscometer through filling tube into the lower reservoir by using glass pipette. Beware to introduce enough samples in other to bring the level between lines in a place on to the reservoir.
- 4. Than ensure to place the viscometer into the holder and gently insert it into the constant temperature bath. In a vertical format align the viscometer in the beaker bath if self aligning holder shouldn't be used.
- 5. Ensure the viscometer roughly stays in the beaker bath for approximately 20 minutes for the sample to come up to the temperature of the bath.
- 6. Calculation of the time interval (efflux time) was measured; which takes the leading edge of the meniscus of each sample to descend from the upper edge of the upper timing mark M₁ right up to the upper edge of the lower timing mark M₂.
- 7. The suction should be applied to capillary tube (1), 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 M1. 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.
- 8. Kinematic viscosity of each samples were calculated by the multiplication of the efflux time (t) by viscometer constant (k). To calculate for the absolute measurement, kinematic energy correction (y) was subtracted from efflux time (t) and then multiplied by constant (k). Kinematic viscosity v (mm²/s) directly.

$$v = k(t - y) \tag{3.1}$$

Where y is the kinetic energy correction (HC) described in table 3.3

9. Without recharging the viscometer, for more accurate value, repeat steps 6 to 8 two times for each experiment for each sample at the same degree of temperature.

Furthermore, the following must be noted.

• Calibration: In order to determine the relationship between the time of flow and the kinematic viscosity, a calibration of the instrument is needed. The calibration was done by the manufacturer, SI Analytics GmbH, Mainz according to ASTM D 2525/ D 446 and ISO/DIS 3105. The instrument constant k were determined and given as in Table 3.3. The calibration constant can be used up to the temperature of 140° C. The influence of the temperature on the capillary constant due to thermal expansion of the glass is very small, $3.3 \times 10^{-6} \frac{1}{K}$ (Poonam et al., 2011).

Table 3.3: Kinematic Energy Correction Ubbelohde Viscometer ISO 3105/DIN51 562/Part1/BS188/NFT 60-100 Ref.No.501...530...532. Correction seconds ^A

Flow	100		(Capillary n	0		
time	0	0c	0a	I	Lc	La	1
40	_B	_B	_B	1.03	0.45	0.15	
50	_B	_B	_B	3.96	0.66	0.29	0.10
60	_B	_B	_B	2.75	0.46	0.20	0.07
70	_B	_B	_B	2.02	0.34	0.15	0.05
80	_B	_B	4.78 ^B	1.55	0.26	0.11	0.04
90	_B	_B	3.78 ^B	1.22	0.20	0.09	0.03
100	_B	7.07 ^B	3.06 ^B	0.99	0.17	0.07	0.02
110	_B	5.84 ^B	2.53	0.82	0.14	0.06	0.02
120	_B	4.91 ^B	2.13	0.69	0.12	0.05	0.02
130	_B	4.18 ^B	1.81	0.59	0.10	0.04	0.01
140	_B	3.61 ^B	1.56	0.51	0.08	0.04	0.01
150	_B	3.14 ^B	1.36	0.44	0.07	0.03	0.01
160	_B	2.76	1.20	0.39	0.06	0.03	0.01
170	_B	2.45	1.06	0.34	0.06	0.02	0.01
180	_B	2.18	0.94	0.30	0.05	0.02	0.01
190	_B	1.96	0.85	0.28	0.05	0.02	0.01
200	10.33 ^B	1.77	0.77	0.25	0.04	0.02	0.01
225	8.20	1.40	0.60	0.20	0.03	0.01	0.01
250	6.64	1.13	0.49	0.16	0.03	0.01	< 0.01
275	5.47	0.93	0.40	0.13	0.02	0.01	< 0.01
300	4.61	0.79	0.34	0.11	0.02	0.01	<0.01

Table 3.3: Continued

325	3.90	0.66	0.29	0.09	0.02	0.01
350	3.39	0.58	0.25	0.08	0.01	0.01
375	2.95	0.50	0.22	0.07	0.01	0.01
400	2.59	0.44	0.19	0.06	0.01	< 0.01
425	2.30	0.66	0.29	0.09	0.01	< 0.01
450	2.05	0.58	0.25	0.08	0.01	< 0.01
475	1.84	0.50	0.22	0.07	0.01	
500	1.66	0.44	0.19	0.06	0.01	
550	1.37	0.23	0.1	0.03	0.01	
600	1.15	0.20	0.09	0.03	0.01	
650	0.98	0.17	0.07	0.03	< 0.01	
700	0.85	0.14	0.06	0.02	< 0.01	
750	0.74	0.13	0.05	0.02	<0.01	
800	0.65	0.11	0.05	0.01		
850	0.57	0.10	0.04	0.01		
900	0.51	0.09	0.04	0.01		
950	0.46	0.08	0.03	0.01		
1000	0.42	0.07	0.03	0.01		

A from the above the correction stated are related to the respective theoretical constant.

3.6.1 Flow Chart for Determining Kinematic Viscosity

In other to entirely appreciate the methodology, a system flow chart is designed. Figure 3.7 exemplifies the methodology flow chart for determination of kinematic viscosity using an ubbelohde viscometer.

^B for a more accurate measurement, the flow time shouldn't be applied. The selection of a smaller sized viscometer of capillary diameter.

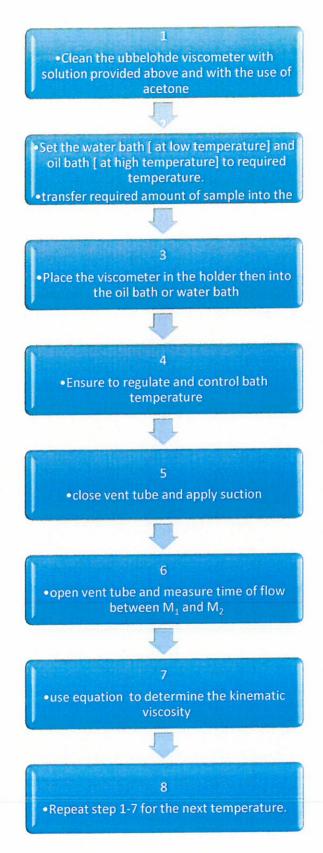


Figure 3.5: Methodology flow chat

These measurements should be prepared at room temperature. At all times ensure a gradually pour the solution in other to avoid bubbles that mat are slow to escape and may affect the experimental result.

The measurements of the kinematic viscosity for each sample have been performed according to the flow chart given and calculations of kinematic viscosities. Table 4.1 shows the results of kinematic viscosity for biodiesel binary blends with diesel fuel, benzene and kerosene, euro diesel winter, and euro diesel summer at 40°C.

The flow time, the kinematic viscosity has been calculated by using equation above. In Table 3.4, the other parameters are also given for calculation of the viscosity such as "k" (k is constant of capillary) and "HC" or "y" (HC is kinetic energy correction) kinematic viscosity values are used are the kinematic viscosity values in table and figures.

Table 3.4: Kinematic Viscosity Calculation

Time	Constant for capillary no	Kinetic	Kinematic
(sec)	"I" at low temperature	energy	viscosity
(500)	from 20°C - 60°C and	correction	(mm²/s)
	"Oc" high temperature	(HC)	(
	from 70°C -140°C	()	
427	0.009132	0.09	3.8986
252	0.009132	0.13	2.3001
310	0.009132	0.09	2.8301
389	0.009132	0.06	3.5518
401	0.009132	0.09	3.6976
	252 310 389	(sec) "I" at low temperature from 20°C - 60°C and "Oc" high temperature from 70°C -140°C 427 0.009132 252 0.009132 310 0.009132 389 0.009132	(sec) "I" at low temperature energy from 20°C - 60°C and correction "Oc" high temperature from 70°C -140°C 427 0.009132 0.09 252 0.009132 0.13 310 0.009132 0.09 389 0.009132 0.06

In the same manner and using the flow chart, the kinematic viscosity of all 21 samples has been calculated from 20°C up to 140°C.

CHAPTER 4 RESULT AND DISCUSSION

Several measurements took place in order to verify that the experimental values of kinematic viscosity were precise. To demonstrate the presented results, ensure the required accuracy, a number of plots was prepared. Kinematic viscosity were plotted against temperature shown in figures below.

The experimental data on the kinematic viscosity (mm²/s), for blend of biodiesel with diesel fuel, benzene, kerosene and euro diesel summer and euro diesel winter respectively, for twenty-one different compositions between 20°C and 140°C, are presented in appendix 4.

The general biodiesel diesel samples of all blend varies in the range of 6.0269–1.2835mm²/s which are higher than those of diesel fuel, 4.6114– 0.9263mm²/s, in the temperature range 20°C and 140°C.

The relationship between the kinematic viscosity, temperature of the blends of biodiesel + diesel fuel (BD + DF), biodiesel + benzene (BD + B), biodiesel + kerosene (BD + K), biodiesel + euro diesel summer (BD + EDS), and biodiesel + euro diesel winter (BD + EDW), respectively are shown in figures below.

 Table 4.1: Ubbelohde Viscometer Repeatability Results for some Biodiesel Samples

Fluid Type	Temperature (°C)	Measured kinematic viscosity	Average kinematic viscosity	Absolute error (mm ² /s)
		(mm^2/s)	(mm^2/s)	
100% BD + 0% DF	40	4.2183	4.2183	0
		4.2183		
75% BD + 25% DF	40	3.9078	3.8986	0.2354
		3.8895		
50% BD + 50% DF	40	3.5975	3.5975	0
		3.5975		
25% BD + 75% DF	40	3.3417	3.3325	0.9200
		3.3234		
0% BD + 100% DF	40	3.1041	3.1041	0
		3.1041		
100% BD + 0% DF	80	2.0959	2.0931	0.1336
		2.0902		
75% BD + 25% DF	80	1.9303	1.9131	0.8911
		1.8960		
50% BD + 50% DF	80	1.7502	1.7502	0
		1.7502		
25% BD + 75% DF	80	1.6016	1.6016	0
		1.6016		
0% BD + 100% DF	80	1.4331	1.4302	0.2093
		1.4267		
0% BD + 100% EDW	40	3.9352	3.9260	0.2338
		3.9169		
75% BD + 25% EDW	40	3.5518	3.5518	0
		3.5518		
50% BD + 50% EDW	40	3.4970	3.4604	1.0466
		3.4239		
25% BD + 75% EDW	40	3.1315	3.1315	0
		3.1315		
0% BD + 100% EDW	40	3.0950	3.0950	0
		3.0950		
100% BD + 0% EDW	80	1.8846	1.8846	0

Table 4.1: Continued

		1.8846		
75% BD + 25% EDW	80	1.7817	1.7817	0
		1.7817		
50% BD + 50% EDW	80	1.6845	1.6702	0.8489
		1.6559		
25% BD + 75% EDW	80	1.5244	1.5216	0.1837
		1.5187		
0% BD + 100% EDW	80	1.4816	1.4816	0
		1.4816		
0% BD + 100% EDS	40	4.2275	4.2183	0.2176
		4.2092		
75% BD + 25% EDS	40	3.6976	3.6976	0
		3.6976		
50% BD + 50% EDS	40	3.3782	3.36907	0.2694
		3.3599		
25% BD + 75% EDS	40	3.2504	3.2412	0.2830
		3.2321		
0% BD + 100% EDS	40	3.1315	3.1224	0.2906
		3.1133		
100% BD + 0% EDS	80	2.0931	2.0931	0
		2.0931		
75% BD + 25% EDS	80	1.7988	1.7903	0.4725
		1.7817		
50% BD + 50% EDS	80	1.6359	1.6359	0
		1.6359		
25% BD + 75% EDS	80	1.5616	1.5616	0
		1.5616		
0% BD + 100% EDS	80	1.4902	1.4873	0.1946
		1.4845		
100% BD + 0% K	40	3.9443	3.9260	0.4640
		3.9078		
75% BD + 25% K	40	2.8666	2.8301	1.2733
		2.7936		

Table 4.1: Continued

50% BD + 50% F	40	2.2633	2.2267	1.6171
		2.1902		
25% BD + 75% K	40	1.5599	1.5599	0
		1.5599		
0% BD + 100% K	40	1.1726	1.1726	0
		1.1726		
100% BD + 0% K	80	1.9531	1.9502	0.1485
		1.9474		
75% BD + 25% K	80	1.6673	1.6673	0
		1.6673		
50% BD + 50% K	80	1.1919	1.1862	0.4782
		1.1805		
25% BD + 75% K	80	0.9092	0.9035	0.6269
		0.8978		
0% BD + 100% K	80	0.7342	0.7342	0
		0.7342		
100% BD + 0% B	40	4.0995	4.0995	0
		4.0995		
75% BD + 25% B	40	2.3092	2.3001	0.3941
		2.2909		
50% BD + 50% B	40	1.2778	1.2636	1.1113
		1.2493		
25% BD + 75% B	40	0.7570	0.7428	1.8758
		0.7285		
0% BD + 100% B	40	0.4907	0.4907	0
		0.4907		
100% BD + 0% B	80	1.8988	1.8988	0
		1.8988		
75% BD + 25% B	80	1.2436	1.2436	0
		1.2436		
50% BD + 50% B	80	0.9808	0.9751	
		0.9694		
25% BD + 75% B	80	0.6565	0.6565	0
		0.6565		
0% BD + 100% B	80	0.3781	0.3781	0
		0.3781		

4.1 Kinematic Viscosity of Euro Diesel Summer and Euro Diesel Winter

Figure 4.1 and Figure 4.2 describes the kinematic viscosity, at temperature range of 20°C to 140°C for BD + EDW and BD + EDS decreases with an increase in temperature. The kinematic viscosity of euro diesel summer and euro diesel summer are lower than biodiesels. Therefore, the kinematic viscosity of the blend increases with the increase of biodiesel concentration. Finally the kinematic viscosity increases with an increase biodiesel volume content with euro diesel summer and euro diesel winter. Both BD + EDW and BD + EDS has a slight similarities in reference to their kinematic viscosity.

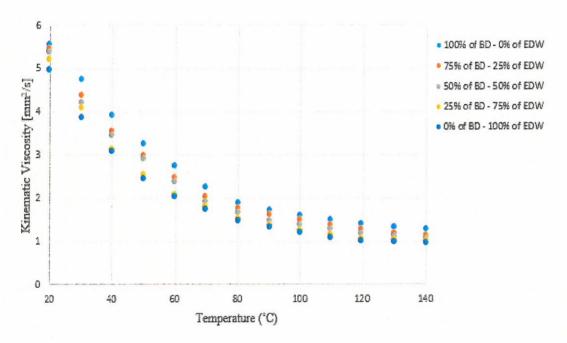


Figure 4.1: Kinematic viscosity of blend of biodiesel (BD) and euro diesel winter (EDW)

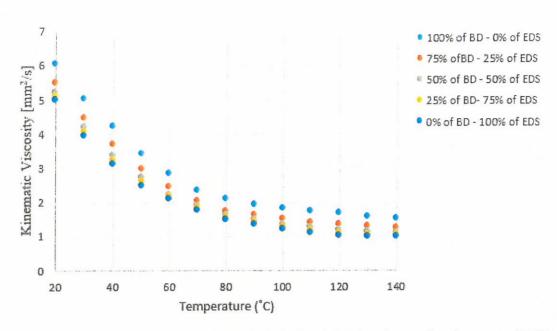


Figure 4.2: Kinematic viscosity of blend of biodiesel (BD) and euro diesel summer (EDS)

4.2 Kinematic Viscosity of Kerosene

Figure 4.3 describes the kinematic viscosity at temperature range of 20° Cto 140° C of BD + K decreases with an increase in temperature. The kinematic viscosity of kerosene are lower than biodiesels. Therefore, the kinematic viscosity of the blend increases with the increase of biodiesel concentration. Finally the kinematic viscosity increases with an increase biodiesel volume content with kerosene, the results of BD + K is weaker than those of BD + EDW and BD + EDS.

