AN INVESTIGATION OF THE BIODIESEL AGEING EFFECTS ON BIODIESEL BLEND PROPERTIES

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

By
ELÇİN ÖZGENÇ

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

NICOSIA, 2018

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Approval of Director of Graduate School of
Applied Sciences

Prof. Dr. Nadire ÇAVUŞ

We certify this thesis is satisfactory for the award of the degree of Master of Science in Mechanical Engineering

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I hereby declare that, all the information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

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Dedicated to humanity …

ABSTRACT

The reduction of non-renewable energy sources has led the people to develop alternative energy sources. The reason for the increase in energy demand is the development of technology, industrialization and population growth. Biodiesel is one of the most important alternative energy sources. This thesis provided a brief introduction on biodiesel and its properties, then, the methodology followed in the thesis was presented. The biodiesel fuel used in this study was derived from waste frying oils. The purpose of this study is to examine the effects of ageing, biodiesel blend with euro-diesel and the storage of the sample at 40 degrees Celsius constant temperature on biodiesel properties by testing kinematic viscosity and density at different temperatures. In addition, pour point, cloud point, total acid number and oxidation stability were examined. The total acid number and oxidation stability parameters were analysed in a certified petrochemical laboratory in Southern Cyprus. The tests were carried out between 50 and 90 degrees Celsius according to ASTM and EN standards also considering the storage period. As a result, kinematic viscosity and density were observed to be decreasing with temperature increase. During the storage period, a decrease in total acid number and an increase in oxidation stability was also observed. Another purpose of this study is to increase the use of biodiesel for a cleaner environment. Biodiesel does not contain toxic gases such as carbon dioxide, carbon monoxide, hydrocarbons and does not harm the environment. Exhaust gas is less toxic.

Keyword: Biodiesel; kinematic viscosity; density; storage period; pour point; cloud point

ÖZET

Yenilenemez enerji kaynaklarının azalması, insanlığı alternatif enerji kaynaklarını geliştirmeye yöneltmiştir. Teknolojinin gelişmesi, sanayileşme ve nüfus artışı sebebiyle enerji talebi artmıştır. Önemi gün geçtikçe artan alternatif enerji kaynakları arasındaki en önemlilerinden biri biyodizeldir. Bu çalışmada kullanılan biyodizel yakıtı atık kızartma yağlarından elde edilmiştir. Bu çalışmanın amacı, biyodizel yaşlandırma etkilerinin biyodizel karışım özellikleri üzerine incelenmesidir. 40 ^{o}C sabit sıcaklıktaki depolanan biyodizel karışımı numunesinin (B80) kinematik viskozite ve yoğunluğunu farklı sıcaklıklarda test ederek numune üzerindeki etkisini araştırmaktır. Buna ek olarak, akma noktası, bulutlanma noktası, toplam asit sayısı, oksidasyon kararlılığı da incelenmiştir. Bu çalışmada ayrı olarak, toplam asit sayısı ve oksidasyon kararlılığı parametreleri Güney Kıbrıs'taki sertifikalı bir petrokimya laboratuvarında analiz edilmiştir. Yapılan deneyler 5 ^{o}C ile 90 ^{o}C arasında ASTM ve EN standartlarına göre depolama süresi dikkate alınarak ölçülmüştür. Elde edilen sonuçlar neticesinde kinematik viskozite ve yoğunluğun, sıcaklığın artmasıyla azaldığı gözlenmiştir. Depolama süresi boyunca toplam asit sayısında azalma, oksidasyon kararlılığında artış gözlenmiştir. Bu çalışmanın diğer bir amacı ise, daha temiz bir çevre için biyodizel kullanımını arttırmaktır. Biyodizel karbondioksit, karbonmonoksit, hidrokarbon gibi zehirli gazlar içermez ve doğaya zarar vermez. Egzoz gazı daha az zehirleyici olur.

Anahtar Kelimeler: Biyodizel; depolama süresi; kinematik viskozite; yoğunluk;
akma noktası; bulut noktası

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LIST OF SYMBOLS USED

A	Area, m^2
D	Capillary diameter, µm
dv	Changing in velocity, m/s^2
dx	Changing in separation height
F	Force, Newton
g	Acceleration due to gravity, m/s^2
G	Universal gas constant, J/mol
h	Planck's constant, J/s
H	Capillary height, mm
K	Viscometer constant,$(mm/s^2)/s$
L	Length of viscometer, mm
P	Flow pressure, Pa
Q	Flow rate, m^3/s
R	Capillary radius, m
R	Radian length, rad
T	Time, min
T	Absolute temperature, Kelvin
V	Volume, m^3
V	Velocity, m/s
v	Flow velocity, gal/min
v_z	Velocity in flow direction
v_r	Velocity in radian direction
v_θ	Velocity in angular direction
X	Elemental length
Y	Correction factor.
Z	Length in flow direction, m
Θ	Angular length, m
ρ	Density, kg/m^3
μ	Dynamic viscosity, kg/ms

v	Kinematic viscosity, m^2/s
τ	Shear stress, N/m^2
$\dot{\gamma}$	Rate of shear, s^{-1}

ABBREVIATIONS USED

AN	Acid Number
ANP	Agencia Nacional do Petroleo
ASTM	American Society for Testing Materials
B100	Biodiesel sample with %100 concentration
CFPP	Cold filter plugging point
CIE	Compressor ignition engines
CN	Cetane number
CP	Cloud Point
EU	European union
FA	Fatty acid
FAME	Fatty acid methyl ester
FFA	Free fatty acid
FP	Flash Point
HHV	Higher heating value
IV	Iodine value
KOH	Potassium Hydroxide
PP	Pour point
US	United States
WCO	Waste cooking oil
WFCE	World Fuel Charter Committee

CHAPTER 1

INTRODUCTION

1.1 World Energy Consumption

Increase in renewable energy resources and their use in transportation systems caused the dependency to fossil fuels to reduce. Fossil fuels cover wide range of use in the industry, which is almost 80%. This amount of fossil fuels has an enormous effect on global warming and CO2 emissions. Europe Parliament Committee agreed to reduce greenhouse emissions by 20% by 2020. (Escobar et al., 2009)

Petroleum, one of the most used energy source, is finite. Therefore, governments shifted towards looking for alternative methods of producing energy. For this aim, biodiesel can be a replacement or blend for fossil fuel. Biodiesel -so called fatty acid methylesters (FAME)- is commonly added to the diesel fuel up to 20%, which is %7 in Germany. (USEPA, 2002) Biodiesel consist fatty acid methylester (FAME) from various vegetable oils. In many countries special production is provided for raw material supply for biodiesel.

1.2 Biodiesel Fuel

Biodiesel is generally found in form of blend. As an example, B20 describe the percentage of the biodiesel added to the blend. Therefore, B100 is 100% biodiesel fuel. It is made up of vegetable oil or some animal fats. Mainly soybean and corn oil are extracted from seeds. It can be used in all diesel engines without modification. This provides enormous range of use for biodiesel fuel.

Diesel cycle is suitable for biodiesel fuel. Therefore, diesel engines are suitable with biodiesel fuel. (Tutunea et al.,2013) Literature shows that biodiesel has higher flashpoint and cetane number compared to the diesel fuel and increases lubricity. (Lujaji et al.) Although biodiesel has major cons compared to petroleum fuels, one of the main drawback is its instability for oxidization. This has an impact on the quality of the fuel. Because of the nature of biodiesel fuel, it is susceptible to oxidization. Therefore, this may cause engine and injector problems. (Dunn RO., 2014)

Table 1.1: Advantages and Disadvantages of Using Biodiesel in Diesel Engines. (Elias et al., 2017)

Pros	CO_2 waste is reduced by using less fossil fuels
	Lower PM emissions increases lubricity
Cons	NO_x emissions are higher due to oxygenated compounds
	Corrosion of metal parts
	Sludge formation and oil degradation

1.3 Aim of Thesis

Due to the changing values of the biodiesel fuel under specific conditions, it is necessary to observe the effect of some specific experiments. Therefore, this study aims to observe the effect of temperature and storage on the biodiesel fuel which is produced by the waste vegetable oils. The properties being investigated are kinematic viscosity, density, cold flow properties, acid value and oxidation stability.

1.4 Outline of Thesis

Chapter 1: History of biodiesel fuel, energy consumption over the world and usage of biodiesel.

Chapter 2: General information about biodiesel fuel and theoretical background of the measurement of storage conditions and its effect.

Chapter 3: Experimental setup for aging of biodiesel fuel, information about the process and setup.

Chapter 4: Discussion about experimental result and valuation of study.

Chapter 5: Results of biodiesel blends and suggestions about future works.

CHAPTER 2

PROPERTIES OF BIODIESEL

2.1 Viscosity

Viscosity can be described as a resistance of a fluid to flow. Fluids that have large viscosities have higher internal friction, which ends by higher resistance against flow. Fluids with lower viscosity tends to have lower internal friction, thus lower resistance.

2.1.1 Types of Viscosity

2.1.1.1 Dynamic (absolute) viscosity

Dynamic Viscosity or Absolute Viscosity can be described as a measurement of internal resistance. It is the tangential force required to move one horizontal plane with respect to another plane. Shear stress sometimes causes shear viscosity to happen. When shear stress is produced - τ – shear viscosity is produced. Shear stress can be explained by the following equations:

First, shear strain is defined as;

$$\gamma = \frac{dx}{dy} \tag{2.1}$$

The rate of shear strain is change of strain with the time;

$$\dot{\gamma} = \frac{\gamma}{dt} \tag{2.2}$$

Therefore, dynamic viscosity can be expressed as;

$$\mu = \frac{\tau}{\dot{\gamma}} = \tau\frac{dy}{du} \tag{2.3}$$

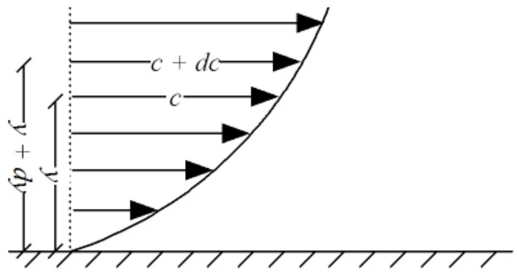


Figure 2.1: Diagram of fluid flow near the wall

2.1.1.2 Kinematic viscosity

Kinematic viscosity is the ratio of dynamic viscosity of fluid to the density of fluid. No force is involved in kinematic viscosity. Therefore, it is not affected by any forces.

$$\nu = \frac{\mu}{\rho} \tag{2.4}$$

2.1.2 Importance of viscosity

Vegetable oils which biodiesel fuel is produced are much more viscous than diesel fuel. They have higher cloud point and they are reactive to oxygen. (Agarwal et al., 2008) Fuel characteristic of biodiesel fuel causes durability problems on long-term. Therefore, it is important to observe the aging effect on the viscosity. Fuels that have higher viscosities can cause problems with atomization and it damages the fuel injector. This can cause poor engine performance and can even damage the engine. On the other hand, fuels with lower viscosity can cause lubricity problems. Therefore, finding the optimum viscosity is crucial.

Table 2.1: Kinematic viscosity of diesel fuel standards. (Gerpen et al., 2005)

Standard	**Location**	**Method**	**Fuel Type**	**Kinematic Viscosity [mm^2/s]**
EN 14214	EU	ISO 3104	Biodiesel	3.5 – 5.0
ASTM D6751	US	ASTM D445	Biodiesel	1.9 - 6.0
EN 590	EU	ISO 3104	Petro diesel	2.0 – 4.5
ASTM D975	US	ASTM D445	Petro diesel	x.9 – 4.1

There are some factors that affect the viscosity of the fuel such as temperature and pressure. With the increasing temperature, viscosity decreases, which means that the fuel becomes more fluidic. For the pressure increase, viscosity of fuel increases slightly. Although pressure has some effect on the viscosity of the fluid, it is not as effective as temperature because fluids are non-compressible.

2.1.3 Measurement of viscosity

Measuring viscosity is important to determine if the fluid is suitable for some specific application. Therefore, viscometers have been invented to measure the viscosity of the fluid. Measurements for viscosity are made according to the ASTM D6751. (Appendix) There are two different categories of viscometers:

- Capillary Viscometers
- Rational Viscometers

2.1.4 Capillary viscometers

Capillary viscometer calculations are made based on some specific equations, also known as Hagen-Poiseuille equations.