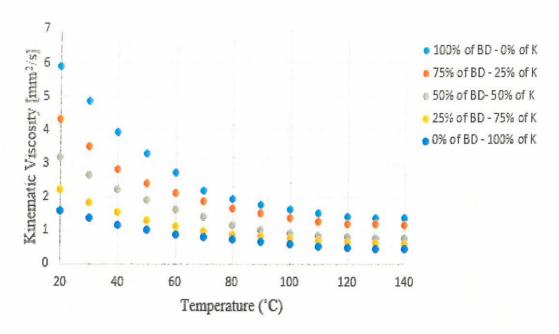


Figure 4.3: Kinematic viscosity of blend of biodiesel (BD) and kerosene (K)

4.3 Kinematic Viscosity of Benzene

Figure 4.4 describes the kinematic viscosityat temperature range of 20°C to 140°C of BD + B decreases with an increase in temperature. At high temperature above the room temperature of benzene, evaporation takes place. Likewise when temperature increases for pure benzene it's impossible to reach high temperature above 80°C, with blends of biodesel volume content it's possible to attain high temperature upto 140°C. The kinematic viscosity of benzene are lower than biodiesels. Therefore, the kinematic viscosity of the blend increases with the increase of biodiesel concentration. Finally the kinematic viscosity increases with an increase biodiesel volume content with kerosene, the results of BD + B and BD + K is weaker than those of BD + EDW and BD + EDS.

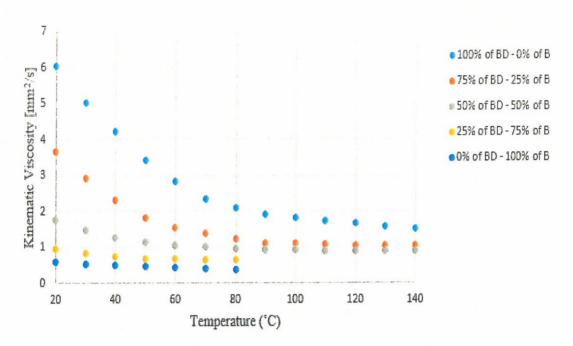


Figure 4.4: Kinematic viscosity of blend of biodiesel (BD) and benzene (B)

4.4 Kinematic Viscosity of Diesel Fuel

Figure 4.5 shows that kinematic viscosity decreases as temperature increases as expected. From the figures we could see that for all samples tested, as temperature increases, the kinematic viscosity decreases. This is in line with other samples tested all over the world. It can be explained by kinetic molecular theory. The kinematic viscosity of diesel fuels are lower than biodiesels. Therefore, the kinematic viscosity of the blend increases with the increase of biodiesel concentration.

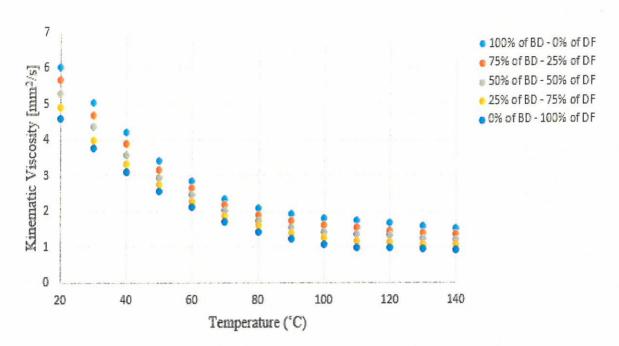


Figure 4.5: Kinematic Viscosity of blend of biodiesel (BD) and diesel fuel (DF)

4.5 Experimental Strategy for Predicting Kinematic Viscosity using the RSM model

The generalized equation for predicting the kinematic viscosity of blends for each sample was given using the RSM model. It's important to understand that using the RSM takes in amount of some advantages of dropping the number of experimental runs, in which it's important and necessary to provide statistically satisfactory results in these project we will concentrate on the statistical modeling in other to develop a well appropriate and approximate model using the equation 4.1 below.

$$f(x,y) = P00 + P10 * x + P01 * y + P20 * x^{2} + P11 * x * y + P02 * y^{2} + P30 * x^{3} + P21 * x^{2} * y + P12 * x * y^{2} + P03 * y^{3} + P31 * x^{3} * y + P22 * x^{2} * y^{2} + P13 * x * y^{3} + P04 * y^{4} + P32 * x^{3} * y^{2} + P23 * x^{2} * y^{3} + P14 * x * y^{4} + P05 * y^{5}$$

$$(4.1)$$

Where,

x= temperature

y = volume fraction of biodiesel

Table 4.2: Binary Fuel Blends with Biodiesel using the RSM model

Polynomial		Values			
equation					
coefficient					
-	Kinematic	Kinematic	Kinematic	Kinematic	Kinematic
	viscosity	viscosity	viscosity	viscosity	viscosity
	BD+K	BD+DF	BD+EDW	BD+B	BD+EDS
p00	2.157	6.641	7.561	1.135	7.601
p10	-0.03331	-0.1171	-0.1582	-0.0146	-0.1566
p01	2.011	1.879	1.95	-2.792	0.518
p20	0.000257	0.0007855	0.001338	4.47E-05	0.001294
p11	-0.0418	-0.0236	-0.05334	0.04825	0.005279
p02	10.25	-3.884	-1.561	13.71	-0.7838
p30	-7.80E-07	-1.71E-06	-3.91E-06	0.00E+00	-3.68E-06
p21	0.000229	0.000346	0.000375	-0.00037	-6.56E-05
p12	-0.1291	-0.02722	0.113	-0.06302	0.01779
p03	-14.62	12.79	-0.5959	-16.1	2.579
p31	5.97E-07	-1.89E-06	-5.23E-07	0.00E+00	1.50E-07
p22	0.000429	0.0004579	-0.00103	0.000865	0.000121
p13	0.158	-0.05665	-0.02911	-0.1013	-0.00442
p04	8.622	-10.5	-0.7533	13.23	-0.7785
p32	-3.32E-06	2.45E-09	1.88E-06	0.00E+00	-
p23	0.000366	-0.0002464	0.000366	0	-
p14	-0.1173	0.0536	-0.01892	0	-
p05	0.2332	2.111	1.689	0	17

4.5.1 Kinematic Viscosity with RSM model

As a validation, Figures 4.6 to Figures 4.9 of the kinematic viscosity of fuel blends with biodiesel, gives a straight line result in correctation coefficient are greater than 0.99 which implies that all our correlations are scientifically correct and alright. Figure 4.9 having R² as 0.9616 proves incorrect because its below the correctation coefficient of 0.99. It should be taken into account that the higher R² values shows that the relation has high accuracy, the euro diesel summer the correctation coefficient is raised to the powers of 4.

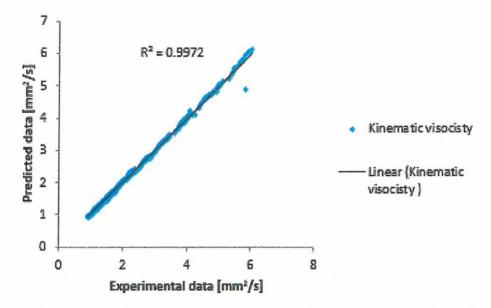


Figure 4.6: Kinematic viscosity of experimental data of blend of biodiesel (BD) and diesel fuel (DF)

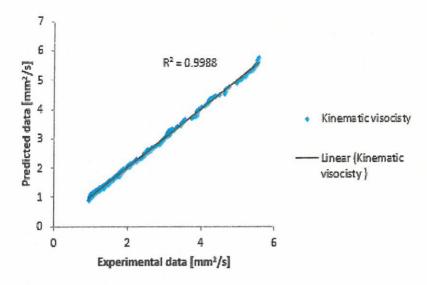


Figure 4.7: Kinematic viscosity of experimental data of blend of biodiesel (BD) and euro diesel winter (EDW)

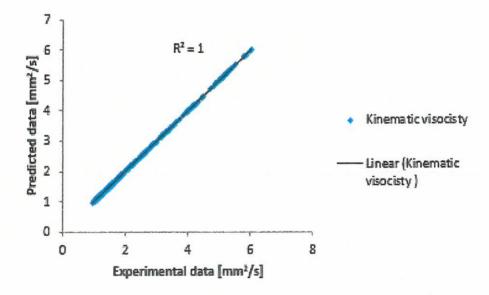


Figure 4.8: Kinematic viscosity of experimental data of blend of biodiesel (BD) and euro diesel summer (EDS)

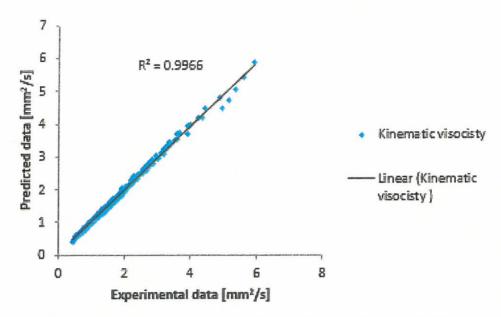


Figure 4.9: Kinematic viscosity of experimental data of blend of biodiesel (BD) and kerosene (K)

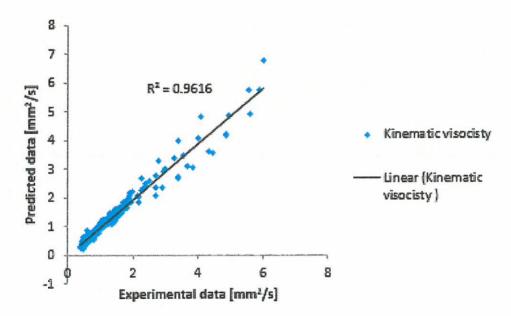


Figure 4.10: Kinematic viscosity of experimental data of blend of biodiesel (BD) and benzene (B)

4.5.2 Kinematic Viscosity with RSM model for an Ensemble Contour Plot

Figures 4.10 to Figures 4.14 below shows the kinematic viscosity of an ensemble contour plots of the fitted surface of blend of biodiesel with diesel fuel, euro diesel summer, euro diesel winter, kerosene and benzene. At an elevated temperature the effect of temperature become more dominant and then all samples seems to have approximately same kinematic viscosity values at a particular temperature this is also because of the effect of the flash point.

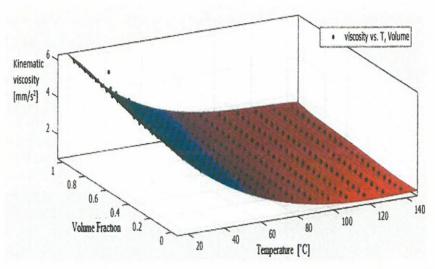


Figure 4.11: The display of kinematic viscosity of an ensemble contour plots of the fitted surface of blend of biodiesel (BD) and diesel fuel (DF)

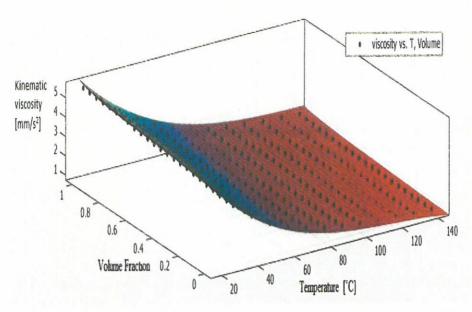


Figure 4.12: The display of kinematic viscosity of an ensemble contour plots of the fitted surface of blend of biodiesel (BD) and euro diesel winter (EDW)

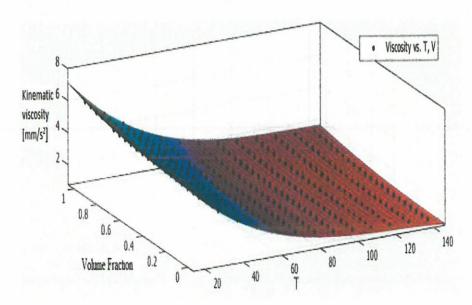


Figure 4.13: The display of kinematic viscosity of an ensemble contour plots of the fitted surface of blend of biodiesel (BD) and euro diesel summer (EDS)

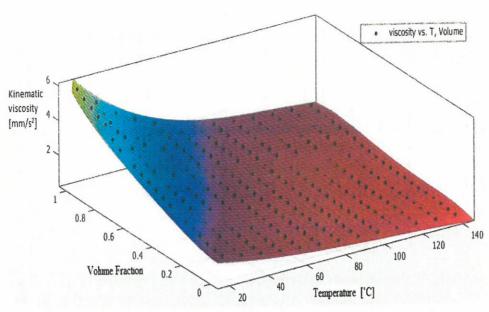


Figure 4.14: The display of kinematic viscosity of an ensemble contour plots of the fitted surface of blend of biodiesel (BD) and kerosene (K)

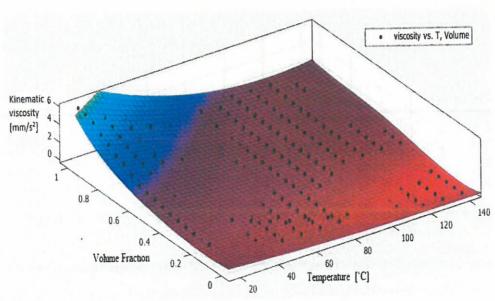


Figure 4.15: The display of kinematic viscosity of an ensemble contour plots of the fitted surface of blend of biodiesel (BD) and benzene (B)

In similar way, we can also find computational empirical coefficients for the kinematic viscosity composition relationships. Several test was done from 20°C to 140°C for five blends of diesel. Viscosity depends on the temperature change can be found using Andrade equation (Esteban et al., 2012).

Andrade equations are;

$$\ln(v) = A + \left(\frac{B}{T}\right) \quad or \quad v = A^{I} exp\left(\frac{B}{T}\right) \tag{4.2}$$

Where A, B and C are constant coefficients for the fluid, η is kinematic viscosity and T refers to the temperature. At temperature above the normal boiling point, the ln υ versus (1/T) relationship becomes non-linear and is described by a number of semi-empirical methods including those based on the principle of corresponding state. At this state kinematic viscosity is often represented by the Andrade equation or a modified form proposed by Tat and Van Gerpen.

$$\ln(v) = A + \left(\frac{B}{T}\right) + \frac{C}{T^2} \tag{4.3}$$

A third degree empirical equation was developed in other to calculate the kinematic viscosity of the five blends with biodiesel. The regression is computed for temperature range between 20°C to 140°C which is given by the general form of the equation as a function of biodiesel fraction is given by:

$$\eta_{blend} = AxT^3 + BT^2 + CT + D \tag{4.4}$$

Where η is the kinematic viscosity (mm²/s), A, B, C, D are coefficients and x is the desire binary blend of each specific percentage of blend in our mixture. The correlation coefficients is given by equation 4.4 for each fuel sample were given in Table 4.3, the abosolute error are found in Table 4.1.