Let’s consider fully developed flow through a straight pipe. Figure x shows the cross section of this pipe. In case that z-axis is the flow direction and rotational symmetry exists to make flow two dimensional.

$$\nu_z \neq 0, \quad \nu_r = 0, \quad \nu_\theta = 0 \tag{2.5}$$

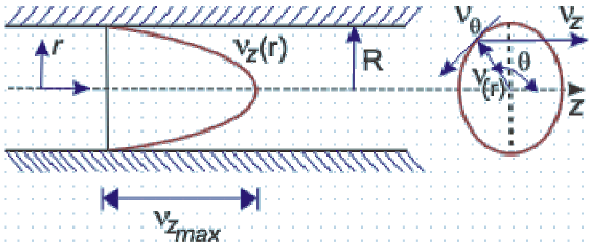


Figure 2.2: Flow Through a Vertical Pipe

From the continuity equation, we obtain

$$\frac{d\nu_z}{dz} = 0 \text{ which means } \nu_z = \nu_z(r,t) \tag{2.6}$$

By inserting the above equations to the Navier-Stokes equations, we finally obtain

$$\frac{d\nu_z}{dz} = -\frac{1}{\rho} \cdot \frac{\partial p}{\partial z} + \nu \left(\frac{\partial^3 \nu_z}{\partial r^3} + \frac{1}{r} \cdot \frac{\partial \nu_z}{\partial r} \right) \tag{2.7}$$

For the steady flow, the governing equations becomes

$$\frac{\partial^2 \nu_z}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial \nu_z}{\partial r} = \frac{1}{\mu} \cdot \frac{dp}{dz} \tag{2.8}$$

The boundary conditions are

at $r = 0$, ν_z is finite and

at $r = R,\ \nu_z = 0$

Which yields to

$$\nu_z = \frac{R^3}{4\mu}\left(-\frac{dp}{dz}\right)\left(1 - \frac{r^3}{R^3}\right) \tag{2.9}$$

And

$$-\frac{dp}{dz} = \frac{\Delta p}{L} \tag{2.10}$$

Now, discharge through pipe is given with

$$Q = \pi R^2 \nu_{z_{av}} \tag{2.11}$$

or

$$Q = \frac{\pi D^4}{128\mu}\left(\frac{dp}{dz}\right) \tag{2.12}$$

also

$$Q = \frac{V}{t} \tag{2.13}$$

where Q is the overall flow rate, V is volume and t is time.

$$\nu = \frac{\mu}{\rho} \tag{2.14}$$

and

$$\Delta p = \rho g h \tag{2.15}$$

Then,

$$\nu = \frac{\mathrm{\pi g H}R^4}{8LV} \cdot t \tag{2.16}$$

K, calibration constant is

$$K = \frac{\pi g H R^4}{8LV} \tag{2.17}$$

So,

$$\nu = Kt \tag{2.18}$$

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

$$\nu = \frac{10\pi g D^4 H t}{138LV} - \frac{E}{t^3} \tag{2.19}$$

Where e is the correction factor.

2.1.4.1 Types of capillary viscometers

There are many capillary viscometers for different use and needs. These different types are given in the Appendix.

2.2 Density

Density is the ratio of mass to the volume. It can be considered as an important property of fluid because if gives us the ratio us compound in the fuel, which is very important for bio-diesel fuel. Measurements for density are made according to the ASTM D941-88. (Appendix)

2.3 Cold Flow Properties

- Cloud point
- Pour point
- Cold filter plugging point

2.3.1 Cloud point

Temperature when wax crystals start to appear when fuel is cooled is described as Cloud point. When biodiesel is compared to the petrol-based fuels, petrol-based diesel has lower cloud point than biodiesel. Measurements for cloud point are made according to the ASTM D2500-09. (Appendix)

2.3.2 Cold filter plugging point (CFPP)

It is the temperature when fuel crystals cause the test filter to plug. Most fuels can be used below the cloud point but above the cold filter pluging point. The CFPP is considered to be a better indication of low temperature operability. Measurements for cold filter plugging point are made according to the ASTM D6371-05. (Appendix)

2.3.3 Pour point

It is the temperature where fuel have so many aggregated crystals that it will not flow any longer. This temperature does not give the exact value of the pour point because filter would be clogged before it reaches to the pour point. When biodiesel is compared to the petrol-based diesel, biodiesel has higher pour point. Measurements for density are made according to the ASTM D97-05. (Appendix)

2.4 Some Other Important Properties of Biodiesel

2.4.1 Acid number

Acid number is the measurement of mass of potassium hydroxide that is needed to make the acidic constitutes neutral. It is measured in grams per gram of sample. As biodiesel consists fatty acids, it is the measurement of carboxylic acid. (Appendix)

2.4.2 Calorific value

It is the amount of energy required to finish the combustion process of the fuel. It is measured in units of energy per amount of material. By other means, it is the amount of energy released when combustion process ends. (Klopfsentein W.E., 1985) Calorific value of biodiesel - 12% -is lower than petroleum diesel, which indicates that it has lower energy compared to the petroleum diesel.

2.4.3 Cetane number

Cetane number (CN) can be explained as the quality of a fuel for compression ignition engines (CIE). CIE engines have self-ignition. CN is inversely proportional with the delay

– the amount of time necessary between ignition and the first increase in the pressure-. Therefore, higher CN means shorter ignition delay.

2.4.4 Flash point

Flash point is the minimum temperature required or measured for vapour of the fuel to the first flash of ignition under specific conditions such as 101.3 kPa. It can indicate the flammable fuels such as petrol from the combustible fuels such as diesel. (Liu et al., 2010)

2.4.5 Oxidation stability

Due to the storage conditions and time spend during storage, oxidation is an important factor to be considered. It is known that increasing acidity causes viscosity to increase and this plugs the filters. (Monyem et al., 2001) Thus, oxidation property must be considered as an important factor to prevent engine damage for biodiesel fuels. (Appendix)

CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Bio-Diesel Sample

In this experimental study, waste frying oils were used as the bio-diesel sample supplied by Ambrosia Oils Ltd. Table 3.1 below shows the values of the standard properties when the bio-diesel sample is produced. The blend used for these experiments have properties of 80% bio-diesel and 20% petroleum-diesel. In addition, most important features for us in this experimental study are the viscosity at 40 °C and the density at 15 °C.

Table 3.1: Bio-diesel with chemical properties

NAME	METHOD	UNIT	SPECS		RESULT
			Min.	**Max.**	
FAME content	EN 14103	mass %	96.5		>99.5
Density at 15 °C	ISO 12185	kg/m^3	860.0	900.0	878.4
Kinematic Viscosity at 40 °C	EN ISO 3104	mm^2/s	3.500	5.000	4.483
Flash point (rapid equilibrium)	ISO 3679	°C	101		>140
Cetane Number	EN15195	-	51.0		59.7
Copper Corrosion (3hrs/50 °C)	EN ISO 2160	-		Class1	1A
Oxidation stability (110 °C)	EN 14112	hours	8.0		>11
Acid number	EN 14104	mg KOH/g		0.50	0.31
Iodine value	EN 14111	gI2/100g		120	74
Linolenic acid methyl ester	EN 14103	mass %		12.0	2.6

Table 3.1: Continued

Polyunsaturated methyl esters (>= 4 double bounds)	EN 15779	mass %	1.0	<0.10
Methanol	EN 14110	mass %	0.20	0.02
Glyceride content	EN 14105	mass %		
Mono-glyceride		mass %	0.70	0.21
Di-glyceride		mass %	0.20	0.02
Tri-glyceride		mass %	0.20	<0.03
Free glycerol		mass %	0.02	<0.010
Total glycerol		mass %	0.25	0.065
Water Karl Fischer	EN ISO 12937	mg/kg	300	160
Contamination	EN 12662-98	mg/kg	24	<6
Sulphated ash	ISO 3987	mass %	0.02	<0.005
Sulphur (S)	EN ISO 20846	mg/kg	10.0	9.8
Group I metals (Na+K)	EN 14538	mg/kg	5.0	<2.0
Group II metals (Ca+Mg)	EN 14538	mg/kg	5.0	<2.0
Phosphorus content	EN 14107	mg/kg	4.0	<4
Cold Filter Plugging Point	EN 116	^{o}C	+5	+5
Melting Point of Organic chemicals	ISO 6321	^{o}C		+10
Kinematic Viscosity at 20 ^{o}C	ASTM D 445	mm^2/s		7.2

Table 3.2: Standards for euro-diesel

PROPERTIES	**UNITS**	**LIMITS**		**RESULTS**	**METHOD**
		LOW	**HIGH**		

Density 15^0C	kg/m^3	820.0	845.0	827.8	ASTM D 4052
Cetane Number		51.5		55.0	ASTM D 613
Cetane Index		47.0		54.8	ASTM D 4737
Kinematic Viscosity 40 ^{o}C	Cst	2.0	4.5	2.8	ASTM D 445
Cold Filter Plugging Point, CFPP	Deg C		5	-6	IP 309
Sulphur Content	mg/kg		10.0	5.3	ASTM D 5453
Copper Strip Corrosion, 3 Hrs 50 ^{o}C	No.		1	1	ASTM D 130
Oxidation Stability	mg/l		25	3	ASTM D 2274
Carbon Residue (on 10 pct residue	WtPct		0.30	0.01	ASTM D 4530
Water Content	mg/kg		200	39	ASTM D 6304
Total Contamination	mg/kg		24	2	IP 440
Ash Content	WtPct		0.010	0.000	ASTM D 482
Strong Acid No.		NIL		NIL	ASTM D 974
Total Acid No.	Mg.Koh/gr		0.2	0.1	ASTM D 664
Flash Point	Deg C	55		67	ASTM D 93
Recovered 250 ^{o}C	Vol Pct		65	40	ASTM D 86
Recovered at 350^0C	Vol Pct	85		92	ASTM D 86
95% Recovered	^{o}C		360	360	ASTM D 86
Lubricity, wsd 1.4, 60^oC	UM		440	985	ISO 12156/1
Polycyclic aromatic Hydrocarbons	WtPct		11	2	IP 391

Table 3.2: Continued

Density in Air	kg/m^3			826.7	CALC
Colour (ASTM)	Scale		1.0	0.5	ASTM D 1500
Appearance		&Bright		Clear&Bright	ASTM D 4176

3.2 Experimental Set-Up

The theoretical background of viscosity and density with bio-diesel fuel is very important to fully understand the relationship of viscosity and density with the temperature.

3.2.1 Storage of bio-diesel blend

Bio-diesel blend sample was analyzed to determine tis viscosity, density, oxidation stability, total acid number and cold flow properties. We want to observe the properties of the biodiesel blend sample as it was held up in an oven kept at a constant temperature at 40°C as shown in the Figure 3.1. Oven was designed from an old dish washer machine. Temperature in the oven was controlled by a digital calibrated thermometer. Thus, it was possible to keep biodiesel samples at a constant temperature. When the thermocouple of thermometer inside the oven shows the ambience temperature lower than 40 °C, it gives a signal to the relay to open the circuit that turns on the lambs. When the temperature reaches 40 °C, relay cuts of the circuit so lamps turn off as shown in Figure 3.2.

Figure 3.1: Constant temperature furnace controlled by digital thermometer

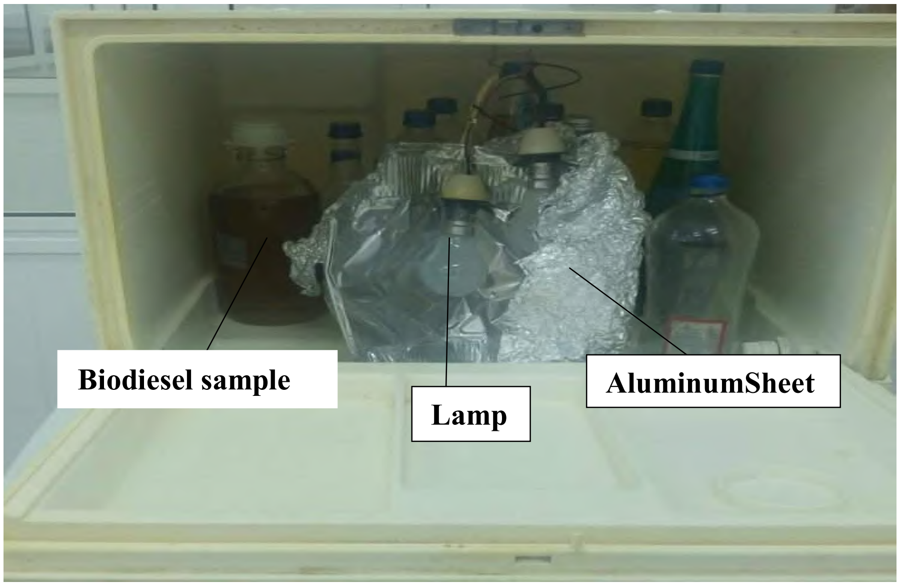


Figure 3.2: Constant temperature furnace

3.2.2 Measurement of kinematic viscosity

Measurement of kinematic viscosity is a very critical part of in this study, because it helps us to understand how this bio-diesel blend will response while stored. It is known that the kinematic viscosity is directly related to the temperature depending during the storage period. The measurement of the viscosity is carried out with a tool known as a viscometer. In this study, the kinematic viscosity of bio-diesel blend was measured by Ubbelohde viscometer. An Ubblelohde type viscometer Figure 3.3 is an instrument that uses a capillary based method of measuring viscosity and it is recommend for higher viscosity cellulosic polymersolutions. The advantage of this type of viscometer is that the values obtained are independent of the total volume. The device was developed by the German chemist Leo Ubbelohde (1877-1964) (Viswanath, 2007).

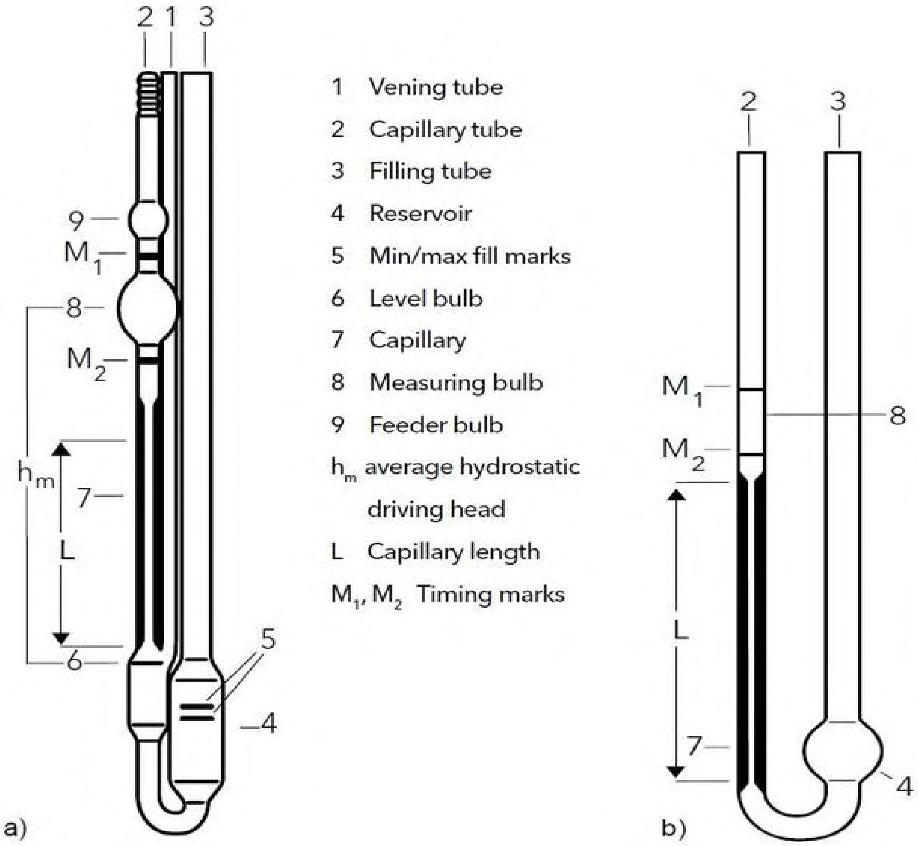


Figure 3.3: Illustrated diagram of ubbelohde viscometer

The viscometer in Figure 3.3 is basically venting tube (1), capillary tube (2), filling tube (3), reservoir (4), level bulb (6), capillary (7), measuring bulb (8), pre-run bulb (9), measurement area (8). These marks on the viscometer define not only the flow volume of the biodiesel blend but also the average hydrostatic driving head. These marks on the viscometer define not only the flow volume of the biodiesel blend but also the average hydrostatic driving head. It has the same viscometer constant for all temperatures; It shows great speed and correct accuracy. Low quantity of sample is needed during experiment, also low sensitivity to error and cost effectiveness. As shown in Figure 3.3the Ubbelohde viscometer is U-shaped of glassware with a reservoir on one side and a measuring bulb with a capillary on the other side. The liquid is allowed to travel back through the measuring bulb and the time it takes for the liquid to pass through two calibrated marks is a measure for viscosity. The Ubbelohde instrument has a third arm extending from the end of the capillary and open to the atmosphere. In this way the pressure head only depends on a fixed height and no longer on the total volume of liquid. The reason for the decision to use the Ubbelohde viscometer is that it provides flexibility in application, transparency and high temperature measurement. The Ubbelohde viscometer constant value, K, (mm^2/s) was specified with as shown below the Table 3.3 given by the manufacturer company and they calibrated. For the aim of this work and measurement, taking into account the kinematic viscosity range, three viscometer of size O_C, I and I_C were chosen in this study to measure the kinematic viscosity.

Table 3.3: Ubbelohde viscometer technical specifications

Type No.	Capillary No.	Capillary Dia I± 0.01 [mm]	Constant K (mm^2/s)s	Measuring range [mm^2/s]
525 03	O_C	0.36	0.002856	0.6 3
525 10	I	0.58	0.009820	2 …. 10
525 13	I_C	0.78	0.02944	6 …. 30
525 20	II	1.03	0.08947	20 100
525 23	II_C	1.36	0.2812	60 …. 300

The corrected flow time multiplied by the viscometer constant K directly gives the kinematic viscosity as given in Equation (3.1) for absolute measurement.

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

In the experiment formula with the Equation 3.1 was used to get the kinematic viscosity values. In order of given v, K, t and y symbolize the kinematic viscosity, the calibration constant, measured time of flow and kinematic energy correction. The kinetic energy correction y is given in terms of flow time for each viscometer as shown in Table 3.4 below.

Table 3.4: Table of kinematic viscosity correction

Ubbelohde viscometer ISO 3105/DIN51 562/Part/BS188/NFT 60-100,
Ref.No.501…530…532
Correction seconds[A]:

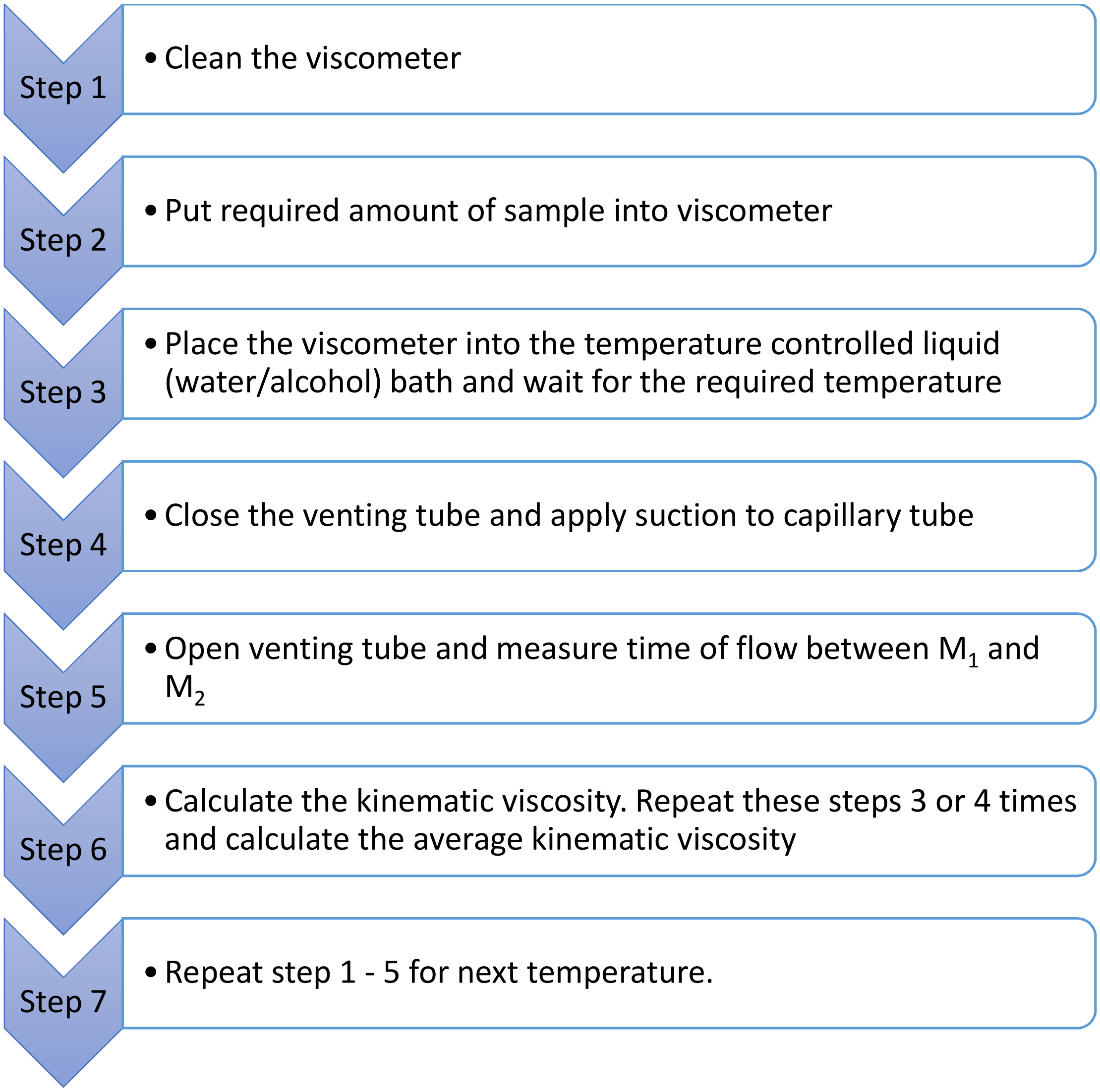
Flow	**Capillary No**						
time	**0**	**0_C**	**0_a**	**I**	**I_C**	**I_a**	**1**
40	-[B]	-[B]	-[B]	1.03	0.45	0.15	
50	-[B]	-[B]	-[B]	3.96	0.66	0.29	0.10
60	-[B]	-[B]	-[B]	2.75	0.46	0.20	0.07
70	-[B]	-[B]	-[B]	2.02	0.34	0.15	0.05
80	-[B]	-[B]	4.78[B]	1.55	0.26	0.11	0.04
90	-[B]	-[B]	3.78[B]	1.22	0.20	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
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] The correction seconds stated are related to the respective theoretical constant

[B] For precision measurement, these flow times should not be applied. Selection of a viscometer with smaller capillary diameter is suggested.

3.2.2.1 Procedure of measuring the kinematic viscosity using Ubbelohde viscometer

The kinematic viscosity was measured using an Ubbelohde viscometer as previously mentioned. The flow chart in Figure 3.4 summarizes how the measurement process of kinematic viscosity step by step using the Ubbelohde viscometer.



Step 1
- Clean the viscometer

Step 2
- Put required amount of sample into viscometer

Step 3
- Place the viscometer into the temperature controlled liquid (water/alcohol) bath and wait for the required temperature

Step 4
- Close the venting tube and apply suction to capillary tube

Step 5
- Open venting tube and measure time of flow between M_1 and M_2

Step 6
- Calculate the kinematic viscosity. Repeat these steps 3 or 4 times and calculate the average kinematic viscosity

Step 7
- Repeat step 1 - 5 for next temperature.

Figure 3.4: Flow chart of kinematic viscosity using Ubbelohde viscometer

Step One

Clean the Ubbelohde viscometer using cleaning materials. The cleaning material must be prepared at the right proportions. To prepare the cleaning material 70% distilled water, 15% hydrogen peroxide and 15% muriatic acid were used. After this cleaning material were used, cleaning with acetone was completed. The reason for using acetone is that it has drying property. The capillary tube must be dry and dust free to start the experiment process.

Step Two

We need to fill the viscometer with enough biodiesel and we need to be sure that biodiesel is between the two lines on the tube thus the quantity of liquid charged will not block the air tube during use.

Step Three

Place the viscometer in the fluid chamber on the device, which keeps the temperature constant using an electromagnetic hot plate. The blend of biodiesel should be at the same temperature as the bath temperature and it will take about 20 minutes to reach this temperature. Figure 3.5 as shown measure the viscosity of biodiesel sample.

Figure 3.5: Measure the viscosity of biodiesel sample in the temperature range 30^{o}C - 90^{o}C

Step Four

Next from Step 3, occlude venting tube and apply quite slowly suction to the capillary tube with the suction instrument which is shown in Figure 3.6. Make sure that upper timing mark is at a minimum 2 cm below the bath liquid level. Carry out suction to the capillary tube until the liquid get about 5 mm above the upper timing mark. Bring the venting tube to the level of the upper timing mark. Suction continues until the venting tube opens.

Figure 3.6: Suction instrument

Step Five

After step 4, the timing tube is released and the flow of liquid is allowed. The flow time t of the mixture from the upper timing mark M1 down to the upper edge of the lower timing mark M2 is measured. The time required for the liquid to pass through the two calibrated marks is a measure of viscosity.

Step Six

The kinematic viscosity of the sample is obtained by multiplying the flow time t by the calculating "v" using formula in Equation 3.1 above. Repeat the procedure 3 or 4 times until you find near values. When near values are found, the arithmetic average is taken. It can be passed to new measurements at different temperature values.

Step Seven

Repeat Step 1 - 5 for next temperature. After all measurements have been completed, the viscometer were cleaned and the measuring instruments are stored in a convenient place.

For example;

- Determining the kinematic viscosity of (%80BD + %20ED) at 40 ^{o}C

- Capillary I constant (K) = 0,009820 (mm^2/s)/s
- Flow time (average time) (t) = 447.1321 s
- Kinematic energy correction (HC) y for 447.1321 = 0.08

$v = K (t – y)$

$v = 0.009820 (447.1321 – 0.08) = 4.3901\ mm^2/s$

3.2.3 Measurement of density

Density, in physics and chemistry, is the amount of matter per unit volume under a certain temperature and pressure. The density measurement of the biodiesel blend is measured with a device called a pycnometer. Measurements of the biodiesel sample were made using pycnometer between 5 ^oC to 20 ^oC and 30 ^oC to 90 ^oC. The biodiesel mixture was cooled or heated, and the measurements were made with a pycnometer when the temperatures reached to the desired level. The measurements were made three or four times for each temperature level and the arithmetic average of the results was calculated and written in the equation. In the equation used to calculate the density, the pycnometer empty mass [g], the pycnometer mass filled with biodiesel [g], and the pycnometer volume [ml] are used. The density of the sample of the biodiesel blend given in the Equation 3.2 is obtained in kg/m^3.

$$\rho = \frac{(m_{full} - m_{empty})}{V} \tag{3.2}$$

3.2.2.2 Pycnometer

This device is made of glass or metal material. However, a glass pycnometer was used in this experiment. Because of that the pycnometer was made of glass, one must be careful that it could break so easily. The pycnometer should be calibrated before starting the experiment. This calibration process must be performed to determine the empty pycnometer mass and volume so that we can accurately determine the density of the biodiesel blend. As the biodiesel blend is heated, the volume increases and the density decreases, when the biodiesel blend is cooled the volume decreases and density increases. So, air bubbles and excess liquid come out of the capillary tube. The flow chart in Figure

3.7 summarizes how the measurement procedure of density can be step by step using the Pycnometer.