Table 4.3: Equation for Prediction of Kinematic Viscosity of Five Blends with Biodiesel

System	Samples	Α	В	С	D	\mathbb{R}^2
						2.2222
BD+DF	100%BD - 0%DF	-3E-06	0.0013	- 0.1662	8.9204	0.9988
	75%BD - 25%DF	-3E-06	0.0012	- 0.1578	8.4055	0.9996
	50%BD - 50%DF	-3E-06	0.0011	- 0.1438	7.7772	0.9998
	25%BD - 75% DF	-2E-06	0.0009	- 0.1282	7.1267	0.9997
	0%BD - 100%DF	-2E-06	0.0008	- 0.1171	6.637	0.9998
BD+EDW	100%BD - 0% EDW	6E-06	5E-06	- 0.0971	7.4776	0.9996
	75%BD - 25% EDW	-4E-06	0.0013	- 0.1587	8.1196	0.9995
	50%BD - 50% EDW	-4E-06	0.0013	- 0.1589	8.0408	0.9989
	25%BD - 75% EDW	-4E-06	0.0015	- 0.1725	6.6119	0.9773
	0%BD - 100% EDW	-4E-06	0.0013	- 0.1574	7.5696	0.9988
BD+EDS	100%BD - 0% EDS	-3E-06	0.0013	-0.1668	8.9375	0.9989
<i>BB</i> - <i>B2</i> -	75%BD - 25% EDS	-3E-06	0.0013	- 0.16	8.2355	0.9997
	50%BD - 50% EDS	-4E-06	0.0013	- 0.159	7.9031	0.9997
	25%BD - 75% EDS	-4E-06	0.0013	- 0.1575	7.7476	0.9993
	0%BD - 100% EDS	-4E-06	0.0013	- 0.1558	7.5779	0.999
BD+K	100%BD - 0% K	-3E-06	0.0013	- 0.1629	8.6835	0.9994
	75%BD - 25% K	-3E-06	0.001	- 0.1186	6.2868	0.9968
	50%BD - 50% K	-1E-06	0.0005	- 0.0707	4.4116	0.9994
	25%BD - 75% K	-1E-06	0.0004	- 0.0554	3.1635	0.9996
	0%BD - 100% K	-6E-07	0.0002	- 0.0332	2.1755	0.9989
BD+B	100%BD - 0% B	-3E-06	0.0011	- 0.1571	8.7125	0.9993
	75%BD - 25%B	-3E-06	0.0011	-0.1238	5.7222	0.9989
	50%BD - 50% B	-1E-06	0.0004	- 0.0413	2.4086	0.995
	25%BD - 75% B	-2E-06	0.0005	- 0.033	1.4612	0.999
	0%BD - 100% B	-3E-07	9E-05	- 0.0099	0.77	0.999

From Table 4.4 above, the correlation coefficients are also greater than 0.99 which implies that all our correlations are scientifically alright, in like manner all correlations are within an acceptable range.

From appendix 3 of the experimental data's for kinematic viscosity and Table 4.4, these data's values can be used by design engineers to optimize the performance in the fuel system and even to control combustion in,

- Temperature
- Turbulence
- Time.

The temperature at each point in the fuel system is very important which is one of the major dependent factors of viscosity. In like manner, the turbulence is very important. The turbulence cannot be determined without the Reynolds's number, a determining parameter derived from viscosity. Also the time flow rate is of great importance.

With reference to the following articles stated below, in comparison of my experimental results by using the software, which helped to predict the accurate response by using a desirability approch response surface methodology (RSM). Thus the RSM is found to be a more effective method for a multi-objective optimization in sectors that require optimium response. In reference to the above statements, I concluded scientifically that my results for the correlation coefficients of each binary blends with biodiesel are accurate, scientifically alright, in like manner all correlations are within an acceptable range.

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- Krishnamoorthi, M., 2015, 'Response Surface Methodology based Optimization of diesel mustard Biodiesel – diethyl ether ternary blend ratios to improve engine performance', Journal of chemical and pharmaceutical research, Vol. 7, no. 8, pp. 249-256.
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CHAPTER 5 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Experimentally, kinematic viscosity for each blends of biodiesel with diesel fuel, benzene, kerosene, euro diesel summer and euro diesel winter was stated in addition the exactness of two separate equation are recommended in correlation and also to help predict the biodiesel properties in order to evaluate the result data. The following study, conclusions where been made:

Firstly, the property of biodiesel fuels, viscosity of blends of biodiesel with diesel fuel, benzene, kerosene, euro diesel summer, euro diesel winter, increase a corresponding increase in the biodiesel content. Using the response to surface methodology equation the prediction of these mixes gives a good and accurate result. RSM method has a great benefit and advantage is that it helps to reduce the number of experimental runs, which is important in other to provide an adequate and statistically satisfactory results. The mathematical equations developed to correlate the kinematic viscosity, of the five types of biodiesel mixtures have different forms.

Secondly, at room temperature, benzene evaporates during the experimental procedure at kinematic viscosity above 80 °C, with blends of biodiesel volume fraction content it's possible to obtain temperature upon 140 °C. For biodiesel + benzene and biodiesel + kerosene blends, the results are weaker and for benzene insignificant. this is because kerosene and benzene has a larger structural difference between the "pure" mechanisms of the systems, which governs different specific intermolecular interactions between the components affecting the properties of the mixtures.

5.2 Recommendation

Future works are also recommended, suggestions on the effect of pressure on the kinematic viscosity of biodiesel sample, and the investigation and experimental runs on the flash point, and cloud point of these five blends of different fuels with biodiesel samples should be carried out.

Secondly, the fuel properties of biodiesel vary from those of diesel fuels. This means that different engine performance and emissions will occur when biodiesel is used in diesel

engines. Therefore, more studies are needed about the fuel properties of biodiesels, diesel fuels and their blends before using in a diesel engine

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APPENDICES

APPENDIX 1
KINEMATIC VISCOSITY CONVERSION FACTORS

Multiply by			То			
From	Stoke	CentiStokes	m²/s	m²/h	ft ² /s	ft²/h
Stoke	1	100	1.00*10-4	3.60*10-1	1.076*10 ⁻³	3.875969
CentiStokes	0.01	1	1.00*10-6	3.60*10 ⁻³	1.08*10-5	0.03876
m^2/s	1.00*104	1.00*106	1	3.60*10 ³	1.08*101	3.88*10 ⁴
m^2/h	2.78	2.78*10 ²	2.78*10-4	1	2.99*10 ⁻³	1.08*10 ¹
ft²/s	929.0	9.29*104	9.29*10 ⁻²	3.34*10 ²	1	3.60*10 ³
ft²/h	0.258	25.8	2.58*10 ⁻⁵	9.28*10 ⁻²	2.78*10-4	1

APPENDIX 2 ASTM 446 – 07

Designation: D 445 - 09

P

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 mm²/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 appro-

priate 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

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.

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).

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 $\pm 0.02^{\circ}$ 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 $\pm 0.05^{\circ}$ C.
- 6.4 Temperature Measuring Device in the Range from 0 to $100^{\circ}C$ —Use either calibrated liquid-in-glass thermometers (Annex A2) of an accuracy after correction of $\pm 0.02^{\circ}C$ 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 $\pm 0.05^{\circ}$ 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 $\pm 0.1^{\circ}$ 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 ½ 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 $\pm 0.35\%$ of the certified value have been used. The data to support the limit of $\pm 0.35\%$ 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

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, mm ² /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%

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

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.

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 μ m 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 \pm 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 mm²/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, v_1 and v_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:

 $v_{1,2}$ = determined kinematic viscosity values for v_1 and v_2 , respectively, mm²/s,

C = calibration constant of the viscometer, mm²/s², 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/m³, at the same temperature used for the determination of the kinematic viscosity, and

 ν = kinematic viscosity, mm²/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°C5	0.0020 y	(0.20 %)
Formulated oils at 40 and 100°C6	0.0013 y	(0.13 %)
Formulated oils at 150°C7	0.015 y	(1.5 %)
Petroleum wax at 100°C8	0.0080 y	(0.80 %)
Residual fuel oils at 80 and 100°C9	0.011 (y + 8)	
Residual fuel oils at 50°C9	0.017 y	(1.7 %)
Additives at 100°C10	0.00106 y1.1	

Gas oils at 40°C11	0.0013 (y+1)	
Jet fuels at -20°C12	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°C5	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C6	0.0026 x	(0.26 %)
Formulated oils at 150°C7	0.0056 x	(0.56 %)
Petroleum wax at 100°C8	0.0141 x ^{1.2}	
Residual fuel oils at 80 and 100°C9	0.013(x + 8)	
Residual oils at 50°C9	0.015 x	(1.5 %)
Additives at 100°C10	0.00192 x1.1	
Gas oils at 40°C ¹¹	0.0043 (x+1)	
Jet fuels at -20°C12	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.

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 mm²/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 mm²/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 mm²/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 mm²/s at 50°C and from 5 to 170 mm²/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 mm²/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 mm²/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 mm²/s at 20°C and were first available in 1997.

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 mm²/s at 40°C and from 2 to 43 mm²/s at 100°C, and were first published in 1989. Request See Guide D 6074.



Base oils at 40 and 100°C5	0.0065 x	(0.65 %)
Formulated oils at 40 and 100°C6	0.0076 x	(0.76 %)
Formulated oils at 150°C7	0.018 x	(1.8 %)
Petroleum wax at 100°C8	0.0366 x1.2	
Residual fuel oils at 80 and 100°C9	0.04(x + 8)	
Residual oils at 50°C9	0.074 x	(7.4 %)
Additives at 100°C10	0.00862 x1.1	
Gas oils at 40°C11	0.0082 (x+1)	
Jet fuels at -20°C12	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

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, 4 mm ² /s			
A. Ostwald Type	es 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	0.5 to 100 000			
Ubbelohde dilution ^B (B)				
Cannon-Ubbelohde semi-micro	0.4 to 20 000			
C. Reverse-flow Types for	r 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.

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

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

D!#:	Approximate Kinematic Viscosity, mm ² /s							
Designation	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			

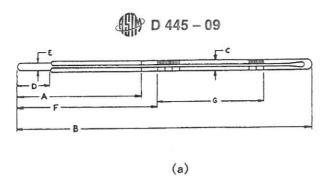
A2.2.2.1 The interval for ice-point recalibration shall be no longer than six months (see NIST GMP 11). For new thermometers, monthly checking for the first six months is recommended. A change of one or more scale divisions in the ice point means that the thermometer may have been overheated or damaged, 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.

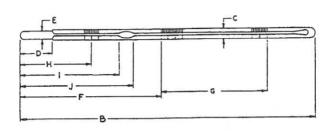
A2.2.2.2 Other thermometric devices, if used, will also require periodic recalibration. Keep records of all recalibration.

A2.2.3 Procedure for Ice-point Recalibration of Liquid-inglass 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





(b)

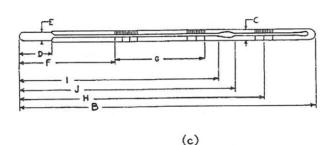


FIG. A2.1 Thermometer Designs

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 CHU Kauai, HI Ottawa, Canada 2.5, 5, 10, 15, MHz 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.

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Designation: D 446 - 07

P

Designation: 71/2/95

Standard Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers¹

This standard is issued under the fixed designation D 446; 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 These specifications cover operating instructions for glass capillary kinematic viscometers of all the types described in detail in Annex A1, Annex A2, and Annex A3 as follows:

Modified Ostwald viscometers, Annex A1 Suspended-level viscometers, Annex A2 Reverse-flow viscometers, Annex A3

- 1.2 The calibration of the viscometers is described in Section 6.
- 1.3 This standard covers some widely used viscometers suitable for use in accordance with Test Method D 445. Other viscometers of the glass capillary type which are capable of measuring kinematic viscosity within the limits of precision given in Test Method D 445 may be used.
- 1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

2. Referenced Documents

- 2.1 ASTM Standards:2
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D 2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards
- 2.2 ISO Documents:3
- ISO 3104 Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity

ISO 3105 Glass Capillary Kinematic Viscometers— Specifications and Operating Instructions

ISO 5725 Basic Methods for the Determination of Repeatability and Reproducibility of a Standard Measurement Method

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

ISO Guide 25 General Requirements for the Calibration and Testing Laboratories

2.3 NIST Standards:4

NIST 1297 Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results

3. Materials and Manufacture

- 3.1 Fully annealed, low-expansion borosilicate glass shall be used for the construction of all viscometers. The size number, serial number, and manufacturer's designation shall be permanently marked on each viscometer. All timing marks shall be etched and filled with an opaque color, or otherwise made a permanent part of the viscometer. See detailed description of each type of viscometer in Annex A1, Annex A2, and Annex A3.
- 3.2 With the exception of the FitzSimons and Atlantic viscometers, all viscometers are designed to fit through a 51-mm hole in the lid of a constant-temperature bath having a liquid depth of at least 280 mm; and it is assumed that the surface of the liquid will be not more than 45 mm from the top of the bath lid. For certain constant-temperature baths, especially at low or high temperatures, it may be necessary to construct the viscometers with the uppermost tubes longer than shown to ensure adequate immersion in the constant-temperature bath. Viscometers so modified can be used to measure kinematic viscosity within the precision of the test method. The lengths of tubes and bulbs on the figures should be held within $\pm 10\%$ or ± 10 mm, whichever is less, such that the calibration constant of the viscometer does not vary by more than $\pm 15\%$ from the nominal value.

¹ These specifications and operating instructions are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved Jan. 1, 2007. Published January 2007. Originally approved in 1966 as D 2515 – 66. Redesignated D 446 in 1977. Last previous edition approved in 2006 as D 446 – 06.

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.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

4. Nomenclature for Figures

4.1 The figures in the annexes contain letters to designate specific parts of each viscometer. These letters are also used in the text of the standard when reference to the viscometers is given. The more frequently used letters on the figures in the annexes are as follows:

Α	lower reservoir
В	suspended level
	bulb
C and J	timing bulbs
D	upper reservoir
E, F, and I	timing marks
G and H	filling marks
K	overflow tube
L	mounting tube
M	lower vent tube
N	upper vent tube
P	connecting tube
R	working capillary

5. Viscometer Holder and Alignment

5.1 All viscometers which have the upper meniscus directly above the lower meniscus (Cannon-Fenske routine in Annex A1 and all in Annex A2) shall be mounted in a constant temperature bath with tube L held within 1° of the vertical as observed with a plumb bob or other equally accurate inspection means. A number of commercially available holders are so designed that the tube L is held perpendicular to the lid of a constant-temperature bath; nevertheless, the viscometer should be tested with a plumb line in order to ensure that the tube L is in a vertical position.

5.1.1 Those viscometers whose upper meniscus is offset from directly above the lower meniscus (all others in Annex A1 and all in Annex A3) shall be mounted in a constant-temperature bath with tube L held within 0.3° of the vertical.

5.2 Round metal tops, designed to fit above a 51-mm hole in the lid of the bath, are frequently cemented on to the Zeitfuchs, Zeitfuchs cross-arm, and Lantz-Zeitfuchs viscometers which then are permanently mounted on the lid of the bath. Also a rectangular metal top, 25 mm × 59 mm, is often cemented on to the Zeitfuchs cross-arm and Zeitfuchs viscometers. Viscometers fitted with metal tops should also be set vertically in the constant-temperature bath with the aid of a plumb line.

5.3 In each figure, the numbers which follow the tube designation indicate the outside tube diameter in millimetres. It is important to maintain these diameters and the designated spacing to ensure that holders will be interchangeable.

6. Calibration of Viscometers

6.1 Procedures:

6.1.1 Calibrate the kinematic glass capillary viscometers covered by this standard using the procedures described in Annex A1, Annex A2, and Annex A3.

6.2 Reference Viscometers:

6.2.1 Select a clear petroleum oil, free from solid particles and possessing Newtonian flow characteristics, with a kinematic viscosity within the range of both the reference viscometer and the viscometer to be calibrated. The minimum flow time shall be greater than that specified in the appropriate table of the annex in both the reference viscometer and the viscometer which is to be calibrated in order that the kinetic energy correction (see 7.1 and 7.2) may be less than 0.2 %.

6.2.2 Select a calibrated viscometer of known viscometer constant C_1 . This viscometer may be a reference viscometer (driving head at least 400 mm) that has been calibrated by the step-up procedure using viscometers of successively larger capillary diameters, starting with distilled water as the basic kinematic viscosity standard or a routine viscometer of the same type that has been calibrated by comparison with a reference viscometer. See Test Method D 2162.

6.2.3 Mount the calibrated viscometer together with the viscometer to be calibrated in the same bath and determine the flow times of the oil in accordance with Test Method D 445.

6.2.3.1 The calibration of the reference viscometer should only be carried out by a reputable laboratory meeting the requirements of, for example, ISO Guide 25.

6.2.4 Calculate the viscometer constant C_1 as follows:

$$C_1 = (t_2 \times C_2)/t_1 \tag{1}$$

where:

 C_1 = the constant of the viscometer being calibrated,

 t_1 = the flow time to the nearest 0.1 s in the viscometer being calibrated,

 C_2 = the constant of the calibrated viscometer, and

 t_2 = the flow time to the nearest 0.1 s in the calibrated

6.2.5 Repeat 6.2.1-6.2.3 with a second oil whose flow times are at least 50 % longer than the first oil. If the two values of C_1 differ by less than 0.2 % for those viscometers listed in Annex A1 and Annex A2 and less than 0.3 % for those viscometers listed in Annex A3, use the average. If the constants differ by more than this value, repeat the procedure taking care to examine all possible sources of errors.

6.2.5.1 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 than 0.1 %, correct the calibration constant as follows:

$$C_2 = (g_2/g_1) \times C_1 \tag{2}$$

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

6.3 Certified Viscosity Reference Standards:

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

6.3.1.1 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.

6.3.2 Select from Table 1 a certified viscosity reference standard with a kinematic viscosity at the calibration temperature within the kinematic viscosity range of the viscometer to be calibrated and a minimum flow time greater than that specified in the appropriate table of the annex. Determine the flow time to the nearest 0.1 s in accordance with Test Method D 445 and calculate the viscometer constant, C, as follows:

$$C = \nu/t \tag{3}$$

TABLE 1 Certified Viscosity Reference Standards

			Approximate Kinema	tic Viscosity, mm ² /s		
Designation	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	(***)		
S30000		81 000	23 000	11 000		

where:

 ν = the kinematic viscosity, mm²/s, for the certified viscosity reference standard, and

t =the flow time, s.

6.3.3 Repeat with a second certified viscosity reference standard whose flow times are at least 50 % longer than the first certified viscosity reference standard. If the two values of C differ by less than 0.2 % for those viscometers listed in Annex A1 and Annex A2 and less than 0.3 % for those viscometers listed in Annex A3, use the average as the viscometer constant for the viscometer being calibrated. If the constants differ by more than this value, repeat the procedure taking care to examine all possible sources of errors.

6.4 Expression of Constant:

6.4.1 Report the constant to the nearest 0.1% of the determined value. This generally means four significant figures from $1\times 10^{\rm N}$ to $6.999\times 10^{\rm N}$ and three significant figures from $7\times 10^{\rm N}$ to $9.99\times 10^{\rm N}$.

7. Kinematic Viscosity Calculation

7.1 Basic Formula:

7.1.1 Kinematic viscosity, expressed in mm²/s, can be calculated from the viscometer dimensions as follows:

$$\nu = (10^6 \pi g D^4 H t / 128 V L) - E/t^2 \tag{4}$$

where:

 ν = the kinematic viscosity, mm²/s,

g = the acceleration due to gravity, m/s²,

D = the diameter of the capillary, m,

L = the length of the capillary, m,

H = the average distance between the upper and lower menisci, m,

V = the timed volume of liquids passing through the capillary, m^3 (approximately the volume of the timing bulb),

E = the kinetic energy factor, mm²·s, and

t =the flow time, s.

7.1.2 If the viscometer is selected so that the minimum flow time shown in the tables of Annex A1, Annex A2, and Annex A3 are exceeded, the kinetic energy term, E/t^2 , becomes insignificant and Eq 4 may be simplified by grouping the non-variable terms into a constant, C, as follows:

$$v = C \cdot t \tag{5}$$

7.2 Kinetic Energy Correction:

7.2.1 The viscometers described in the Annex A1, Annex A2, and Annex A3 are designed such that the kinetic energy

correction term, E/t^2 , is negligible if the flow time is more than 200 s. In the case of several sizes of viscometers for the measurement of low-kinematic viscosity liquids, a minimum flow time greater than 200 s is required in order that the kinetic energy correction term, E/t^2 , shall be negligible. The minimum flow times required are set out as footnotes to the appropriate tables of viscometer dimensions given in the Annex A1, Annex A2, and Annex A3.

7.2.2 For viscometers whose constants are 0.05 mm²/s² or less, a kinetic energy correction can be significant if the minimum 200 s flow is not observed. Where this is not possible, Eq 5 takes on the following form:

kinematic viscosity,
$$mm^2/s = Ct - E/t^2$$
 (6)

where:

 $E = \text{kinetic energy factor, } mm^2 \times s,$

 $C = \text{viscometer constant, } \text{mm}^2/\text{s}^2,$

t = flow time, s.

7.2.3 Although the kinetic energy factor, E, is not a constant, it may be approximated by means of the following equation:

$$E = 52.5 \ V^{3/2} / L (Cd)^{1/2} \tag{7}$$

where:

(using the units given in Figs. A1.1-A3.4)

V = volume of the timing bulb, mL,

L = capillary working length, mm,

d = capillary working diameter, mm,

 $C = \text{viscometer constant, } \text{mm}^2/\text{s}^2.$

Note 1—The kinetic energy factor for certain viscometer designs and flow time use can result in significant kinematic viscosity errors. Determine the effect of the kinetic energy factor for viscometers not described in this specification.

7.3 Maximum Flow Time:

7.3.1 The limit of 1000 s has been set arbitrarily for convenience as the recommended maximum flow time for the viscometers covered by this standard. Longer flow times may be used.

7.4 Surface Tension Correction:

7.4.1 If the two menisci have different average diameters during the flow time and if the surface tension of the sample differs substantially from the calibrating liquid, a surface tension correction is necessary. The changed C constant, C_2 , is given approximately as follows:

$$C_2 = C_1 [1 + (2/g h)(1/r_u - 1/r_l) \cdot (\gamma_1/\rho_1 - \gamma_2/\rho_2)]$$
 (8)

where:

g = the acceleration due to gravity, m/s²,

h = the average driving head, m,

 r_u = the average radius of the upper meniscus, m, r_t = the average radius of the lower meniscus, m,

 γ = the surface tension, N/m, and

 ρ = the density, in kg/m³.

Subscripts 1 and 2 relate to values with the calibrating liquid and the test portion, respectively.

7.4.2 While this correction applies to all viscometers, a number of viscometers are designed to minimize the surface tension correction. The greatest correction normally encountered is with a viscometer calibrated with water and used for oils. Generally, viscometers are calibrated and used with hydrocarbons whose surface tensions are close enough for these corrections to be insignificant.

7.5 Effect of Temperature:

7.5.1 The viscometer constant, C, is independent of temperature for all those viscometers which have the volume of sample adjusted at bath temperature and in the case of all suspended-level viscometers.

7.5.2 The following viscometers, which have a fixed volume charged at ambient temperature, have a viscometer constant, *C*, which varies with temperature: Cannon-Fenske routine, Pinkevitch, Cannon-Manning semi-micro, Cannon-Fenske opaque.

7.5.3 The following equation can be used to calculate the viscometer constant at temperatures other than the calibration temperature for the Cannon-Fenske routine, Pinkevitch, and Cannon-Manning semi-micro viscometers:

$$C_2 = C_1 [1 + (4000 V(\rho_2 - \rho_1))/\pi D^2 h \rho_2)]$$
 (9)

where:

 C_1 = the constant of the viscometer when filled and calibrated at the same temperature,

V = the volume of charge, mL,

D = the average diameter of the meniscus in the lower reservoir for the Cannon-Fenske routine, Pinkevitch, and Cannon-Manning semi-micro viscometers, and in the upper reservoir of the Cannon-Fenske opaque viscometer, mm,

h = the average driving head, mm,

 ρ_1 = the density of the test liquid at the filling temperature, kg/m³ × 10⁻³, and

 ρ_2 = the density of the test liquid at the test temperature, kg/m³ × 10⁻³.

7.5.4 The temperature dependence of C for the Cannon-Fenske opaque (reverse-flow) viscometer is given as follows:

$$C_2 = C_1 [1 - (4000 V(\rho_2 - \rho_1))/(\pi D^2 h \rho_2)]$$
 (10)

8. Keywords

8.1 kinematic viscosity; viscometer; viscosity

ANNEXES

(Mandatory Information)

A1. MODIFIED OSTWALD VISCOMETERS

A1.1 General

A1.1.1 The following viscometers of the modified Ostwald type for transparent liquids follow the basic design of the Ostwald viscometer, but are modified to ensure a constant volume test portion in the viscometer as described in A1.1.2 and A1.1.3.

A1.1.2 These viscometers are used for the measurement of the kinematic viscosity of transparent Newtonian liquids up to 20 000 mm²/s.

A1.1.3 For the modified Ostwald viscometers, detailed drawings, size designations, nominal constants, kinematic viscosity range, capillary diameter, and bulb volumes for each viscometer are shown in Figs. A1.1-A1.7.

A1.1.3.1 Constant volume at filling temperature:

- (1) Cannon-Fenske routine viscometer
- (2) Cannon-Manning semi-micro viscometer
- (3) Pinkevitch viscometer

A1.1.3.2 Constant volume at the test temperature:

- (1) Zeitfuchs viscometer⁵
- (2) SIL viscometer

(4) BS/U-tube miniature viscometer

A1.2 Operating Instructions

A1.2.1 A standard operating procedure applicable to all glass capillary kinematic viscometers is contained in Test Method D 445. Operating instructions for the modified Ostwald viscometers are outlined in A1.2.2-A1.2.7 with emphasis on procedures that are specific to this group of viscometers.

Note A1.1—ISO methods 3104 and 3105 correspond to Test Methods D 445 and D 446, respectively.

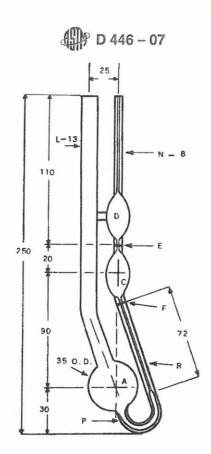
A1.2.2 Select a clean, dry calibrated viscometer which will give a flow-time greater than 200 s or the minimum shown in the table of dimensions, whichever is greater.

A1.2.3 Charge the viscometer in the manner dictated by the design of the instrument, the operation being in conformity with that employed when the unit 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.

Note A1.2—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.

⁽³⁾ BS/U-tube viscometer

 $^{^5\,}Zeitfuchs$ is a tradename of Cannon Instrument Co., P. O. Box 16, State College, PA 16804–0016.



Note 1-All dimensions are in millimetres.

Note 2—For size 25 only, the capillary N extends straight through bulbs D and C to about 10 mm below bulb C; the timing mark F encircles this capillary.

Size No. Appr	Approximate Constant,	Approximate Constant, Viscosity Range, Tul	Inside Diameter of Tube R, mm	Inside Diameter of Tubes	Bulb Volume, mL (±5 %)	
	(11111 75)/5	mm²/s	(±2 %)	N, E, and P, mm —	D	С
25	0.002	0.5 ^A to 2	0.30	2.6 to 3.0	3.1	1.6
50	0.004	0.8 to 4	0.44	2.6 to 3.0	3.1	3.1
75	0.008	1.6 to 8	0.54	2.6 to 3.2	3.1	3.1
100	0.015	3 to 15	0.63	2.8 to 3.6	3.1	3.1
150	0.035	7 to 35	0.78	2.8 to 3.6	3.1	3.1
200	0.1	20 to 100	1.01	2.8 to 3.6	3.1	3.1
300	0.25	50 to 250	1.27	2.8 to 3.6	3.1	3.1
350	0.5	100 to 500	1.52	3.0 to 3.8	3.1	3.1
400	1.2	240 to 1200	1.92	3.0 to 3.8	3.1	3.1
450	2.5	500 to 2500	2.35	3.5 to 4.2	3.1	3.1
500	8	1600 to 8000	3.20	3.7 to 4.2	3.1	3.1
600	20	4000 to 20 000	4.20	4.4 to 5.0	4.3	3.1

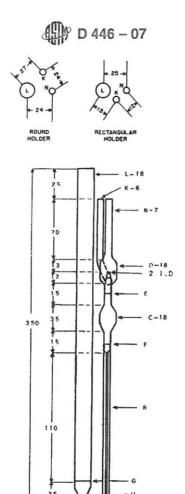
A 250-s minimum flow time; 200-s minimum flow time for all other units.

FIG. A1.1 Cannon-Fenske Routine Viscometer for Transparent Liquids

A1.2.3.1 To charge the Cannon-Fenske routine, Cannon-Manning semi-micro, and Pinkevitch viscometers, invert the viscometer and apply suction to tube L (the Pinkevitch viscometer has a side arm O to which vacuum is applied, with the finger on tube L being used to control the liquid flow) with tube N immersed in the liquid sample. Draw the sample to timing mark F for the Cannon-Fenske routine and Pinkevitch viscometers and to filling mark G for the Cannon-Manning semi-micro viscometer. Mount the viscometer upright in the constant-temperature bath keeping tube L vertical.

A1.2.3.2 Mount the Zeitfuchs viscometer in the constanttemperature bath, keeping tube L vertical. Pour sample through tube L to fill mark G. Allow 15 min for the sample to attain bath temperature and become free of air bubbles. Attach the vacuum line with stopcock and trap to tube K. Slowly draw the sample into timing bulb C by partially opening the stopcock in the vacuum line and partially closing tube N with the finger. Allow the excess liquid to flow into bulb D and through tube K into the trap in the vacuum line. When the liquid in tube L reaches a point 2 mm to 5 mm above filling mark H, hold it at this point by alternately closing the opening tube N to the atmosphere with the finger for the time in the Table A1.1 shown as follows to permit the sample to drain from the walls of tube L.

(1) Adjust the working volume by drawing the meniscus at the bottom of the column of the liquid exactly to filling mark H, making sure that the sample completely fills the viscometer between mark H and the tip of the overflow in bulb D; after this



Note-All dimensions are in millimetres.

Size No.	Approximate Constant, (mm²/s)/s	Kinematic Viscosity Range, ^A mm ² /s	Inside Diameter of Tube R, mm (±2 %)	Inside Diameter of Tubes P, E, and F, mm	Volume, Bulb C, ml (±5 %)
1	0.003	0.6 to 3	0.42	3.8 to 4.2	3.0
2	0.01	2 to 10	0.59	3.8 to 4.2	4.0
3	0.03	6 to 30	0.78	3.8 to 4.2	4.0
4	0.1	20 to 100	1.16	3.8 to 4.2	5.0
5	0.3	60 to 300	1.54	3.8 to 4.2	5.0
6	1.0	200 to 1000	2.08	3.8 to 4.2	5.0
7	3.0	600 to 3000	2.76	3.8 to 4.2	5.0

A 200-s minimum flow time for all units.

FIG. A1.2 Zeitfuchs Viscometer for Transparent Liquids

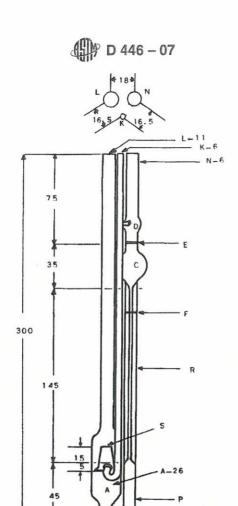
final adjustment of the working volume, remove the finger and close or remove the connection to the vacuum source. The final adjustment may be more conveniently made by disconnecting the vacuum and applying pressure to the mounting tube L by use of a rubber bulb.

A1.2.3.3 Charge the SIL viscometer by tilting it about 30° from the vertical, with bulb A below capillary R. Introduce enough of the sample into tube L for bulb A to fill completely and overflow into the gallery. Return the viscometer to the vertical position and mount it in the constant-temperature bath so that tube L is vertical. The quantity of sample charged should be such that the level in the lower reservoir is 3 mm to 14 mm above opening S. The sample will rise in capillary R

somewhat higher than opening S. After the temperature equilibrium has been reached, remove any excess sample from the gallery by suction applied to tube K.

A1.2.3.4 Mount the BS/U-tube or BS/U/M miniature viscometer in the constant-temperature bath keeping the tube L vertical. Using a long pipette to minimize any wetting of tube L above filling mark G, fill bulb A with a slight excess of the sample. After allowing the sample to attain the bath temperature, adjust the volume of the sample to bring the liquid level within 0.2 mm of filling mark G by withdrawing the sample with a pipette.

A1.2.4 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because this time



Note—All dimensions are in millimetres.