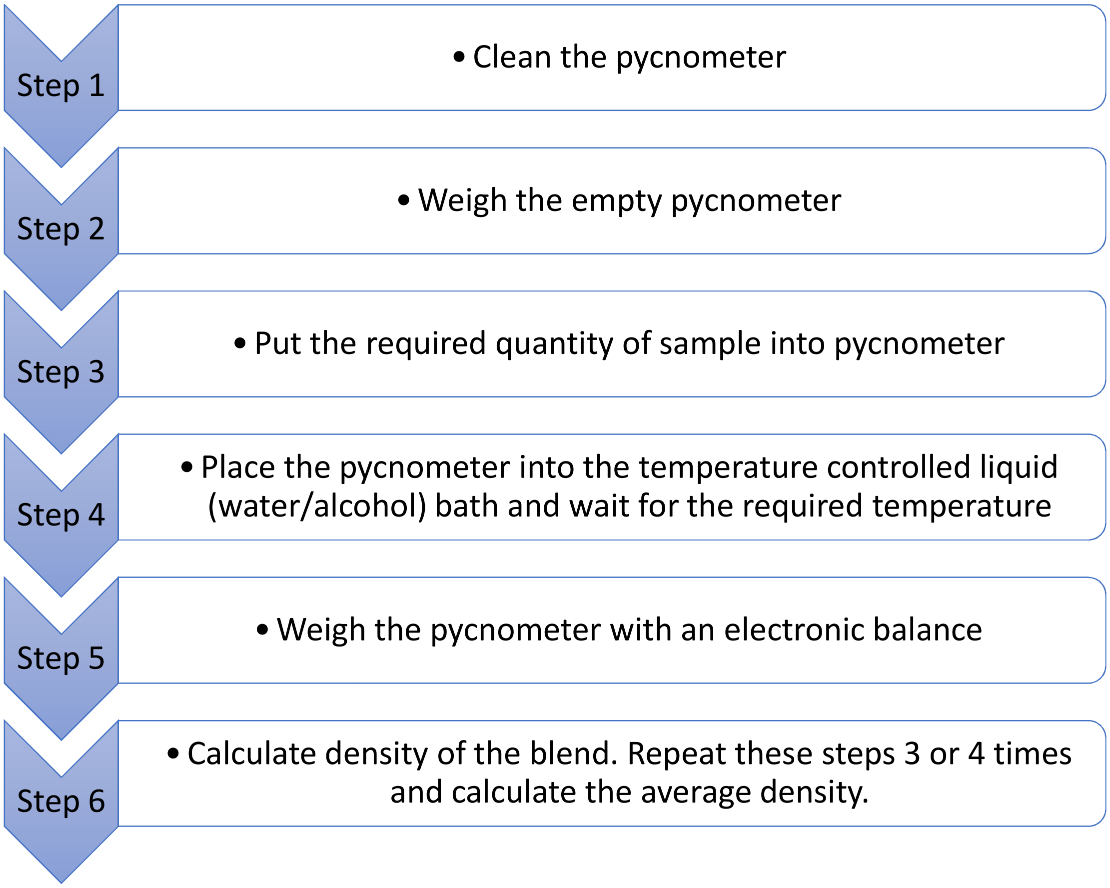


Figure 3.7: Flow chart of density using Pycnometer

Step One

Clean the Pycnometer using the cleaning materials. The cleaning material must be prepared at the right proportions. To make cleaning material, 70% distilled water, 15% hydrogen peroxide, 15% muriatic acid is used. After, this cleaning material is used, cleaning with acetone is completed. The reason for using acetone is that it has drying property. Pycnometer must be dry and dust free to start the experiment process.

Step Two

We need to weigh the empty pycnometer with on electronic balance. And to be sure that before filling the flask with biodiesel blend.as shown in Figure 3.8. Empty pycnometer mass needed to use the Equation 3.2

Figure 3.8: Pycnometer weigh

Step Three

We need to fill the pycnometer with enough biodiesel. in addition to this we need to be sure that excess biodiesel and air bubbles will overflow from the pycnometer. Figure 3.9 below shows the empty pycnometer.

Figure 3.9: Overflow gap of pycnometer

Step Four

Placed the pycnometer into the temperature-controlled liquid in heating bath or cooling bath, accordingly wait for the required temperature. The required temperature becomes homogenous in at least 15 minutes in the beaker or cooling bath.

Step Five

As shown in Figure 3.10, the pycnometer filled with the biodiesel mixture is weighed on the electronic scale.

Figure 3.10: Pycnometer on the electronic scale

Step Six

The density of the sample is obtained by the calculating "p" using formula in Equation 3.2. Repeat the procedure 3 or 4 times until you find near values. When near values are found, the arithmetic average is taken. It can be passed to new measurements at different temperature values. After all measurements have been completed, the pycnometer is cleaned and the measuring instruments are stored in a convenient place.

For example;

- Determining the density of (%80BD + %20ED) at 15^{0}C
- m_{empty}= 42.763 g
- m_{full} = 127.699 g
- V = 99.693 g

$$\rho = \frac{(m_{full} - m_{empty})}{\mathrm{V}} = \frac{(127.699-42.763)}{99.693} = \frac{84.936}{99.693} = 0.8520 \text{ g/l}$$

ρ = 0.8520 × 1000 = 851.9756 kg/m^3

3.2.2.3 Electromagnetic hot plates

The density and kinematic viscosity was measured using a Pycnometer as previously mentioned. Heating of the biodiesel blend in the pycnometer is provided by this electromagnetic hot plate. By fixing the temperature at a certain value, the biodiesel blend can reach the same temperature homogeneously. The name of this device used is hiedolphmr hi-tec as an electromagnetic heater. The plate on the device is manufactured from aluminum, which performs the heating faster, and is highly needed to determine the density and kinematic viscosity at 30 ^{o}C to 90 ^{o}C at high temperatures. Digital temperature settings and variable speed are available. The temperature can be adjusted up to 300 ^{o}C and the speed can be adjusted between 100 to 1400 rpm. For the values to be measured according to the temperature effect, water is used as liquid in the glass beaker. Figure 3.11 shows the image of the device we used in the experiment.

Figure 3.11: Electromagnetic hot plates

3.2.3 Cooling bath system

The purpose of this system is to cool the liquid cooling bath and keep it homogeneously at a constant temperature. Then, it was used to measure the kinematic viscosity and density. In cooling bath, pure alcohol is used instead of water as cooling fluid. The reason is that water freezes at 0 ^{o}C, whereas pure alcohol freezes at -117 ^{o}C. The alcohol used in the device is 97% pure and is supplied from a local alcohol factory in Northern Cyprus. The kinematic viscosity and density measurement range is made between 5 ^{o}C and 20 ^{o}C in the cooling bath. Figure 3.12 shows the image of the cooling bath we used in the experiment.

Elements of the cooling bath setup

1-Cooling bath reservoir

2- Alcohol

3-Ubbelohde viscometer with holder

4- Coil

5- Isolator (Styrofoam)

6- Thermostat

7- Compressor

8- Radiator

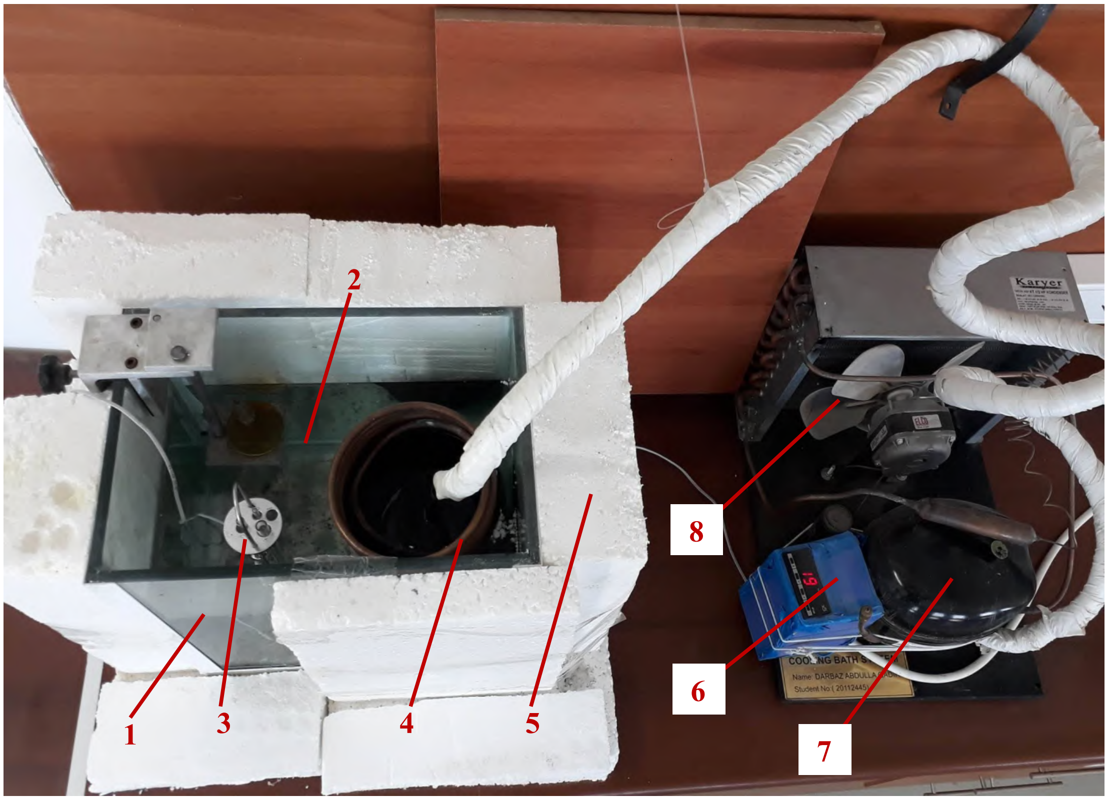


Figure 3.12: Cooling bath setup

3.2.4 Cold flow properties

In this study, the cold flow properties of the biodiesel blend are investigated. It is the cold flow property that defines the fluidity property of the biodiesel blend at low temperatures. In this experiment, cold flow properties such as cloud point (CP), cold filter plugging point (CFPP) and pour point (PP) of the biodiesel blend were measured. Figure 3.13 and Figure 3.14 shows the images of the setup we used in the experiment. The sample was measured in accordance with the American standard test method to measure cold flow properties, taking into consideration the cloud point, cold filter plugging point and pour point, ASTM D2500, ASTM D6371-05 and ASTM D97 respectively.