Size No.	Approximate Constant, (mm²/s)/s	Kinematic Viscosity Range, ^A mm ² /s	Inside Diameter of Tube <i>R</i> , mm (±2 %)	Inside Diameter of Tubes <i>E</i> and <i>P</i> , mm	Volume, Bulb <i>C</i> , mL (±5 %)
0C	0.003	0.6 to 3	0.41	4.5 to 5.5	3.0
1	0.01	2.0 to 10	0.61	4.5 to 5.5	4.0
1C	0.03	6 to 30	0.79	4.5 to 5.5	4.0
2	0.1	20 to 100	1.14	4.5 to 5.5	5.0
2C	0.3	60 to 300	1.50	4.5 to 5.5	5.0
3	1.0	200 to 1000	2.03	4.5 to 5.5	5.0
3C	3.0	600 to 3000	2.68	4.5 to 5.5	5.0
4	10.0	2000 to 10 000	3.61	4.5 to 5.5	5.0

A 200-s minimum flow time for all units.

FIG. A1.3 SIL Viscometer for Transparent Liquids

will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial (30 min should be sufficient except for the highest kinematic viscosities). One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

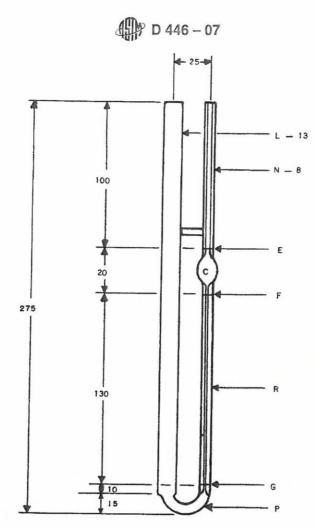
A1.2.5 Use vacuum (or pressure if the sample contains volatile constituents) to draw the sample through bulb C to about 5 mm above upper timing mark E. Release the vacuum, and allow the sample to flow by gravity.

A1.2.6 Measure, to the nearest 0.1 s, the time required for the leading edge of the meniscus to pass from timing mark E

to timing mark F. If this flow time is less than the minimum flow time specified for the viscometer, select a viscometer with a smaller diameter capillary and repeat steps A1.2.3-A1.2.6.

A1.2.7 Repeat steps A1.2.5 to A1.2.6 making a duplicate measurement of flow time. If the two measurements agree within the determinability given in Test Method D 445 for the product being measured, use the average for calculating kinematic viscosity.

A1.2.8 Clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by rinsing with a completely volatile solvent. Dry the viscometer by passing a slow stream of filtered, dry air through the viscometer for 2 min, or until the last trace of



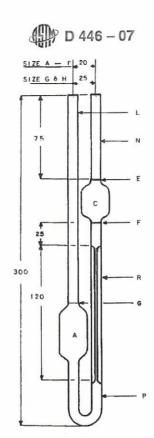
Note-All dimensions are in millimetres.

Size No.	Approximate		Inside Diameter of Tube	Inside Diamete	er of Tubes	Volume, Bulb
	Constant, (mm ² /s)/s	Viscosity Range, ^A mm ² /s	<i>R</i> , mm — (±2 %)	N and F, mm	P, mm	C, mL (±5 %)
25	0.002	0.4 to 2.0	0.22 ± 0.01	1.0 to 1.2	0.4 to 0.7	0.31
50	0.004	0.8 to 4	0.26 ± 0.01	1.0 to 1.2	0.5 to 0.8	0.31
75	0.008	1.6 to 8	0.31 ± 0.01	1.1 to 1.3	0.6 to 0.8	0.31
100	0.015	3 to 15	0.36 ± 0.02	1.2 to 1.4	0.7 to 0.9	0.31
150	0.035	7 to 35	0.47 ± 0.02	1.2 to 1.4	0.8 to 1.0	0.31
200	0.1	20 to 100	0.61 ± 0.02	1.4 to 1.7	0.9 to 1.2	0.31
300	0.25	50 to 250	0.76 ± 0.02	1.5 to 1.8	1.2 to 1.6	0.31
350	0.5	100 to 500	0.90 ± 0.03	1.8 to 2.2	1.5 to 1.8	0.31
400	1.2	240 to 1200	1.13 ± 0.03	2.0 to 2.4	1.6 to 2.0	0.31
450	2.5	500 to 2500	1.40 ± 0.04	2.2 to 2.6	2.0 to 2.5	0.31
500	8	1600 to 8000	1.85 ± 0.05	2.4 to 2.8	2.5 to 2.8	0.31
600	20	4000 to 20 000	2.35 ± 0.05	3.0 to 3.4	2.7 to 3.0	0.31

A 200-s minimum flow time for all units.

FIG. A1.4 Cannon-Manning Semi-Micro Viscometer for Transparent Liquids

solvent is removed. The use of alkaline cleaning solutions is not recommended as changes in the viscometer calibration may occur.

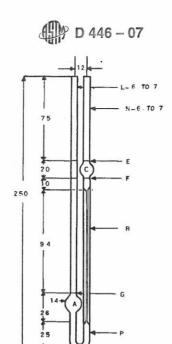


Note-All dimensions are in millimetres.

Size	Nominal Viscometer	Kinematic Viscosity	Inside Diameter Outside Diameter of Tubes ^A		Volume Bulb	Vertical Distance	Outside Diameter of Bulbs A and C,	
No. Constant, (mm²/s)/s		of Tube R, mm (±2 %)	L and P, mm	N, mm	C, mL (±5 %)	F to G, mm	mm	
A	0.003	0.9 ^B to 3	0.50	8 to 9	6 to 7	5.0	91 ± 4	21 to 23
B	0.00	2.0 to 10	0.71	8 to 9	6 to 7	5.0	87 ± 4	21 to 23
C	0.03	6 to 30	0.88	8 to 9	6 to 7	5.0	83 ± 4	21 to 23
2767		20 to 100	1.40	9 to 10	7 to 8	10.0	78 ± 4	25 to 27
D	0.1	60 to 300	2.00	9 to 10	7 to 8	10.0	73 ± 4	25 to 27
E	0.3		2.50	9 to 10	7 to 8	10.0	70 ± 4	25 to 27
F	1.0	200 to 1000				20.0	60 ± 3	32 to 35
G	3.0	600 to 3000	4.00	10 to 11	9 to 10			32 to 35
Н	10.0	2000 to 10 000	6.10	10 to 11	9 to 10	20.0	50 ± 3	32 10 33

FIG. A1.5 BS/U-Tube Viscometer for Transparent Liquids

^A Use 1 to 1.25 mm wall tubing for N, P, and L. ^B 300 s minimum flow time; 200 s minimum flow time for all other sizes.

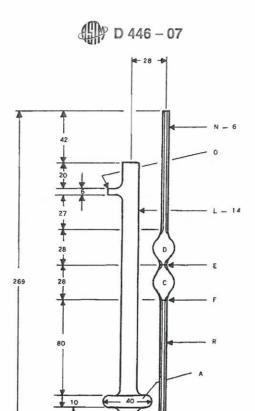


Note-All dimensions are in millimetres.

Size No.	Nominal Viscometer Constant, (mm ² /s)/s	Kinematic Viscosity Range, ^A mm ² /s	Inside Diameter of Tube R, mm (±2 %)	Outside Diameter of Tubes L, N, and P ^B , mm	Volume Bulb C, mL (±5 %)
M1	0.001	0.2 to 1	0.20	6 to 7	0.50
M2	0.005	1 to 5	0.30	6 to 7	0.50
M3	0.015	3 to 15	0.40	6 to 7	0.50
M4	0.04	8 to 40	0.50	6 to 7	0.50
M5	0.1	20 to 100	0.65	6 to 7	0.50

FIG. A1.6 BS/U/M Miniature Viscometer for Transparent Liquids

^A 200 s minimum flow time for all sizes. ^B Use 1 to 1.25 mm wall tubing for N, P, and L.



Note-All dimensions are in millimetres.

Size	Nominal Viscometer	Kinematic Viscosity	Inside Diameter of	Bulb Volu (±5	
No.	Constant, (mm²/s)/s	Range, ^A mm ² /s	Tube R, mm (±2 %)	D	С
0	0.0017	0.6 ^A to 1.7	0.40	3.7	3.7
1	0.0085	1.7 to 8.5	0.60	3.7	3.7
2	0.027	5.4 to 27	0.80	3.7	3.7
3	0.065	13 to 65	1.00	3.7	3.7
4	0.14	28 to 140	1.20	3.7	3.7
5	0.35	70 to 350	1.50	3.7	3.7
6	1.0	200 to 1000	2.00	3.7	3.7
7	2.6	520 to 2600	2.50	3.7	3.7
8	5.3	1060 to 5300	3.00	3.7	3.7
9	9.9	1980 to 9900	3.50	3.7	3.7
10	17	3400 to 17 000	4.00	3.7	3.7

^A 350 s minimum flow time: 200 s minimum flow time for all other sizes.

FIG. A1.7 Pinkevitch Viscometer for Transparent Liquids

TABLE A1.1 Drainage Time for Various Kinematic Viscosity Ranges in the Zeitfuchs Viscometer

Kinematic Viscosity of Sample,	Drainage Time,
mm²/s	S
Under 10	10 to 20
10 to 100	40 to 60
100 to 1000	100 to 120
Over 1000	180 to 200

A2. SUSPENDED LEVEL VISCOMETERS FOR TRANSPARENT LIQUIDS

A2.1 General

A2.1.1 The suspended level viscometers include the BS/IP/SL, BS/IP/SL(S), BS/IP/MSL, Ubbelohde, FitzSimons, Atlantic, Cannon-Ubbelohde, and Cannon-Ubbelohde semi-micro designs. The distinctive feature of suspended-level viscometers is that the liquid is suspended in the capillary which it fills completely. This suspension ensures a uniform driving head of liquid independent of the quantity of sample charged into the viscometer, making the viscometer constant independent of temperature. By making the diameter of the lower meniscus approximately equal to the average diameter of the upper meniscus, the surface tension correction is greatly reduced. Suspended-level viscometers are used for the measurement of the kinematic viscosities of transparent, Newtonian liquids up to 100 000 mm²/s.

A2.1.2 For the suspended-level viscometers, detailed drawings, size designations, nominal viscometer constants, kinematic viscosity range, capillary diameter and bulb volumes for each viscometer are shown on Figs. A2.1-A2.7.

A2.2 Operating Instructions

A2.2.1 A standard operating procedure, applicable to all glass capillary kinematic viscometers, is contained in Test Method D 445. Operating instructions for the suspended-level types are outlined in A2.2.2-A2.2.7 with emphasis on procedures that are specific to this group of viscometers.

Note A2.1—ISO methods 3104 and 3105 correspond to Test Methods D 445 and D 446, respectively.

A2.2.2 Select a clean, dry calibrated viscometer which will give a flow time greater than 200 s or the minimum shown in the table of dimensions, whichever is greater.

A2.2.3 Charge the sample into 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 Note A1.1).

A2.2.3.1 Charge the Ubbelohde and Cannon-Ubbelohde viscometers by tilting the instrument about 30° from the vertical and pouring sufficient sample through the L into bulb A so that when the viscometer is returned to the vertical the meniscus is between fill marks G and H, and tube P completely fills without entrapping air. Mount the viscometer in the constant-temperature bath keeping tube L vertical. To facilitate charging very viscous liquids, the viscometer may be inverted with tube L placed in the sample. Apply vacuum to tube N, closing tube M by a finger or rubber stopper; draw sufficient sample into tube L such that after wiping L clean and placing the viscometer in the constant-temperature bath, bulb A will fill as described above. The Cannon-Ubbelohde Semi-Micro design omits marks G and H since this viscometer is designed both for semi-micro and dilution use; pour sufficient sample through L into bulb A to ensure that capillary R and bulb C can be filled as described in A2.2.6.

A2.2.3.2 Charge the BS/IP/SL, BS/IP/SL(S), BS/IP/MSL, and FitzSimons viscometers through tube L with sufficient sample to fill bulb A, but not bulb B. The viscometer may be mounted vertically in the constant-temperature bath either prior to or following charging of the sample into the viscometer.

A2.2.3.3 Permanently mount the Atlantic viscometer in the constant-temperature bath with the enlargement S resting on the top-split collar, and the lower end of capillary tube R, 25 mm from the bottom of the bath. Pour the sample into a clean 50-mL beaker. Charge the viscometer by positioning the beaker and sample under tube L so that it will be completely immersed in the sample. Slowly apply vacuum to tube N by turning the three-way stopcock O to vacuum. Draw the sample into the viscometer filling capillary R, timing bulb C, and partially filling upper bulb D. Close stopcock O, holding the sample in the viscometer. If only a small sample is available, a short length of rubber-tipped glass tubing can be placed in the beaker with the rubber against the bottom of capillary tube R, and the sample drawn up as above.

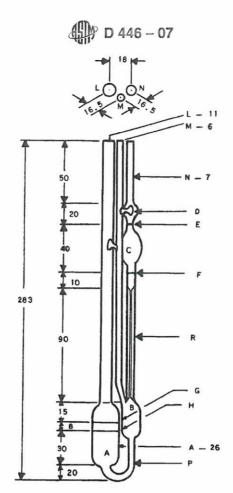
A2.2.4 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because this time will vary for different instruments, for different temperatures and for different kinematic viscosities, establish a safe equilibrium time by trial (30 min should be sufficient except for the highest kinematic viscosities). One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

A2.2.5 Except for the Atlantic viscometer which already has the sample in position, close tube M with the finger and use vacuum (or pressure, if the sample contains volatile constituents) to draw the sample slowly through bulb C to about 8 mm above upper timing mark E. Release vacuum from tube N and immediately place a finger from tube M to tube N, holding the meniscus above timing mark E until the lower meniscus has dropped below the end of capillary R in bulb B. Release finger and allow the sample to flow by gravity.

A2.2.6 Measure, to the nearest 0.1 s, the time required for the leading edge of the meniscus to pass from timing mark E to timing mark F. If this flow time is less than 200 s, select a smaller capillary viscometer and repeat A2.2.3-A2.2.6.

A2.2.7 Repeat steps A2.2.6 and A2.2.7 making a duplicate measurement of flow time. If the two measurements agree within the determinability given in Test Method D 445 for the product being measured, use the average for calculating kinematic viscosity.

A2.2.8 Clean viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by rinsing with a completely volatile solvent. Dry the viscometer by passing a slow stream of filtered, dry air through the viscometer for 2 min, or until the last trace of solvent is removed. The use of alkaline cleaning solutions is not recommended as changes in the viscometer calibration may occur.

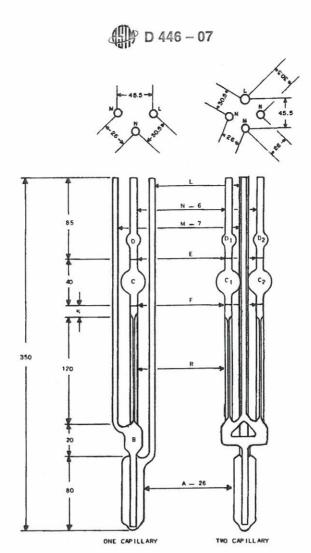


Note-All dimensions are in millimetres.

Size No.	Approximate Constant, (mm²/s)/s	Kinematic Viscosity Range, ^A mm²/s	Inside Diameter of Tube <i>R</i> , mm (±2 %)	Volume, Bulb <i>C</i> , mL (±5 %)	Inside Diameter of Tube P, mL (±5 %)
0	0.001	0.3 ^A to 1	0.24	1.0	6.0
0C	0.003	0.6 to 3	0.36	2.0	6.0
OB	0.005	1 to 5	0.46	3.0	6.0
1	0.01	2 to 10	0.58	4.0	6.0
1C	0.03	6 to 30	0.78	4.0	6.0
1B	0.05	10 to 50	0.88	4.0	6.0
2	0.1	20 to 100	1.03	4.0	6.0
2C	0.3	60 to 300	1.36	4.0	6.0
2B	0.5	100 to 500	1.55	4.0	6.0
3	1.0	200 to 1 000	1.83	4.0	6.0
3C	3.0	600 to 3 000	2.43	4.0	6.0
3B	5.0	1 000 to 5 000	2.75	4.0	6.5
4	10	2 000 to 10 000	3.27	4.0	7.0
4C	30	6 000 to 30 000	4.32	4.0	8.0
4B	50	10 000 to 50 000	5.20	5.0	8.5
5	100	20 000 to 100 000	6.25	5.0	10.0

A 300-s minimum flow time; 200-s minimum flow time for all other units.