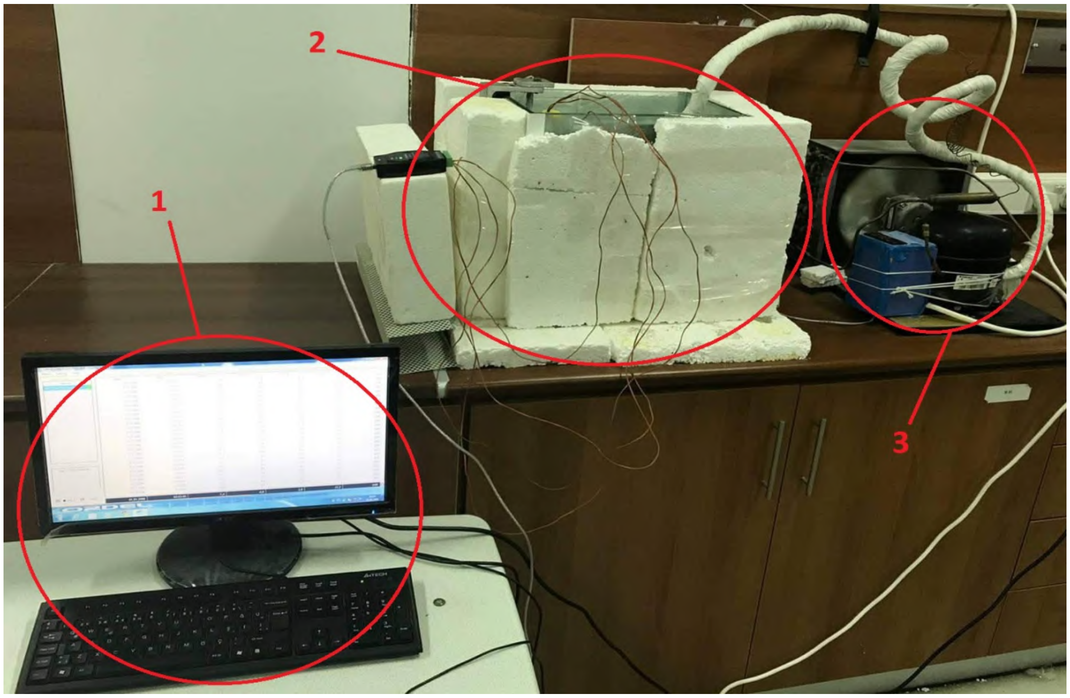


Figure 3.13: Cold flow properties measurement main elements

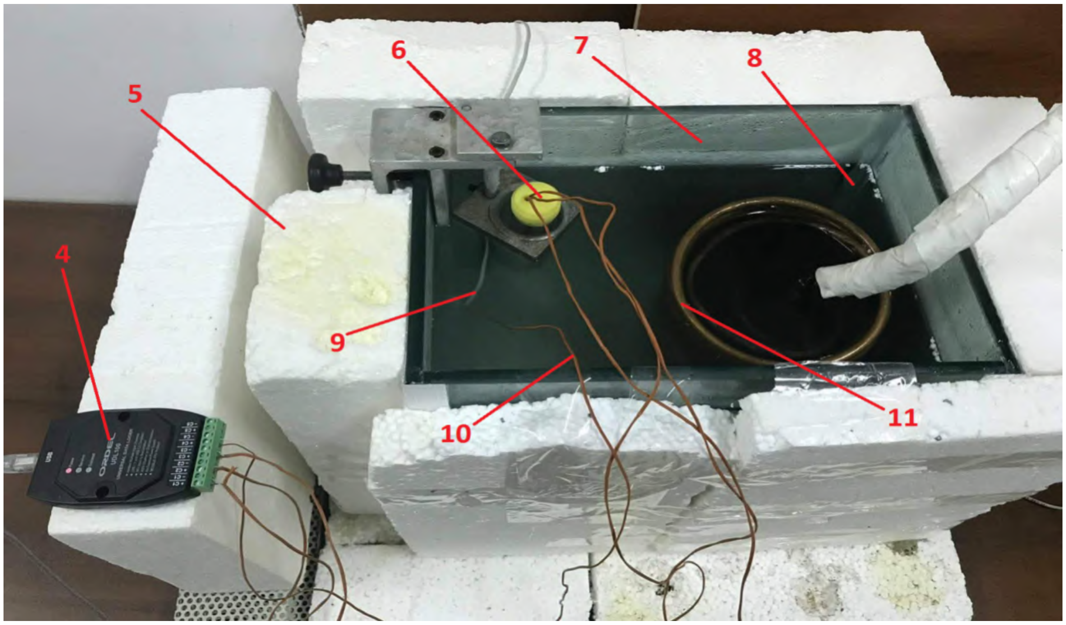


Figure 3.14: Cold flow properties experimental set-up

Elements of the setup

1- Data system (Main element)

2- Cooling bath (Main element)

3- Compressor system (Main element)

4- Data logger

5- Insulator (Styrofoam)

6- Glass of test jar

7- Cooling bath reservoir

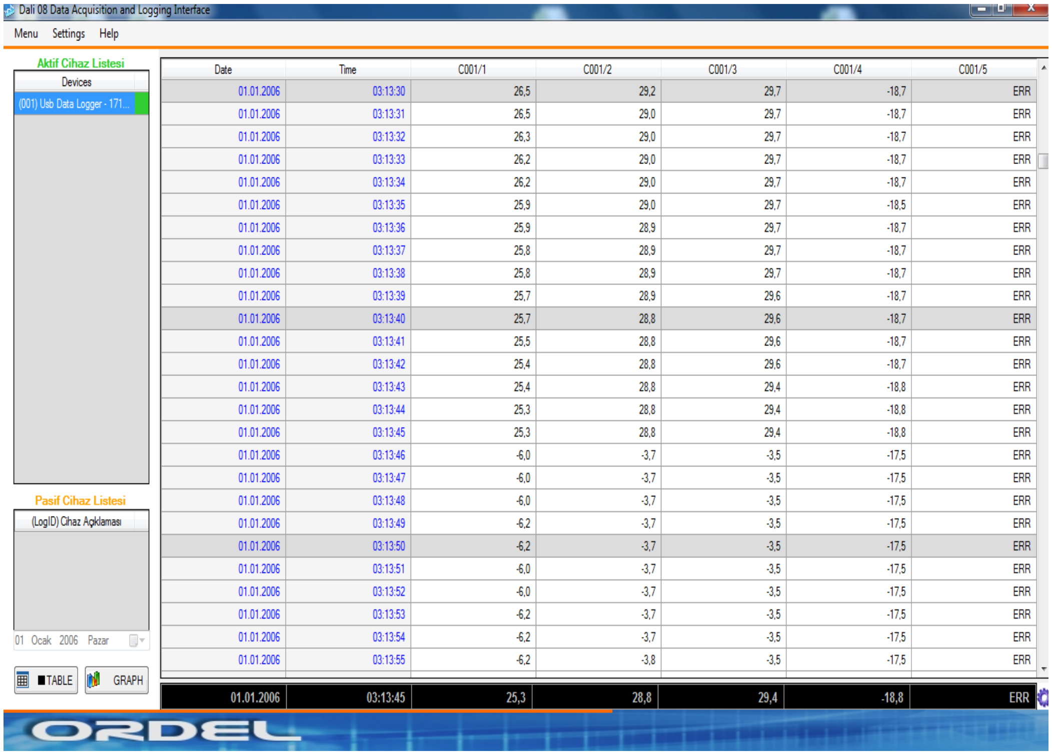
8- Alcohol

9- Thermocouple of compressor system

10- Fourth thermocouple of data logger

11- Coil of compressor system

Alcohol is used as the cooling fluid which is resistant to -117 ^{o}C in the cooling bath reservoir. The alcohol temperature in the cooling bath is controlled by a thermostat. The temperature control is controlled by placing the thermocouple in alcohol and connecting it to the thermostat. A compressor is used to keep the temperature of the cooling bath constant. The thermostat automatically turns on and off the compressor system by measuring the temperature. Styrofoam insulation is provided so that the temperature inside the cooling bath reservoir is not affected by room temperature. The data logger is recorded by providing the computer connection of the device with the special software program named in order to record the data obtained from the thermocouple placed in the alcohol.



Date	Time	C001/1	C001/2	C001/3	C001/4	C001/5
01.01.2006	03:13:30	26,5	29,2	29,7	-18,7	ERR
01.01.2006	03:13:31	26,5	29,0	29,7	-18,7	ERR
01.01.2006	03:13:32	26,3	29,0	29,7	-18,7	ERR
01.01.2006	03:13:33	26,2	29,0	29,7	-18,7	ERR
01.01.2006	03:13:34	26,2	29,0	29,7	-18,7	ERR
01.01.2006	03:13:35	25,9	29,0	29,7	-18,5	ERR
01.01.2006	03:13:36	25,9	28,9	29,7	-18,7	ERR
01.01.2006	03:13:37	25,8	28,9	29,7	-18,7	ERR
01.01.2006	03:13:38	25,8	28,9	29,7	-18,7	ERR
01.01.2006	03:13:39	25,7	28,9	29,6	-18,7	ERR
01.01.2006	03:13:40	25,7	28,8	29,6	-18,7	ERR
01.01.2006	03:13:41	25,5	28,8	29,6	-18,7	ERR
01.01.2006	03:13:42	25,4	28,8	29,6	-18,7	ERR
01.01.2006	03:13:43	25,4	28,8	29,4	-18,8	ERR
01.01.2006	03:13:44	25,3	28,8	29,4	-18,8	ERR
01.01.2006	03:13:45	25,3	28,8	29,4	-18,8	ERR
01.01.2006	03:13:46	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:47	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:48	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:49	-6,2	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:50	-6,2	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:51	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:52	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:53	-6,2	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:54	-6,2	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:55	-6,2	-3,8	-3,5	-17,5	ERR
01.01.2006	03:13:45	25,3	28,8	29,4	-18,8	ERR

Figure3.15: Screen display of the software program of the data logger

An example of a B80 D20 biodiesel blend was placed in the test tube to measure the cold flow properties. Keep the biodiesel blend and thermocouples at 45 ml in the Figure 3.16 as shown in the test tube from the glass.

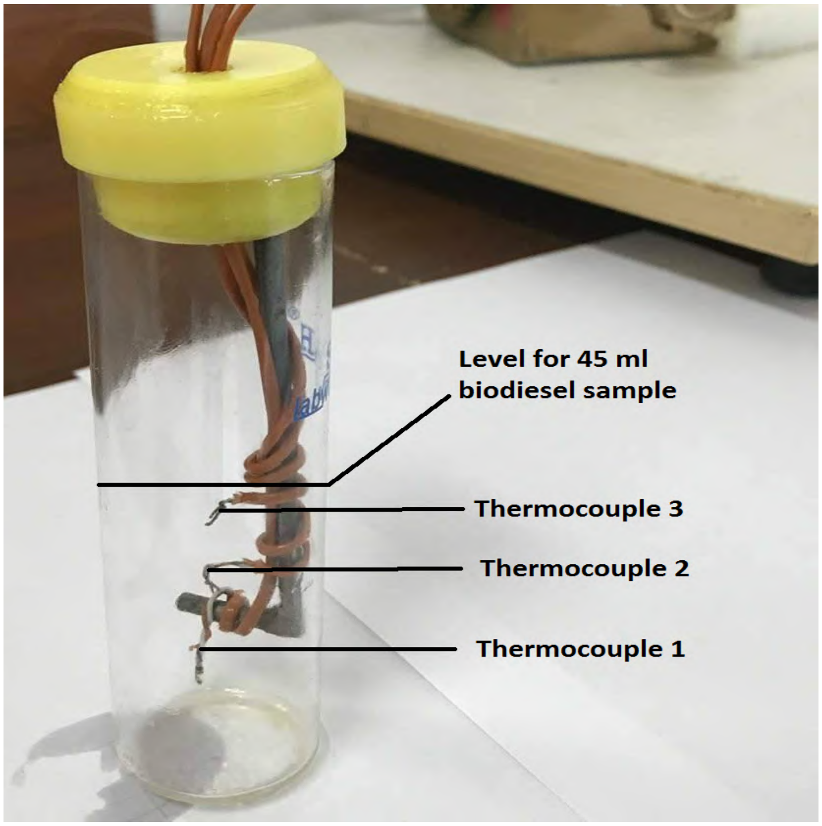


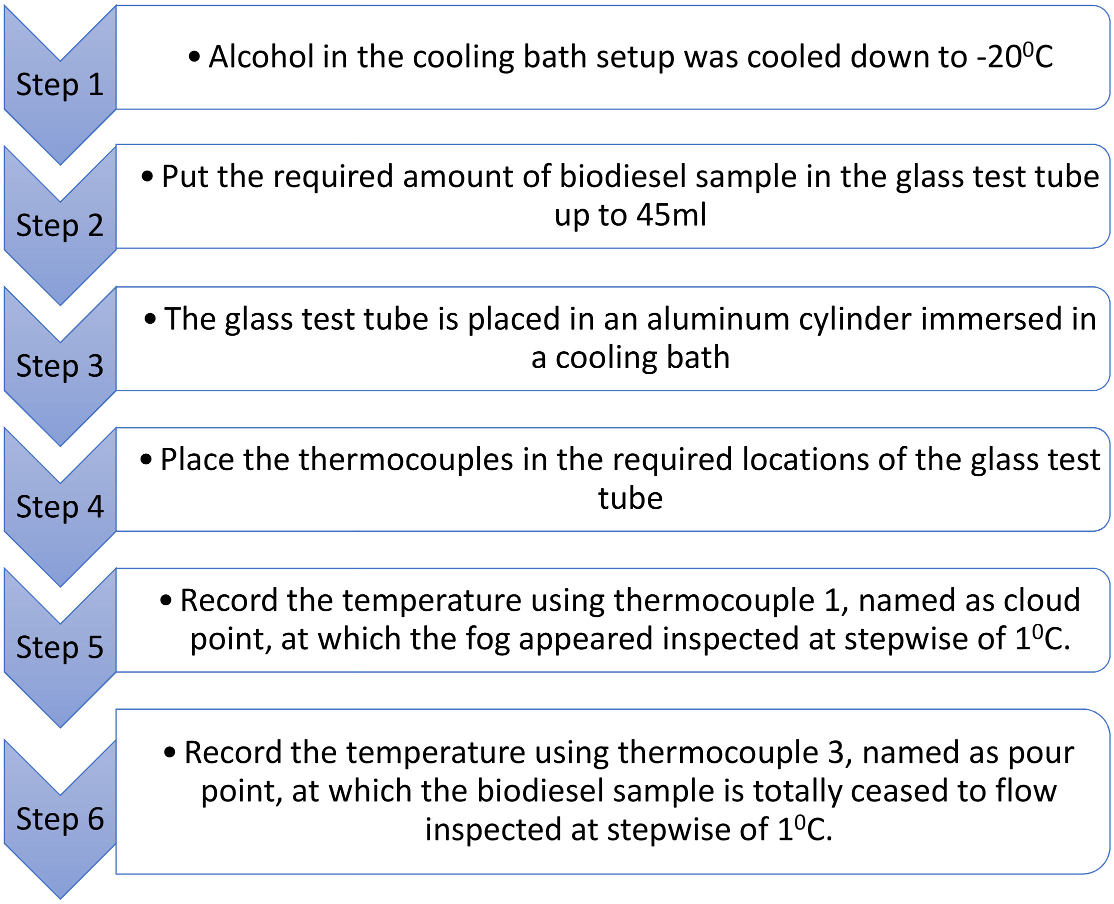
Figure 3.16: Glass of test tube with thermocouples

Normally five different thermocouples are connected to the data logger. However, four thermocouples are connected in this setup. The location of the thermocouples is located at the bottom, middle and top of the test tube so as not to exceed 45 ml. In this experiment, cloud point, pour point and cooling curve values are measured. Cold filter plugging point is determined using cooling curve. There are 3 thermocouples in the test tube to find these values. The fourth thermocouple is also placed in the alcohol and its task is to send information by measuring the temperature of the alcohol. Its function is the same as the thermostat that allows the compressor system to operate.

For measuring procedure flow chart of pour point and pour point as shown in Figure 3.17. When the cloud point is determined, it is cooled in a fuel-controlled manner as defined in

ASTM D2500, and a fog can be visually inspected in the normally clear biodiesel blend. When this fog is visually noticed, it can be analyzed by the data logger and monitored at what temperature point it takes place. It can also be analyzed from the cooling curve graph.

The American Standard, ASTM D97, is used to measure the pour point of the fuels identified in the test method. A second measure of the performance of the biodiesel blend at low temperatures is the pour point. The pour point is the lowest temperature the biodiesel sample will flow.



Step 1
- Alcohol in the cooling bath setup was cooled down to -20^{0}C

Step 2
- Put the required amount of biodiesel sample in the glass test tube up to 45ml

Step 3
- The glass test tube is placed in an aluminum cylinder immersed in a cooling bath

Step 4
- Place the thermocouples in the required locations of the glass test tube

Step 5
- Record the temperature using thermocouple 1, named as cloud point, at which the fog appeared inspected at stepwise of 1^{0}C.

Step 6
- Record the temperature using thermocouple 3, named as pour point, at which the biodiesel sample is totally ceased to flow inspected at stepwise of 1^{0}C.

Figure 3.17: For measuring procedure flow chart of cloud point and pour point

3.2.5 Acid number and oxidation stability

A certified laboratory in the Greek Cypriot region was sent to measure the acidity and stability of the oxidation. The name of this laboratory is the Nortest Petrochemical laboratory. Test methods ASTM D664-04 (2017) and EN 15751: 2014 were used to measure acid number and oxidation stability, respectively. The ASTM 15751 standard specifies a test method for determining the oxidation stability of fuels by measuring the induction period of diesel engine fuels up to 48 hours. ASTM D664 is a measure of the abrasivity and long-term stability of the acidic components in the biodiesel. This standard measures acidic components in biodiesel.

CHAPTER 4

RESULTS AND DISCUSSION

In this chapter, we discussed the results and evaluation of the experiments. Before starting the experiments, we measure the kinematic viscosity of a known fluid to ensure the accuracy of the devices. This fluid is pure water. In literature the kinematic viscosity of pure water is given as 0.80908 mm^2/s at 30 ^oC. When the same experiment was carried out under the same conditions, the kinematic viscosity of pure water at 30 ^oC was measured as 0.803 mm^2/s using a Ubbelohde viscometer. Little difference was observed between the value given in the literature and the pure water experiment we analysed. The absolute error calculated is less than 1%. This indicates that the margin of error is not a problem and the calibration performed to the device is good. Repeatability tests were performed to ensure a precise measurement and the results were very close. To obtain the best result, the measured temperature is repeated and the average is taken.

Kinematic viscosity and density were measured between 5 ^oC and 90 ^oC for the blend B80 - 80% biodiesel and 20% euro-diesel. Measurements were made over a 90-day storage period at a constant temperature of 40 °C to measure the kinematic viscosity. On the other hand, kinematic viscosity was measured over 12 weeks at temperatures ranging from 5 °C to 90 °C. Likewise, density measurements were made at a fixed temperature of 15 °C for 90 days and for 12 weeks at temperatures ranging from 5 °C to 90 °C. These measurements were made to determine if it was time and temperature dependent.

4.1 Kinematic Viscosity Experiments

In this section, the results of the kinematic viscosity experiments measured depending on the temperature and storage time of the biodiesel blend are examined.

4.1.1 Kinematic viscosity over a storage period of 90 days at temperature 40 ^{o}C

Table 4.1 below shows kinematic viscosity measurements at a constant 40 ° C temperature every 10 days for the 90 days storage period of the biodiesel blend. These measurements have been made according to the international standard ASTM D6751. (Appendix)

Table 4.1: Influence of storage period (days) at constant storage temperature on kinematic viscosity of biodiesel blend in mm^2/s

B80 Sample	
Day	**Viscosity at 40 ^{o}C**
0	4.190
10	4.236
20	4.278
30	4.335
40	4.387
50	4.430
60	4.453
70	4.506
80	4.551
90	4.585

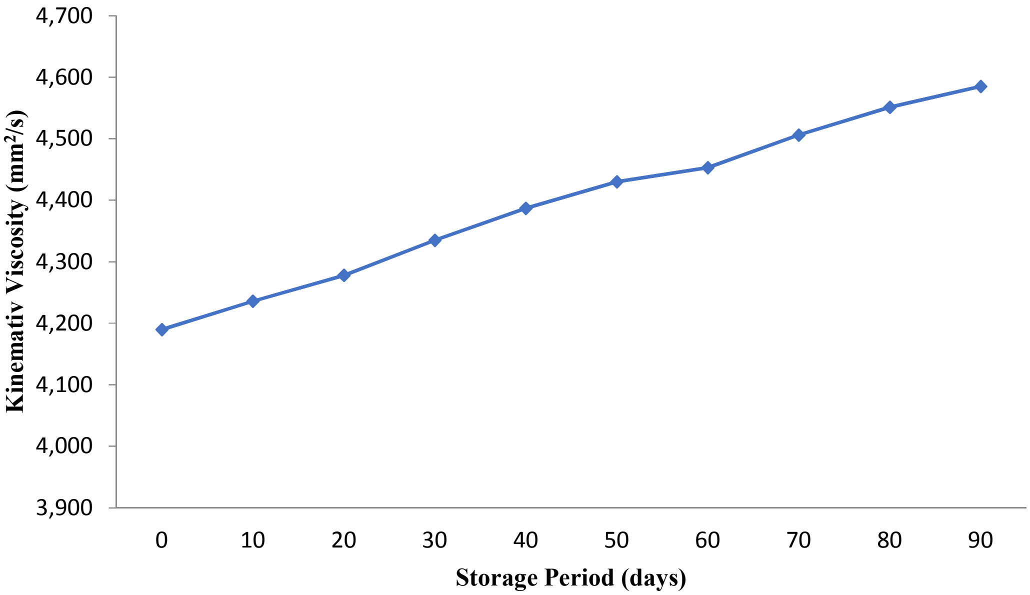


Figure 4.1: Kinematic viscosity vs storage period for 90 days at 40 °C

As seen in Figure 4.1, as storage time increases, kinematic viscosity slightly increases. During the storage period an increase in the total acid number was observed on the biodiesel blend sample and it is known that it would lead to oxidation. As the chain length of the hydrocarbons in the biodiesel increases, the viscosity value increases and as the number of double bonds increases, the viscosity decreases. As the number of hydrogens decreases, the chain length of hydrocarbons become shorter. Impure biodiesel and oxidation products increase viscosity.

4.1.2 Kinematic viscosity over testing temperatures

Table 4.2 below shows kinematic viscosity measurements at temperatures from 5 °C to 90°C for a period of 4 weeks for a 12-week storage time of the biodiesel blend.

Table 4.2: Influence of storage period (weeks) and testing temperatures on kinematic viscosity of biodiesel blend in mm^2/s

B80 Sample				
Temperature T (°C)	**0 week**	**4 week**	**8 week**	**12 week**
5	10.330	10.431		
8	10.040	10.154	10.291	10.49
10	8.970	9.157	9.285	9.57
15	7.400	7.558	7.860	7.82
20	6.760	6.899	6.955	6.94
30	5.200	5.313	5.519	5.58
40	4.190	4.335	4.453	4.58
50	3.390	3.463	3.591	3.55
60	2.850	2.932	3.115	3.10
70	2.480	2.557	2.700	2.72
80	2.140	2.203	2.367	2.40
90	1.780	1.829	2.092	2.12

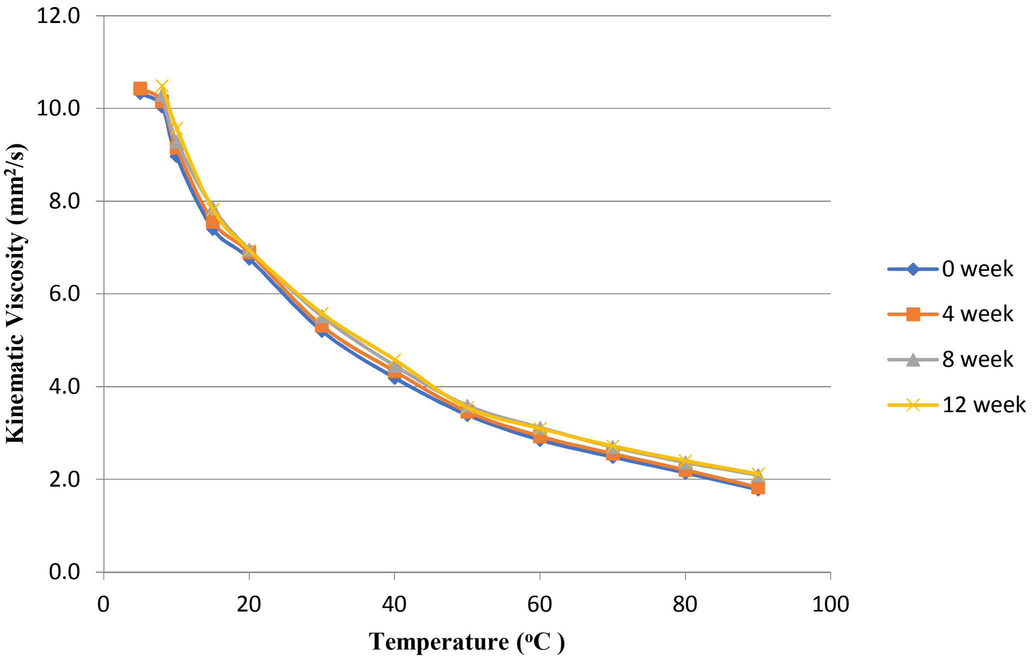


Figure 4.2: Kinematic viscosity - temperatures - storage period relation

Unlike the storage period, the kinematic viscosity decreases with increasing temperature during the 12 weeks experimental period with the biodiesel mixture. As the temperature increases, the viscosity decreases. When liquid, there is an additional attractive and cohesive force between the molecules. Therefore, both cohesion and molecular exchange affects the kinematic viscosity. Due to the decrease in kinematic viscosity due to temperature, the viscosity coefficient of the liquid increases as the temperature increases, the viscosity decreases and the fluidity property increases.

4.2 Density Experiments

In this section, the results of the density experiments measured depending on the temperature and storage time of the biodiesel blend are examined.

4.2.1 Density over a storage period of 90 days at temperature 15 ^{o}C

Table 4.3 shows the density measurement for biodiesel blend over 12 weeks for each 10 days at constant temperature 15 ^{o}C.

Table 4.3: Influence of storage period (days) at constant storage temperature on density of biodiesel blend in kg/m^3

B80 Sample	
Day	**Density at 15 ^{o}C**
0	862.640
10	865.228
20	867.824
30	868.699
40	871.305
50	873.919
60	874.400
70	875.274
80	876.150
90	879.426

As seen on Table 4.3, increasing temperature decreases density. Density measurements were made at 15 ^{o}C, which is a standard for ASTM. It was expected to observe slight decrease in density because increasing temperature increases the molecular movements which leads to an increase in volume which then leads to an increase in density.

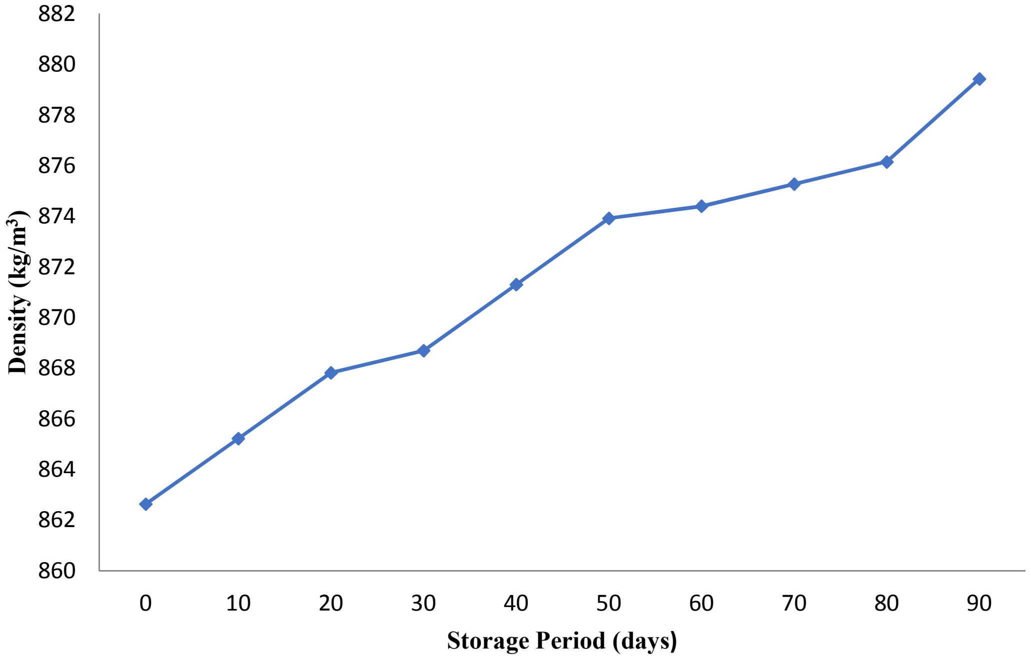


Figure 4.3: Density vs storage period for 90 days at 15 ^{o}C

Density measurements seen on Figure 4.3 were made at constant temperature for 90 days. As a result, it was observed that density decreased with increasing temperature. As the chain length of the hydrocarbons in the biodiesel increases, the density value decreases and as the number of double bonds increases, the density increases. Density has also increased over the period of storage due to the oxidation processes leading to an increase in polymer and fatty acid chains. All this pollution makes the fuel more viscous.