FIG. A2.1 Ubbelohde Viscometer for Transparent Liquids

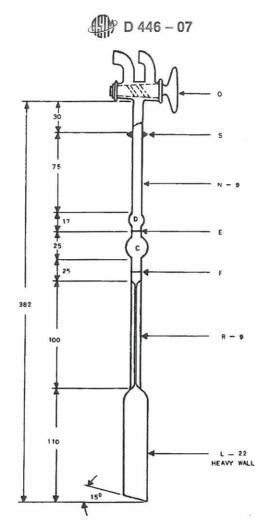


Note-All dimensions are in millimetres.

Size No.	Approximate Constant, (mm²/s)/s	Kinematic Viscosity Range, ^A mm ² /s	Inside Diameter of Tube <i>R</i> , mm (±2 %)	Volume, Bulb <i>C</i> , mL (±5 %)
	0.003	0.6 to 3.0	0.43	3.0
1		2 to 10	0.60	3.7
2	0.01		0.81	3.7
3	0.035	7 to 35		
1	0.10	20 to 100	1.05	3.7
-	0.25	50 to 250	1.32	3.7
5	1.20	240 to 1200	1.96	3.7

A 200-s minimum flow time for all units.

FIG. A2.2 FitzSimons Viscometer for Transparent Liquids

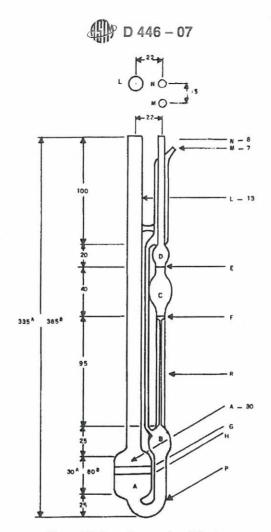


Note-All dimensions are in millimetres.

Size No.	Approximate Constant, (mm ² /s)/s	Kinematic Viscosity Range, mm²/s	Inside Diameter of Tube R , mm $(\pm 2 \%)$	Volume, Bulb <i>C</i> , mL (±5 %)
0C	0.003	0.7 ^A to 3	0.42	3.2
0B	0.005	1 to 5	0.46	3.2
1	0.01	2 to 10	0.56	3.2
1C	0.03	6 to 30	0.74	3.2
1B	0.05	10 to 50	0.83	3.2
2	0.1	20 to 100	1.00	3.2
2C	0.3	60 to 300	1.31	3.2
2B	0.5	100 to 500	1.48	3.2
3	1.0	200 to 1000	1.77	3.2
3C	3.0	600 to 3000	2.33	3.2
3B	5.0	1000 to 5000	2.64	3.2

A 250-s minimum flow time; 200-s minimum flow time for all other units.

FIG. A2.3 Atlantic Viscometer for Transparent Liquids

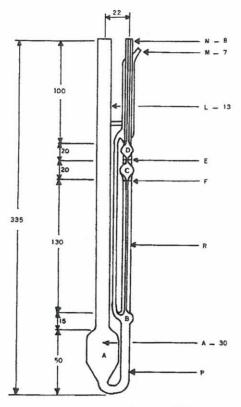


Note-All dimensions are in millimetres.

Size No.	Approximate Constant, (mm²/s)/s	Kinematic Viscosity Range, mm²/s	Inside Diameter of Tube R, mm (±2 %)	Volume, Bulb <i>C</i> , mL (±5 %)
25	0.002	0.5 ^A to 2	0.31	1.5
50	0.004	0.8 to 4.0	0.44	3.0
75	0.008	1.6 to 8.0	0.54	3.0
100	0.015	3 to 15	0.63	3.0
150	0.035	7 to 35	0.78	3.0
200	0.1	20 to 100	1.01	3.0
300	0.25	50 to 250	1.26	3.0
350	0.5	100 to 500	1.48	3.0
400	1.2	240 to 1200	1.88	3.0
450	2.5	500 to 2500	2.25	3.0
500	8	1600 to 8000	3.00	3.0
600	20	4000 to 20 000	3.75	3.0
650	45	9000 to 45 000	4.60	3.0
700	100	20 000 to 100 000	5.60	3.0

A 250-s minimum flow time; 200-s minimum flow time for all other units.

FIG. A2.4 Cannon-Ubbelohde (A) and Cannon-Ubbelohde Dilution (B) Viscometers for Transparent Liquids

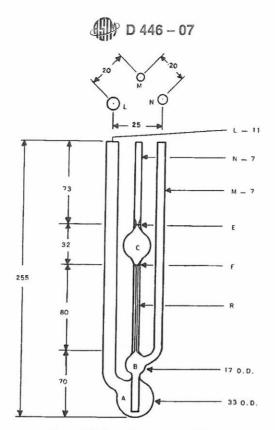


Note-All dimensions are in millimetres.

Size No.	Approximate Constant, (mm ² /s)/s	Kinematic Viscosity Range, ^A mm ² /s	Inside Diameter of Tube <i>R</i> , mm (±2 %)	Volume, Bulb <i>C</i> , mL (±5 %)	Inside Diameter o Tubes <i>N, E, F,</i> and <i>P</i> , mm
25	0.002	0.4 to 2.0	0.22	0.30	1.2 to 1.4
50	0.004	0.8 to 4	0.25	0.30	1.2 to 1.4
75	0.008	1.6 to 8	0.30	0.30	1.2 to 1.4
100	0.015	3 to 15	0.36	0.30	1.2 to 1.4
150	0.035	7 to 35	0.47	0.30	1.2 to 1.4
200	0.1	20 to 100	0.61	0.30	1.4 to 1.7
300	0.25	50 to 250	0.76	0.30	1.5 to 1.8
350	0.5	100 to 500	0.90	0.30	1.8 to 2.2
400	1.2	240 to 1200	1.13	0.30	2.1 to 2.5
450	2.5	500 to 2500	1.40	0.30	2.4 to 2.8
500	8	1600 to 8000	1.85	0.30	2.7 to 3.1
600	20	4000 to 20 000	2.35	0.30	3.7 to 4.0

A 200-s minimum flow time for all units.

FIG. A2.5 Cannon-Ubbelohde Semi-Micro Viscometer for Transparent Liquids



Note-All dimensions are in millimeters.

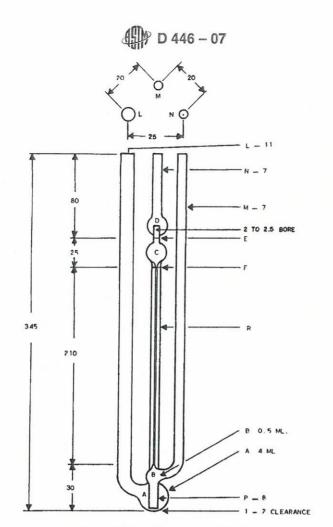
Size No.	Nominal Viscometer Constant, (mm ² /s)/s	Kinematic Viscosity Range, mm²/s	Inside Diameter of Tube R, mm (±2%)	Volume Bulb C, mL (±5%)	Inside Diameter of Tube N, mm	Inside Diameter of Tube at E, mm
1	0.0008	1.05 ^A min	0.36	5.6	2.8 to 3.2	3
2	0.003	2.1 ^B to 3	0.49	5.6	2.8 to 3.2	3
3	0.01	3.8 ^C to 10	0.66	5.6	2.8 to 3.2	3
4	0.03	6 to 30	0.87	5.6	2.8 to 3.2	3
5	0.1	20 to 100	1.18	5.6	2.8 to 3.2	3
6	0.3	60 to 300	1.55	5.6	2.8 to 3.2	3
7	1.0	200 to 1000	2.10	5.6	3.7 to 4.3	4
8	3.0	600 to 3000	2.76	5.6	4.6 to 5.4	5
9	10.0	2000 to 10 000	3.80	5.6	4.6 to 5.4	5

FIG. A2.6 BS/IP/SL(S) Viscometer for Transparent Liquids

A 1320 s minimum flow time;

B 600 s minimum flow time;

C 380 s minimum flow time; 200 s minimum flow time for all other sizes.



Note-All dimensions are in millimetres.

Size No.	ze Viscometer Viscosity Diameter o. Constant, Range, ^A Tube R, r		Inside Diameter of Tube R, mm (±2 %)	Volume Bulb C, mL (±5 %)	Inside Diameter of Tubes N and P, mm	
1	0.003	0.6 to 3	0.35	1.2	4 to 6	
2	0.01	2 to 10	0.45	1.2	4 to 6	
3	0.03	6 to 30	0.62	1.2	4 to 6	
4	0.1	20 to 100	0.81	1.2	4 to 6	
5	0.3	60 to 300	1.10	1.2	4 to 6	
6	1.0	200 to 1000	1.45	1.2	4 to 6	
7	3.0	600 to 3000	1.98	1.2	4 to 6	

A 200 s minimum flow time for all sizes.

FIG. A2.7 BS/IP/MSL Viscometer for Transparent Liquids

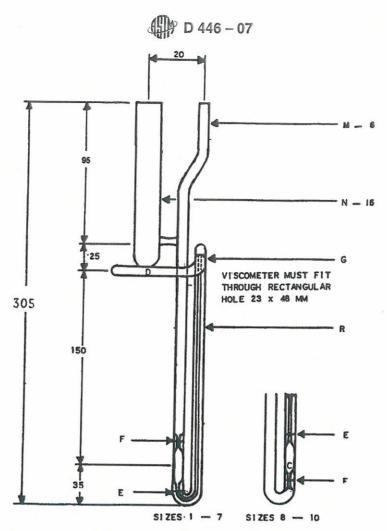
A3. REVERSE FLOW VISCOMETERS FOR TRANSPARENT AND OPAQUE LIQUIDS

A3.1 General

A3.1.1 The reverse-flow viscometers for transparent and opaque liquids include the Zeitfuchs cross-arm, Cannon-Fenske opaque, BS/IP/RF and Lantz-Zeitfuchs viscometers. Unlike the modified Ostwald and suspended-level viscometers, the sample of liquid flows into a timing bulb not previously wetted by sample, thus allowing the timing of liquids whose

thin films are opaque. Reverse-flow viscometers are used for the measurement of kinematic viscosities of opaque and transparent liquids up to 300 000 mm²/s.

A3.1.2 For the reverse-flow viscometers, detailed drawings, size designations, nominal viscometer constants, kinematic viscosity range, capillary diameter and bulb volumes for each viscometer are shown in Figs. A3.1-A3.4.



Note-All dimensions are in millimetres

Size No.	Approximate Constant, (mm ² /s)/s	Kinematic Viscosity Range, ^A mm²/s	Inside Diameter of Tube <i>R</i> , mm (±2 %)	Length of Tube <i>R</i> , mm	Lower Bulb Volume, mL (±5 %)	Horizontal Tube Diameter, mm (±5 %)
1	0.003	0.6 to 3	0.27	210	0.3	3.9
2	0.01	2 to 10	0.35	210	0.3	3.9
3	0.03	6 to 30	0.46	210	0.3	3.9
4	0.10	20 to 100	0.64	210	0.3	3.9
5	0.3	60 to 300	0.84	210	0.3	3.9
6	1.0	200 to 1000	1.15	210	0.3	4.3
7	3.0	600 to 3000	1.42	210	0.3	4.3
8	10.0	2000 to 10 000	1.93	165	0.25	4.3
9	30.0	6000 to 30 000	2.52	165	0.25	4.3
10	100.0	20 000 to 100 000	3.06	165	0.25	4.3

A 200-s minimum flow time for all units.

FIG. A3.1 Zeitfuchs Cross-Arm Viscometers for Transparent and Opaque Liquids

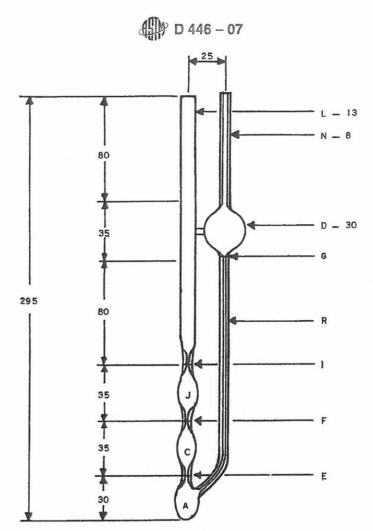
A3.2 Operation Instructions

A3.2.1 A standard operating procedure applicable to all glass capillary kinematic viscometers is contained in Test Method D 445. Operating instructions for the reverse-flow viscometers are outlined in A3.2.2-A3.2.7 with emphasis on procedures that are specific to a particular instrument or this group of instruments.

Note A3.1—ISO methods 3104 and 3105 correspond to Test Methods D 445 and D 446, respectively.

A3.2.2 Select a clean, dry calibrated viscometer which will give a flow time greater than 200~s and a kinetic energy correction of less than 0.2~%.

A3.2.3 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the unit was calibrated. If the sample is thought or known to contain fibers or solid particles, filter through a 75 μ m screen either prior to or during charging. See Note A1.1.



Note-All dimensions are in millimetres.

Size No.	Approximate Constant, (mm²/s)/s	Kinematic Viscosity Range, ^A mm ² /s	Inside Diameter of Tube <i>R</i> , mm (±2 %)	Inside Diameter of Tube N Tubes E, F, and I, mm (±5 %)	Volume, Bulbs A, C, and J, mL (±5 %)	Volume, Bulb <i>D</i> mL (±5 %)
25	0.002	0.4 to 2	0.31	3.0	1.6	11
50	0.004	0.8 to 4	0.42	3.0	2.1	11
75	0.008	1.6 to 8	0.54	3.0	2.1	11
100	0.015	3 to 15	0.63	3.2	2.1	11
150	0.035	7 to 35	0.78	3.2	2.1	11
200	0.1	20 to 100	1.02	3.2	2.1	11
300	0.25	50 to 200	1.26	3.4	2.1	11
350	0.5	100 to 500	1.48	3.4	2.1	11
400	1.2	240 to 1 200	1.88	3.4	2.1	11
450	2.5	500 to 2500	2.20	3.7	2.1	11
500	8	1600 to 8000	3.10	4.0	2.1	11
600	20	4000 to 20 000	4.00	4.7	2.1	13

A 200-s minimum flow time for all units.

FIG. A3.2 Cannon-Fenske Opaque Viscometer for Transparent and Opaque Liquids

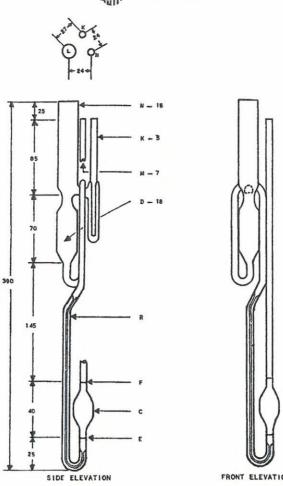
A3.2.3.1 To charge the Cannon-Fenske opaque viscometer, invert the viscometer and apply suction to the tube L, immersing tube N in the liquid sample. Draw liquid through tube N, filling bulb D to filling mark G. Wipe any excess sample off tube N and invert the viscometer to its normal position. Mount the viscometer in the constant temperature bath, keeping tube L vertical. Close tube N with a rubber stopper or a short length of rubber tube with a screw clamp.

A3.2.3.2 Mount the Zeitfuchs cross-arm viscometer in the constant-temperature bath, keeping tube N vertical. Introduce

the test specimen through tube N, taking care not to wet the sides of tube N, into the cross-arm D until the leading edge stands within 0.5 mm of fill mark G on the siphon tube. The volume of the test specimen is dependent on the location of the fill mark G. When the flow time of the lower meniscus is being measured between timing marks E and F (see A3.2.6), the upper meniscus shall be in the horizontal cross-arm D, thus making the location of fill mark G critical.

A3.2.3.3 Mount the Lantz-Zeitfuchs viscometer in the constant-temperature bath, keeping tube N vertical. Introduce





Note-All dimensions are in millimetres.

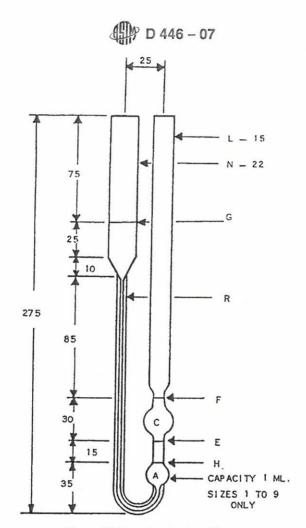
Size No.	Approximate Constant, (mm²/s)/s	Kinematic Viscosity Range, ^A mm²/s	Inside Diameter of Tube <i>R</i> , mm (±2 %)	Length of Tube <i>R</i> , mm	Volume, Bulb C mL (±5 %)
5	0.3	60 to 300	1.65	490	2.7
6	1.0	200 to 1000	2.25	490	2.7
7	3.0	600 to 3000	3.00	490	2.7
8	10.0	2000 to 10 000	4.10	490	2.7
9	30.0	6000 to 30 000	5.20	490	2.7
10	100.0	20 000 to 100 000	5.20	490	0.85

A 200-s minimum flow time for all units.

FIG. A3.3 Lantz-Zeitfuchs Viscometer for Transparent and Opaque Liquids

sufficient sample through tube N to completely fill bulb D, overflowing slightly into overflow tube K. If the sample is poured at a temperature above the test temperature, wait 15 min for the sample in the viscometer to attain bath temperature and add more sample to overflow slightly into tube K.

- A3.2.3.4 Mount the BS/IP/RF viscometer in the constanttemperature bath keeping the straight portion of the capillary tube R vertical by using a plumb-line observed in two directions at right angles, or as stated in the certificate of calibration.
- (1) Allow the viscometer to reach the bath temperature and then pour sufficient of the filtered test sample into the filling tube N to a point just below the filling mark G avoiding wetting the glass above G.
- (2) Allow the liquid to flow through the capillary tube R, taking care that the liquid column remains unbroken, until it reaches a position about 5 mm below the filling mark H and arrest its flow at this point by closing the timing tube L with a rubber bung. It is desirable that the rubber bung is fitted with a glass tube and stopcock so that one can apply a controllable, very slight excess pressure to tube L.
- (3) Add more liquid to the filling tube N to bring the oil surface to just below mark G. Allow the sample to reach the bath temperature and air bubbles to rise to the surface (at least 30 min is required).
- (4) Gently manipulate the stopcock or bung closing the tube L until the level of the liquid is arrested at mark H. The uppermost ring of contact of the sample with the glass should



Note-All dimensions are in millimetres.

Size No.	Nominal viscometer constant (mm²/s)/s	Kinematic viscosity range, ^A mm ² /s	Inside Diameter of tube <i>R</i> , mm (2 %)	Length of tube R, mm	Inside diameter at E, F, and H mm	Volume bulb 0 mL (±5 %)
1	0.003	0.6 to 3	0.51	185	3.0 to 3.3	4.0
2	0.01	2 to 10	0.71	185	3.0 to 3.3	4.0
3	0.03	6 to 30	0.93	185	3.0 to 3.3	4.0
4	0.1	20 to 100	1.26	185	3.0 to 3.3	4.0
5	0.3	60 to 300	1.64	185	3.0 to 3.3	4.0
6	1.0	200 to 1000	2.24	185	3.0 to 3.3	4.0
7	3.0	600 to 3000	2.93	185	3.3 to 3.6	4.0
8	10	2000 to 10 000	4.00	185	4.4 to 4.8	4.0
9	30	6000 to 30 000	5.5	185	6.0 to 6.7	4.0
10	100	20 000 to 100 000	7.70	210	7.70	4.0
11	300	60 000 to 300 000	10.00	210	10.00	4.0

A 200 s minimum flow time for all units.

FIG. A3.4 BS/IP/RF U-Tube Reverse Flow Viscometers for Opaque Liquids

coincide with the bottom of mark H. Add sample to tube N until the uppermost ring of its contact with tube N coincides with the bottom of mark G.

A3.2.4 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because this time will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial (30 min should be sufficient except for the highest kinematic viscosities). One bath is often used to

accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

A3.2.5 For the Cannon-Fenske opaque and BS/IP/RF viscometers, remove the stopper in tubes N and L, respectively, and allow the sample to flow by gravity. For the Zeitfuchs cross-arm viscometer, apply slight vacuum to tube M (or pressure to tube N) to cause the meniscus to move over the siphon tube, and about 30 mm below the level of tube D in

capillary R; gravity flow is thus initiated. For the Lantz-Zeitfuchs viscometer, apply slight vacuum to tube M (or pressure tube N with tube K closed) until the lower meniscus is opposite the lower timing mark E; allow the sample to flow by gravity.

A3.2.6 Measure to the nearest 0.1 s the time required for the uppermost ring of contact of the sample with the glass to rise from the bottom of timing mark E to the bottom of timing mark F. The lower filling mark H, as shown in Fig. A3.4 (BS/IP/RF U-tube) if applicable, must not be confused with the lower timing mark E. Do not use timing marks F and I and bulb J (as shown in Fig. A3.2) for determining viscosity of the sample. If the flow time is less than the minimum specified for the viscometer, select a clean, dry viscometer with a smaller diameter capillary and repeat steps A3.2.2-A3.2.6.

A3.2.7 Using this viscometer after it has been thoroughly cleaned and dried, or a second clean and dry viscometer, repeat

steps A3.2.3-A3.2.6 making a duplicate determination of the kinematic viscosity. If the two determinations agree within the determinability given in Test Method D 445 for the product being measured, report the average of the calculated kinematic viscosities. Note that the precision of the viscometers in Annex A3 is slightly poorer than those in Annex A1 and Annex A2 (see 6.3.3).

A3.2.8 Clean the viscometer thoroughly by several rinsings with the appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the viscometer by passing a slow stream of filtered, dry air through the viscometer for 2 min, or until the last trace of solvent is removed. The use of alkaline cleaning solutions is not recommended as changes in the viscometer calibration may occur.

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SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 446-06) that may impact the use of this standard. (Approved Jan. 1, 2007.)

(1) Revised 6.3 and Table 1.

(2) Removed Footnote 4.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 446–04) that may impact the use of this standard. (Approved May 15, 2006.)

(1) Revised A3.2.6.

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APPENDIX 3
EXPERIMENTAL DATA'S

· · · · · · · · · · · · · · · · · · ·	T	Ι	Kinematic	Kinematic	Kinematic	Kinematic	Kinematic
Temperature	Volume	Volume	Viscosity	Viscosity	Viscosity	Viscosity	Viscosity
(°C)	fraction	fraction	[mm²/s]	[mm ² /s]	[mm²/s]	[mm²/s]	[mm ² /s]
			BD + EDW	BD + K	BD + B	BD + DF	BD + EDS
20	1	0	5.5702	5.8899	5.9904	6.0269	6.0361
20	0.95	0.05	5.552	5.5976	5.8625	5.9995	5.9721
20	0.9	0.1	5.5246	5.3511	5.5885	5.9173	5.8533
20	0.85	0.15	5.5063	5.1319	4.8488	5.8442	5.7985
20	0.8	0.2	5.4789	4.9493	4.3463	5.762	5.7712
20	0.75	0.25	5.4607	4.3462	3.6794	5.689	5.5063
20	0.7	0.3	5.4515	3.981	3.4056	5.5885	5.4424
20	0.65	0.35	5.4424	3.6611	2.7044	5.4789	5.3876
20	0.6	0.4	5.4241	3.6066	2.1503	5.4241	5.3419
20	0.55	0.45	5.415	3.3782	1.956	5.3785	5.2872
20	0.5	0.5	5.4059	3.2047	1.7474	5.2963	5.2324
20	0.45	0.55	5.3602	3.0311	1.5444	5.103	5.2141
20	0.4	0.6	5.3237	2.8575	1.3323	4.9858	5.205
20	0.35	0.65	5.2872	2.6838	1.2321	4.9675	5.1958
20	0.3	0.7	5.2598	2.2909	1.084	4.9401	5.1593
20	0.25	0.75	5.2232	2.2267	0.9694	4.931	5.1228
20	0.2	0.8	5.1958	2.0346	0.8317	4.8945	5.1045
20	0.15	0.85	5.1319	1.9798	0.9694	4.794	5.0771
20	0.1	0.9	5.0771	1.8059	0.7199	4.7392	5.0497
20	0.05	0.95	5.0223	1.6441	0.6651	4.6844	5.0223
20	0	1	4.9767	1.6136	0.6043	4.6114	4.9949
30	1	0	4.7575	4.858	5.5702	5.0315	5.0315
30	0.95	0.05	4.6844	4.4193	4.9219	5.0041	4.9584
30	0.9	0.1	4.6844	4.2183	4.858	4.9219	4.8853
30	0.85	0.15	4.6297	3.961	4.4467	5.8442	4.8123
30	0.8	0.2	4.5015	3.9169	3.8346	4.7758	4.721
30	0.75	0.25	4.3919	3.5244	3.3965	4.6844	4.4741
30	0.7	0.3	4.3737	3.2595	2.9214	4.5655	4.4649
30	0.65	0.35	4.3188	3.1041	2.7203	4.5015	4.4192
30	0.6	0.4	4.2823	2.9854	2.1988	4.4467	4.3005
30	0.55	0.45	4.2457	2.7386	1.8045	4.3828	4.264
30	0.5	0.5	4.2275	2.6473	1.6359	4.3645	4.1909
30	0.45	0.55	4.2092	2.5377	1.4759	4.0904	4.1639
30	0.4	0.6	4.2	2.4097	1.2778	4.0721	4.1453
30	0.35	0.65	4.1909	2.2176	1.1411	4.063	4.1179
30	0.3	0.7	4.1179	1.9342	1.0467	4.0173	4.0995

			Kinematic	Kinematic	Kinematic	Kinematic	Kinematic
Temperature	Volume	Volume	Viscosity	Viscosity	Viscosity	Viscosity	Viscosity
(°C)	fraction	fraction	[mm²/s]	[mm²/s]	[mm²/s]	[mm²/s]	[mm²/s]
30	0.25	0.75	BD + EDW	BD + K	BD + B	BD + DF	BD + EDS
30	0.23		4.0904	1.8611	0.9294	3.9991	4.0721
30		0.8	4.0173	1.7172	0.8317	3.926	4.0539
	0.15	0.85	3.9808	1.6686	0.7256	3.8803	4.0265
30	0.1	0.9	3.926	1.5037	0.6736	3.8346	3.99
30	0.05	0.95	3.8986	1.4177	0.6479	3.789	3.9717
30	0	1	3.8803	1.3754	0.6015	3.7616	3.9352
40	1	0	3.926	3.926	4.0995	4.2183	4.2183
40	0.95	0.05	3.8803	3.5975	4.0173	4.1636	4.1818
40	0.9	0.1	3.8803	3.3508	3.5701	4.0128	4.0721
40	0.85	0.15	3.8255	3.2138	2.9946	3.9991	4.0082
40	0.8	0.2	3.7524	3.1955	2.5194	3.9352	3.8438
40	0.75	0.25	3.5518	2.8301	2.3001	3.8986	3.6976
40	0.7	0.3	3.5335	2.7021	2.1811	3.8164	3.6066
40	0.65	0.35	3.5153	2.5651	1.8017	3.7433	3.5518
40	0.6	0.4	3.497	2.501	1.6074	3.7159	3.4787
40	0.55	0.45	3.4787	2.2815	1.3896	3.6523	3.4239
40	0.5	0.5	3.4605	2.2267	1.2636	3.5975	3.3775
40	0.45	0.55	3.433	2.0989	1.1287	3.4331	3.3599
40	0.4	0.6	3.3691	1.9981	1.0153	3.4056	3.3325
40	0.35	0.65	3.3325	1.8428	0.9351	3.3782	3.3143
40	0.3	0.7	3.2047	1.6503	0.8317	3.3691	3.2777
40	0.25	0.75	3.1315	1.5599	0.7428	3.3325	3.241
40	0.2	0.8	3.1224	1.4762	0.6479	3.2869	3.2047
40	0.15	0.85	3.1133	1.4028	0.6186	3.2412	3.1863
40	0.1	0.9	3.1041	1.311	0.5758	3.2047	3.1681
40	0.05	0.95	3.095	1.2364	0.537	3.1315	3.1407
40	0	1	3.095	1.1726	0.4936	3.1041	3.1224
50	1	0	3.2686	3.2869	3.3965	3.4331	3.4331
50	0.95	0.05	3.1681	2.9579	3.2686	3.3691	3.3051
50	0.9	0.1	3.1315	2.8301	2.9671	3.2687	3.2047
50	0.85	0.15	3.0768	2.6199	2.3823	3.2504	3.1491
50	0.8	0.2	3.0128	2.6473	1.9707	3.1863	3.0861
50	0.75	0.25	2.9854	2.4005	1.8337	3.1589	2.9763
50	0.7	0.3	2.9671	2.2541	1.7602	3.1224	2.9488
50	0.65	0.35	2.9579	2.1811	1.4473	3.095	2.9214
50	0.6	0.4	2.9488	2.0898	1.3696	3.022	2.894
50	0.55	0.45	2.9214	1.9342	1.2493	2.9946	2.8392
50	0.5	0.5	2.9123	1.9159	1.1411	2.9488	2.7386
50	0.45	0.55	2.8758	1.7876	1.0353	2.9123	2.7203
50	0.4	0.6	2.7936	1.696	0.9323	2.8758	2.6838

Temperature (°C)	Volume fraction	Volume fraction	Kinematic Viscosity [mm²/s]	Kinematic Viscosity [mm²/s]	Kinematic Viscosity [mm²/s]	Kinematic Viscosity [mm²/s]	Kinemation Viscosity [mm²/s]
			BD + EDW	BD + K	BD + B	BD + DF	BD + EDS
50	0.35	0.65	2.7386	1.5588	0.8545	2.8575	2.6564
50	0.3	0.7	2.6473	1.4176	0.7742	2.821	2.6381
50	0.25	0.75	2.556	1.3229	0.6936	2.7662	2.6107
50	0.2	0.8	2.5468	1.2836	0.6158	2.7386	2.5925
50	0.15	0.85	2.5286	1.1818	0.5843	2.6838	2.556
50	0.1	0.9	2.5101	1.1452	0.537	2.6199	2.5377
50	0.05	0.95	2.4918	1.0895	0.5171	2.6016	2.5101
50	0	1	2.4645	1.0165	0.4557	2.556	2.4918
60	1	0	2.7386	2.7386	2.8027	2.8484	2.8484
60	0.95	0.05	2.6748	2.5101	2.7203	2.821	2.6473
60	0.9	0.1	2.629	2.4097	2.3823	2.7386	2.6107
60	0.85	0.15	2.5651	2.3092	1.9068	2.7112	2.556
60	0.8	0.2	2.5101	2.245	1.6777	2.6929	2.5101
60	0.75	0.25	2.4827	2.1179	1.5588	2.6473	2.4645
60	0.7	0.3	2.4645	1.9963	1.4763	2.6381	2.4371
60	0.65	0.35	2.4462	1.8241	1.2778	2.6107	2.3823
60	0.6	0.4	2.4188	1.7876	1.2493	2.5742	2.3275
60	0.55	0.45	2.4005	1.6686	1.1434	2.5286	2.2724
60	0.5	0.5	2.3731	1.641	1.061	2.4645	2.2176
60	0.45	0.55	2.3457	1.5493	0.9641	2.4371	2.2267
60	0.4	0.6	2.2909	1.4671	0.8721	2.3823	2.2176
60	0.35	0.65	2.245	1.3566	0.806	2.3549	2.2085
60	0.3	0.7	2.1446	1.2455	0.737	2.3001	2.1994
60	0.25	0.75	2.0989	1.149	0.6765	2.2724	2.1902
60	0.2	0.8	2.0898	1.1361	0.61	2.2541	2.172
60	0.15	0.85	2.0715	1.0439	0.5633	2.2359	2.1628
60	0.1	0.9	2.0715	1.026	0.5142	2.1902	2.1446
60	0.05	0.95	2.0624	0.9514	0.4993	2.1537	2.1263
60	0	1	2.0529	0.9042	0.4262	2.108	2.09
70	1	0	2.2531	2.1988	2.2588	2.3445	2.3445
70	0.95	0.05	2.176	2.1616	2.2474	2.3188	2.0874
70	0.9	0.1	2.1419	2.0559	1.9046	2.2531	2.0759
70	0.85	0.15	2.1216	2.0445	1.6845	2.2245	2.07702
70	0.8	0.2	2.0645	1.9417	1.4559	2.1959	2.0588
70	0.75	0.25	2.0388	1.8988	1.3868	2.1817	2.0531
70	0.7	0.3	2.0188	1.796	1.3123	2.1617	2.016
70	0.65	0.35	1.9988	1.5673	1.2005	2.1388	1.9902
70	0.6	0.4	1.9731	1.5159	1.1748	2.1273	1.9445
70	0.55	0.45	1.9502	1.4645	1.0897	2.1045	1.9217
70	0.5	0.5	1.9131	1.4068	1.0125	2.016	1.9103