4.2.2 Density over testing temperatures

Table 4.4 shows the density measurement for biodiesel blend over 12 weeks for each 4-week periods where temperature ranges from 5 ^{o}C to 90 ^{o}C.

Table 4.4: Influence of storage period (weeks) and testing temperatures on density of biodiesel blend in kg/m^3

B80 Sample				
Temperature T (^{o}C)	**0 week**	**4 week**	**8 week**	**12 week**
5	887.876			
8	879.437	885.614	891.420	896.500
10	870.998	877.116	882.866	887.900
15	862.640	868.699	874.400	879.400
20	854.282	860.283	865.923	870.900
30	845.924	851.866	857.451	862.400
40	835.930	841.801	847.320	852.200
50	834.013	839.871	845.378	850.200
60	824.827	830.621	836.067	840.900
70	817.991	823.737	829.137	833.900
80	804.491	810.142	815.454	820.100
90	798.194	803.801	809.070	813.700

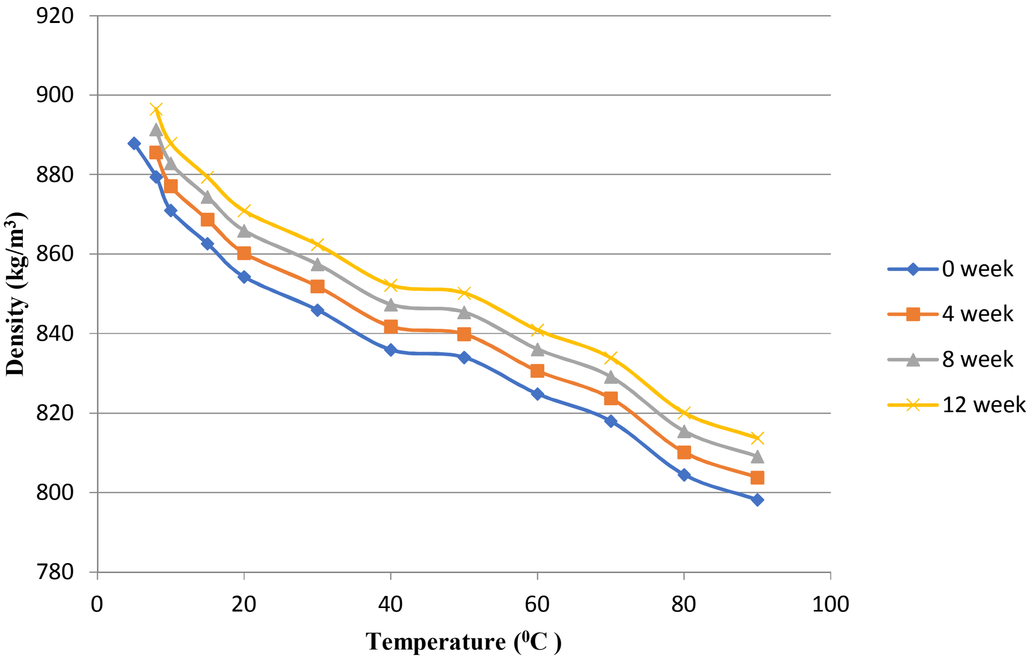


Figure 4.4: Density - temperatures - storage period relation

Figure 4.4 illustrates the temperature – density relationship for 12 weeks period. Each 4 week, another measurements were made until 12th week. Temperature increase for any week increases the volume of the blend. Therefore, density decreases with increasing temperature.

4.3 Acid Number and Oxidation Stability Experiments

Total acid number and oxidation stability analyses were made in Southern part of Cyprus at Nortest Petrochemistry Laboratory. In total, two measurements were made with a month of break.

Table 4.5: Total acid number and oxidation stability test values

B80-D20 Sample				
Test Description-Units	**Test Method**	**Spec. Limits**	**Test Result (1st Month)**	**Test Result (2nd Month)**
Total Acid Number (mgKOH/gr)	D664-04(2017)	-	0.40	0.19
Oxidation Stability (Hours)	EN15751:2014	-	1.0	3.75

Table 4.5 shows the experimental results of total acid number and oxidation stability for B80 biodiesel blend. These tests were measure once a month for a two-month period. As it is seen on table, total acid number decreases during storage period where oxidation stability increased.

4.4 Cold Flow Properties Experiment

The cold flow properties of the biodiesel blend were measured in the previous section. These features are CP, PP and CFPP. Before making the cold flow measurements of the biodiesel mixture, it was mentioned that the device to be measured was calibrated with the data logger. When calibrating, the comparison of the data of the thermostats, which measure the temperature of the cooling bath with the actual temperature, was carried out by recording by the data logger. The graphs obtained as a result of this comparison were repeated three times as shown in Figure 4.5, Figure 4.6, and Figure 4.7 below. It was determined that the results obtained with this calibration were very close to the desired value of 1% error margin. Therefore, device was suitable for use without any problems.

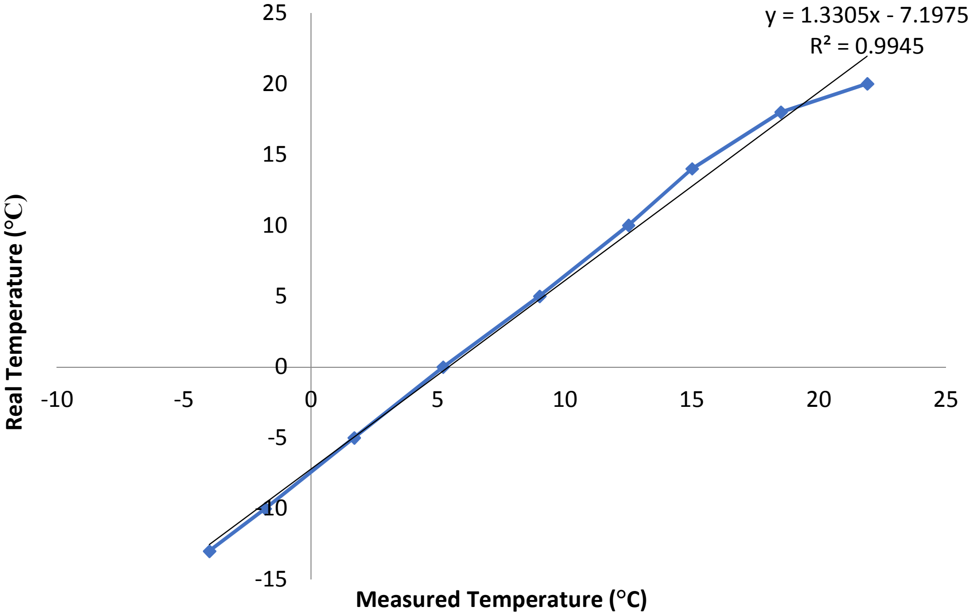


Figure 4.5: First test for the calibration

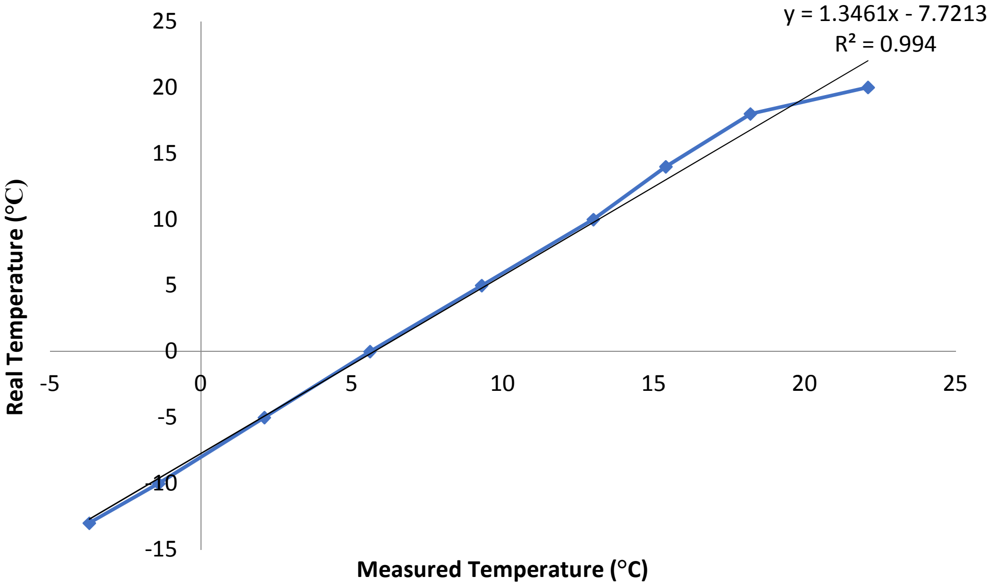


Figure 4.6: Second test for the calibration

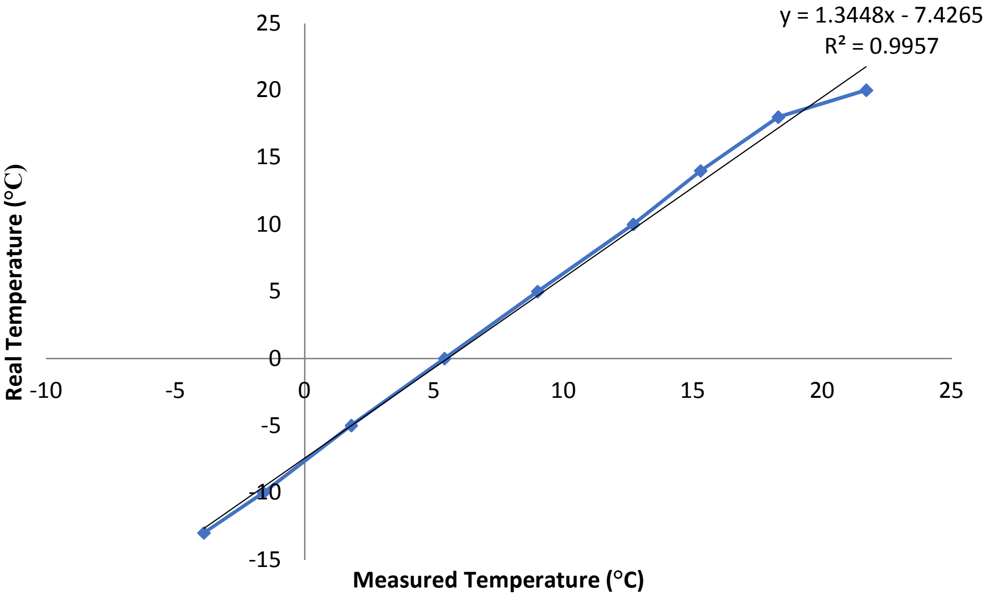


Figure 4.7: Third test for the calibration

The values of the cold flow properties are graphically shown in Figure 4.8. The curves shown in this graph give the bath temperature and the cooling curve of the cooling flow characteristics of the biodiesel mixture sample (B80) using the data logger during cooling and heating cycle scans. Second derivative of temperature (as a function of time) is calculated and plotted to measure the value of the cold flow properties. It was observed that the CP, PP and CFPP values obtained from the observation data in the graph and the cooling curve were respectively 7 ^{o}C, 2 ^{o}C and 4 ^{o}C and almost equal.