Temperature	Volume	Volume	Kinematic Viscosity	Kinematic Viscosity	Kinematic Viscosity	Kinematic Viscosity	Kinema Viscosi
(°C)	fraction	fraction	[mm²/s]	[mm ² /s]	[mm ² /s]	[mm ² /s]	[mm²/s
			BD + EDW	BD + K	BD + B	BD + DF	BD + ED
70	0.45	0.55	1.8988	1.3352	0.9263	1.9788	1.884
70	0.4	0.6	1.876	1.2578	0.8488	1.9502	1.878
70	0.35	0.65	1.8589	1.1776	0.7889	1.9131	1.864
70	0.3	0.7	1.8359	1.1012	0.7256	1.8874	1.853
70	0.25	0.75	1.8102	0.9979	0.6651	1.8646	1.8359
70	0.2	0.8	1.7988	0.9837	0.6043	1.8416	1.827
70	0.15	0.85	1.7845	0.9149	0.5462	1.8245	1.8188
70	0.1	0.9	1.7788	0.9149	0.5021	1.7845	1.8074
70	0.05	0.95	1.7645	0.8346	0.4936	1.7588	1.7902
70	0	1	1.7445	0.8117	0.3994	1.7016	1.7731
80	1	0	1.8846	1.9502	1.8988	2.0931	2.0931
80	0.95	0.05	1.8502	1.8646	1.8874	2.0474	1.8931
80	0.9	0.1	1.8188	1.859	1.5159	1.9902	1.876
80	0.85	0.15	1.7988	1.7959	1.5073	1.9502	1.8445
80	0.8	0.2	1.7931	1.713	1.3009	1.9445	1.8216
80	0.75	0.25	1.7817	1.6673	1.2436	1.9131	1.7502
80	0.7	0.3	1.7674	1.6074	1.1691	1.8846	1.7645
80	0.65	0.35	1.7331	1.3696	1.1411	1.8589	1.7102
80	0.6	0.4	1.7073	1.3466	1.1154	1.8416	1.673
80	0.55	0.45	1.6844	1.255	1.041	1.8045	1.6531
80	0.5	0.5	1.6702	1.1862	0.9751	1.7903	1.6359
80	0.45	0.55	1.6531	1.1411	0.9006	1.713	1.6216
80	0.4	0.6	1.6188	1.0693	0.8403	1.6702	1.6131
80	0.35	0.65	1.5788	1.0182	0.7799	1.6302	1.5988
80	0.3	0.7	1.5559	0.9637	0.7199	1.6159	1.5817
80	0.25	0.75	1.5216	0.9035	0.6565	1.6016	1.5616
80	0.2	0.8	1.513	0.8835	0.5958	1.5701	1.553
80	0.15	0.85	1.5073	0.826	0.537	1.5502	1.5444
80	0.1	0.9	1.5016	0.8146	0.4964	1.5159	1.5387
80	0.05	0.95	1.4902	0.7542	0.4907	1.4845	1.5244
80	0	1	1.4816	0.7342	0.3781	1.4302	1.4873
90	1	0	1.713	1.7931	1.7274	1.9188	1.9188
90	0.95	0.05	1.6816	1.716	1.6331	1.8817	1.7931
90	0.9	0.1	1.6702	1.6702	1.5159	1.8274	1.7588
90	0.85	0.15	1.6131	1.6102	1.3466	1.7931	1.713
90	0.8	0.2	1.6273	1.56	1.1976	1.7788	1.6759
90	0.75	0.25	1.6216	1.5302	1.1297	1.7445	1.6273
90	0.7	0.3	1.5931	1.4267	1.1097	1.693	1.6102
90	0.65	0.35	1.5673	1.2264	1.0869	1.6502	1.5875
90	0.6	0.4	1.5359	1.2119	1.061	1.6216	1.5616

Temperature	Volume	Volume	Kinematic Viscosity	Kinematic Viscosity	Kinematic Viscosity	Kinematic Viscosity	Kinemation Viscosity
(°C)	fraction	fraction	[mm ² /s]	[mm ² /s]	[mm ² /s]	[mm ² /s]	[mm²/s]
			BD + EDW	BD + K	BD + B	BD + DF	BD + EDS
90	0.55	0.45	1.4959	1.1183	1.0006	1.5874	1.5187
90	0.5	0.5	1.4788	1.041	0.9408	1.5616	1.4788
90	0.45	0.55	1.4645	0.9951	0.8806	1.5359	1.4702
90	0.4	0.6	1.4267	0.9437	0.8317	1.5102	1.4588
90	0.35	0.65	1.421	0.9323	0.7713	1.4702	1.4559
90	0.3	0.7	1.3868	0.8545	0.7108	1.4359	1.4445
90	0.25	0.75	1.381	0.8346	0.7100	1.3982	1.4267
90	0.2	0.8	1.3696	0.8231		1.3896	1.44039
90	0.15	0.85	1.3639	0.7628		1.3582	1.3868
90	0.1	0.9	1.3466	0.7428		1.3209	1.3725
90	0.05	0.95	1.3352	0.6822		1.2866	1.3523
90	0	1	1.3352	0.665		1.2293	1.338
100	1	0	1.593	1.6416	1.6045	1.8131	1.8131
100	0.95	0.05	1.5416	1.5902	1.513	1.7702	1.6816
100	0.9	0.1	1.5302	1.493	1.4302	1.693	1.6245
100	0.85	0.15	1.5016	1.4302	1.2293	1.6673	1.5902
100	0.8	0.2	1.513	1.4096	1.124	1.6445	1.5673
100	0.75	0.25	1.5045	1.3953	1.104	1.6131	1.5102
100	0.7	0.3	1.4845	1.2835	1.0667	1.5931	1.4845
100	0.65	0.35	1.4645	1.104	1.0382	1.5673	1.4267
100	0.6	0.4	1.4153	1.0639	1.0239	1.533	1.3982
100	0.55	0.45	1.3896	1.0039	0.9665	1.493	1.381
100	0.5	0.5	1.3696	0.938	0.9206	1.4359	1.3582
100	0.45	0.55	1.3409	0.9035	0.8721	1.381	
100	0.4	0.6	1.3209	0.846	0.8231		1.3209
100	0.35	0.65	1.2981	0.8374	0.7685	1.3696 1.338	1.2866
100	0.3	0.7	1.2835	0.7685	0.7083	1.2923	1.2807
100	0.25	0.75	1.2607	0.766		1.2578	1.2407
100	0.2	0.8	1.2493	0.7513		1.235	1.2835 1.2636
100	0.15	0.85	1.2321	0.7142		1.2033	1.255
100	0.1	0.9	1.2264	0.6822		1.1326	1.2493
100	0.05	0.95	1.2062	0.6243		1.104	1.2293
100	0	1	1.1976	0.5958		1.0639	1.2091
110	1	0	1.4959	1.5302	1.4845	1.7388	1.7388
110	0.95	0.05	1.4125	1.4845	1.4616	1.6816	1.6502
110	0.9	0.1	1.3982	1.4039	1.3896	1.6102	1.6131
110	0.85	0.15	1.3696	1.318	1.1662	1.5874	1.5444
110	0.8	0.2	1.381	1.2835	1.1097	1.5701	1.4267
110	0.75	0.25	1.3725	1.2721	1.0783	1.553	1.4182
110	0.7	0.3	1.3668	1.1662	1.041	1.5302	1.3953

Temperature	Volume	Volume	Kinematic Viscosity	Kinematic Viscosity	Kinematic	Kinematic	
(°C)	fraction	fraction	[mm ² /s]	[mm ² /s]	Viscosity [mm²/s]	Viscosity [mm²/s]	Viscosity [mm ² /s]
			BD + EDW	BD + K	BD + B	BD + DF	BD + EDS
110	0.65	0.35	1.3495	1.0324	1.0239	1.4987	1.3437
110	0.6	0.4	1.3152	0.9637	1.0182	1.4616	1.3266
110	0.55	0.45	1.2923	0.9323	0.9665	1.4182	1.2866
110	0.5	0.5	1.2721	0.8606	0.9149	1.3582	1.255
110	0.45	0.55	1.2264	0.8346	0.8635	1.3409	1.2407
110	0.4	0.6	1.2119	0.777	0.8033	1.3209	
110	0.35	0.65	1.1976	0.7827	0.0174	1.2835	1.2264 1.2179
110	0.3	0.7	1.1776	0.7108		1.2293	1.2005
110	0.25	0.75	1.1605	0.7108		1.1834	1.1776
110	0.2	0.8	1.1411	0.6908		1.1411	1.1434
110	0.15	0.85	1.1212	0.6565		1.0869	1.1297
110	0.1	0.9	1.1069	0.6215		1.0467	1.1212
110	0.05	0.95	1.0983	0.5633		1.0239	1.1126
110	0	1	1.084	0.5404		0.9922	1.0983
120	1	0	1.3953	1.4153	1.3982	1.6759	1.673
120	0.95	0.05	1.318	1.3868	1.3982	1.6131	1.5502
120	0.9	0.1	1.3123	1.3209	1.3466	1.5788	1.533
120	0.85	0.15	1.2664	1.2521	1.1354	1.5359	1.5102
120	0.8	0.2	1.2731	1.2407	1.104	1.5149	1.3982
120	0.75	0.25	1.2778	1.2033	1.0696	1.4702	1.4182
120	0.7	0.3	1.2866	1.1154	1.0267	1.453	1.3352
120	0.65	0.35	1.2636	0.9722	1.0153	1.4153	1.3266
120	0.6	0.4	1.2493	0.9294	1.0125	1.381	1.2923
120	0.55	0.45	1.2062	0.8692	0.9608	1.3495	1.2378
120	0.5	0.5	1.1891	0.8203	0.9092	1.3152	1.1776
120	0.45	0.55	1.1411	0.7946	0.8545	1.2835	1.1605
120	0.4	0.6	1.1297	0.7456	0.8146	1.255	1.1411
120	0.35	0.65	1.0983	0.7399		1.2293	1.124
120	0.3	0.7	1.0783	0.6794		1.2062	1.1126
120	0.25	0.75	1.0639	0.6651		1.1354	1.1012
120	0.2	0.8	1.0496	0.6479		1.0926	1.084
120	0.15	0.85	1.041	0.605		1.0696	1.0812
120	0.1	0.9	1.0353	0.5815		1.0296	1.0583
120	0.05	0.95	1.0267	0.5285		0.9951	1.044
120	0	1	1.021	0.5021		0.9694	1.0267
130	1	0	1.3295	1.3982	1.3352	1.5701	1.5788
130	0.95	0.05	1.2407	1.3323	1.338	1.5359	1.533
130	0.9	0.1	1.2264	1.2578	1.3266	1.5073	1.4873
130	0.85	0.15	1.2179	1.2436	1.0874	1.453	1.4616
130	0.8	0.2	1.1976	1.2005	1.0954	1.4239	1.3725

Т	Volume	Valuma	Kinematic	Kinematic	Kinematic	Kinematic	Kinemation Viscosity
Temperature (°C)	Volume fraction	Volume fraction	Viscosity [mm²/s]	Viscosity [mm²/s]	Viscosity [mm²/s]	Viscosity [mm²/s]	[mm²/s]
(C)	Haction	Haction	BD + EDW	BD + K	BD + B	BD + DF	BD + EDS
130	0.75	0.25	1.1805	1.1919	1.0639	1.4039	1.2835
130	0.7	0.3	1.1719	1.0726	1.021	1.3868	1.2636
130	0.65	0.35	1.1662	0.9437	1.0125	1.3582	1.235
130	0.6	0.4	1.1548	0.8978	1.0067	1.3352	1.2005
130	0.55	0.45	1.1491	0.806	0.9551	1.2835	1.1411
130	0.5	0.5	1.1212	0.777	0.9035	1.2464	1.1297
130	0.45	0.55	1.0983	0.7428	0.8545	1.2264	1.1212
130	0.4	0.6	1.084	0.7079	0.8031	1.2005	1.1126
130	0.35	0.65	1.0783	0.7079	0.8031	1.1776	1.0869
130	0.33	0.03	1.0783	0.6386		1.1776	1.0726
130	0.25	0.75	1.0353	0.6243		1.1334	1.0553
130	0.23	0.73		0.6243		1.0926	1.0353
130	0.2	0.85	1.0296	0.5661			1.0324
130	0.13	0.83	1.0182			1.0353	*
130	0.05	0.95	0.9979	0.5462		1.0125	1.0153
130		1	0.9951	0.5079		0.9437	1.001
140	0		0.978	0.4785	1 2020	0.9351	0.9894
140	_	0	1.2835	1.3696	1.3038	1.5187	1.5187
140	0.95	0.05	1.2233	1.3038	1.3038	1.4987	1.5016
140	0.9	0.1	1.1993	1.2191	1.2807	1.4816	1.4616
140	0.85	0.15	1.1834	1.2264	1.0812	1.4096	1.4014
140	0.8	0.2	1.1662	1.1834	1.084	1.381	1.3409
140	0.75	0.25	1.1269	1.1548	1.0524	1.3696	1.2493
	0.7	0.3	1.1212	1.0667	1.0153	1.3495	1.2264
140	0.65	0.35	1.1126	0.938	1.0067	1.3266	1.2005
140	0.6	0.4	1.1012	0.8892	0.9979	1.3095	1.1434
140	0.55	0.45	1.0983	0.7946	0.9494	1.2578	1.1354
140	0.5	0.5	1.0696	0.7656	0.9035	1.2091	1.1097
140	0.45	0.55	1.061	0.7342	0.8488	1.1748	1.0926
140	0.4	0.6	1.0553	0.6965	0.7974	1.1411	1.084
140	0.35	0.65	1.0496	0.6965		1.1326	1.0726
140	0.3	0.7	1.0353	0.6272		1.1126	1.0526
140	0.25	0.75	1.0153	0.6072		1.0726	1.041
140	0.2	0.8	1.001	0.5872		1.0553	1.021
140	0.15	0.85	0.9922	0.5433		1.0239	1.0096
140	0.1	0.9	0.9837	0.5313		0.9951	1.001
140	0.05	0.95	0.978	0.4936		0.93511	0.9894
140	0	1	0.9694	0.4528		0.9263	0.9779