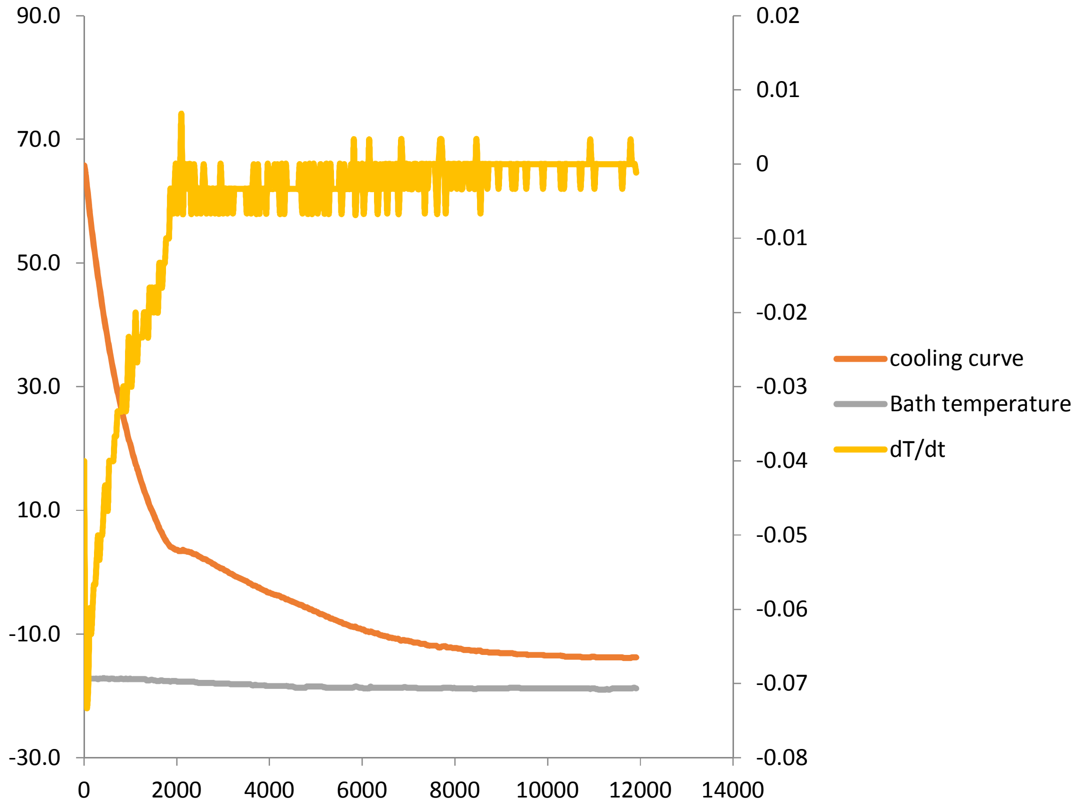


Figure 4.8: Cooling curve of the cooling bath setup

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Biodiesel has many uses besides its use as engine fuel. There are the main ones; in the model plane, in central heating boilers, stoves, lanterns and other heaters, in engine parts oil and grease removal, as generator fuel, as a multipurpose lubricant, as hydraulic fluid, as railway lubricant, in brick production and pottery. Today, commercial vehicles can use 20% biodiesel blend without any changes. If more than 20% biodiesel blend is used, copper pipes should be used instead of plastic pipes. That is, the ignition and fuel equipment should be replaced with materials produced in accordance with biodiesel fuel.

B80 sample experiments were stored in the mechanical engineering laboratory at Near East University at a constant temperature of 40 ^{o}C. As mentioned in previous chapters, the kinematic viscosity of this sample was measured between 5 ^{o}C and 90 ^{o}C in a relationship with ASTM D445-09. The kinematic viscosity at 40 ^{o}C should be within the range of 1.9-6.0 mm^2/s according to this standard. In addition, the density of the biodiesel sample was measured using pycnometer according to ASTM D941-88 to 5 ^{o}C at 90 ^{o}C. The density should be in the range of 880-920 kg/m^3 according to this standard at 15 ^{o}C. The total acid number and oxidation stability that were obtained for this sample were tested using test methods according to ASTM D664-04 (2017) and EN 15751: 2014 respectively. The petrochemical laboratory that performs these analyses is in Southern Cyprus.

The results of the kinematic viscosity and Density tests for the B80 D20 biodiesel blend used in this study can be used in vehicles as they are within the range of the standards. The biodiesel blend can be used in the winter months and no problems are encountered in the first run of the engine. The cold flow properties vary according to the country weather conditions. Because of this, the blend suitable for weather conditions is used. If the biodiesel mixture is not used properly in cold weather conditions, the fuel will gel and block the filters and cause the fuel flow to cease. Also, biodiesel has a good lubrication ability and does not cause high-level engine wear. The biggest advantage of biodiesel is that the exhaust emission values are low.

Results collected are below:

- As the storage period of the tested sample increase, its viscosity was found to be increasing linearly.
- Inversely proportional relationship between the test temperature and kinematic viscosity of the sample can be observed.
- It is noticed that there is an increasing effect of storage period at low temperatures on the viscosity where it starts to decrease at higher temperature.
- Because of the crystallization of the blend below 5 ^{o}C, it is not possible to measure kinematic viscosity below this temperature.
- The density of tested sample was found to be increasing as the storage period increase. While, an inversely proportional relationship between the test temperature and density of the sample is observed.
- The total acid number of tested sample was found to be decreasing as the storage period increase. In contrast oxidation stability increases as the storage period increasing. The smaller the acid value, the higher the quality. Oxidation is chemical event causing adherent and filterable insolubles to form.
- The CP, PP and CFPP values obtained from the observational data on the graph of the cold flow properties and the cooling curve were 7 ^{o}C, 2 ^{o}C and 4 ^{o}C given are almost equal, respectively. The only test we have done through observation in this study is the cloud point end result.

5.2 Further Recommendations

The measurements made on the biodiesel sample could be increased and the behaviours displayed on the longer storage periods could be followed. The storage period of experiments on the total number of acids and oxidation stability could be increased to measure the long-term effects. In addition, there are some important features that affect bio distillation and researches on these. For example, cetane number, sulfated ash, water content and iodine value. As a result, storage time can be increased to examine more precise result in long-term condition, and different results can be obtained by increasing the storage temperature. Different results can be obtained if the biodiesel storage is changed in a dark place and in a metal or plastic container.

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APPENDICES

Designation: D6751 – 09a

Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels[1]

This standard is issued under the fixed designation D6751; 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.

1. Scope*

1.1 This specification covers biodiesel (B100) Grades S15 and S500 for use as a blend component with middle distillate fuels.

1.2 This specification prescribes the required properties of diesel fuels at the time and place of delivery. The specification requirements may be applied at other points in the production and distribution system when provided by agreement between the purchaser and the supplier.

1.3 Nothing in this specification shall preclude observance of federal, state, or local regulations which may be more restrictive.

NOTE 1—The generation and dissipation of static electricity can create problems in the handling of distillate fuel oils with which biodiesel may be blended. For more information on the subject, see Guide D4865.

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

1.4.1 *Exception*—In Annex A1, the values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D189 Test Method for Conradson Carbon Residue of Petroleum Products

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D524 Test Method for Ramsbottom Carbon Residue of Petroleum Products

D613 Test Method for Cetane Number of Diesel Fuel Oil

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D874 Test Method for Sulfated Ash from Lubricating Oils and Additives

D974 Test Method for Acid and Base Number by Color-Indicator Titration

D975 Specification for Diesel Fuel Oils

D976 Test Method for Calculated Cetane Index of Distillate Fuels

D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure

D1266 Test Method for Sulfur in Petroleum Products (Lamp Method)

D1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)

D2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)

D2500 Test Method for Cloud Point of Petroleum Products

D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry

D2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge

D2880 Specification for Gas Turbine Fuel Oils

D3117 Test Method for Wax Appearance Point of Distillate Fuels

D3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry

D3242 Test Method for Acidity in Aviation Turbine Fuel

D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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

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[1] This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0 on Burner, Diesel, Non-Aviation Gas Turbine, and Marine Fuels.

Current edition approved Dec. 1, 2009. Published February 2010. Originally approved in 1999 as PS 121–99. Adopted as a standard in 2002 as D6751–02. Last previous edition approved in 2009 as D6751–09. DOI: 10.1520/D6751-09a.

[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.

D4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry

D4530 Test Method for Determination of Carbon Residue (Micro Method)

D4737 Test Method for Calculated Cetane Index by Four Variable Equation

D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems

D4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry

D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration

D5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence

D5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)

D5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)

D5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)

D6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D6450 Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester

D6469 Guide for Microbial Contamination in Fuels and Fuel Systems

D6584 Test Method for Determination of Free and Total Glycerin in B-100 Biodiesel Methyl Esters by Gas Chromatography

D6890 Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber

D7039 Test Method for Sulfur in Gasoline and Diesel Fuel by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry

D7397 Test Method for Cloud Point of Petroleum Products (Miniaturized Optical Method)

2.2 *Government Standard:*

40 CFR Part 79 Registration of Fuels and Fuel Additives Section 211(b) Clean Air Act[3]

2.3 *Other Documents:*[4]

UOP 389 Trace Metals in Oils by Wet Ashing and ICP-OES

UOP 391–91 Trace Metals in Petroleum Products or Organics by AAS

EN 14112 Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of oxidation stability (Accelerated oxidation test)[5]

EN 14110 Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of methanol content[5]

EN 14538 Fat and oil derivatives—Fatty acid methyl esters (FAME)—Determination of Ca, K, Mg and Na content by optical emission spectral analysis with inductively coupled plasma (ICP OES)[5]

3. Terminology

3.1 *Definitions:*

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

3.1.1.1 *Discussion*—*biodiesel*, as defined above, is registered with the U.S. EPA as a fuel and a fuel additive under Section 211(b) of the Clean Air Act. There is, however, other usage of the term biodiesel in the marketplace. Due to its EPA registration and the widespread commercial use of the term biodiesel in the U.S. marketplace, the term biodiesel will be maintained for this specification.

3.1.1.2 *Discussion*—Biodiesel is typically produced by a reaction of a vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-alkyl esters and glycerin, which is removed. The finished biodiesel derives approximately 10 % of its mass from the reacted alcohol. The alcohol used in the reaction may or may not come from renewable resources.

3.1.2 *biodiesel blend (BXX), n*—blend of biodiesel fuel with diesel fuel oils.

3.1.2.1 *Discussion*—In the abbreviation BXX, the XX represents the volume percentage of biodiesel fuel in the blend.

3.1.3 *biodiesel fuel, n*—synonym for *biodiesel.*

3.1.4 *diesel fuel, n*—middle petroleum distillate fuel.

3.1.5 *free glycerin, n*—a measure of the amount of glycerin remaining in the fuel.

3.1.6 *Grade S15 B100, n*—a grade of biodiesel meeting ASTM Specification D6751 and having a sulfur specification of 15 ppm maximum.

3.1.7 *Grade S500 B100, n*—a grade of biodiesel meeting ASTM Specification D6751 and having a sulfur specification of 500 ppm maximum.

3.1.8 *middle distillate fuel, n*—kerosines and gas oils boiling between approximately 150°C and 400°C at normal atmospheric pressure and having a closed-cup flash point above 38°C.

3.1.9 *total glycerin, n*—the sum of the free glycerin and the glycerin portion of any unreacted or partially reacted oil or fat.

4. Requirements

4.1 The biodiesel specified shall be mono-alkyl esters of long chain fatty acids derived from vegetable oils and animal fats.

4.2 Unless otherwise specified, samples for analysis shall be taken by the procedure described in Practices D4057 or D4177.

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[3] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

[4] Available from ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA. Visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org.

[5] Available from the National CEN Members listed on the CEN website (www.cenorm.be) or from the CEN/TC19 secretariat (astm@nen.nl).

4.3 The biodiesel specified shall conform to the detailed requirements shown in Table 1.

NOTE 2—A considerable amount of experience exists in the U.S. with a 20 % blend of biodiesel, primarily produced from soybean oil, with 80 % diesel fuel (B20). Experience with biodiesel produced from animal fat and other oils is similar. Experience with B20 and lower blends in other applications is not as prevalent. Although biodiesel (B100) can be used, blends of over 20 % biodiesel with diesel fuel (B20) should be evaluated on a case by case basis until further experience is available.

NOTE 3—The user should consult the equipment manufacturer or owner's manual regarding the suitability of using biodiesel or biodiesel blends in a particular engine or application.

5. Test Methods

5.1 The requirements enumerated in this specification shall be determined in accordance with the following methods.

5.1.1 *Flash Point*—Test Methods D93, except where other methods are prescribed by law. Test Methods D3828 or D6450 can also be used. The precision and bias of Test Methods D3828 and D6450 with biodiesel is not known and is currently under investigation. Test Methods D93 shall be the referee method.

5.1.2 *Water and Sediment*—Test Method D2709. Test Method D1796 may also be used. Test Method D2709 shall be the referee method. The precision and bias of these test methods with biodiesel is not known and is currently under investigation.

5.1.3 *Viscosity*—Test Method D445.

5.1.4 *Sulfated Ash*—Test Method D874.

5.1.5 *Oxidation Stability*—Test Method EN 14112.

5.1.6 *Sulfur*—Test Method D5453. Test Method D7039 may also be used. Other test methods may also be suitable for determining up to 0.05 % (500 ppm) sulfur in biodiesel fuels such as Test Methods D1266, D2622, D3120 and D4294 but may provide falsely high results (see X1.5) although their precision and bias with biodiesel is unknown. Test Method D5453 shall be the referee test method.

5.1.7 *Corrosion*—Test Method D130, 3 h test at 50°C.

5.1.8 *Cetane Number*—Test Method D613. Test Method D6890 may also be used. Test Method D613 shall be the referee method.

5.1.9 *Cloud Point*—Test Method D2500. Test Method D5771, D5772, D5773, or D7397 may also be used. Test Method D3117 may also be used because it is closely related. Test Method D2500 shall be the referee test method. The precision and bias of Test Method D3117 for biodiesel is not known and is currently under investigation.

5.1.10 *Acid Number*—Test Method D664. Test Methods D3242 or D974 may also be used. Test Method D664 shall be the referee test method.

5.1.11 *Carbon Residue*—Test Method D4530. A100 % sample shall replace the 10 % residual, with percent residue in the original sample reported using the 10 % residual calculation (see X1.9.1). Test Methods D189 or D524 may also be used. Test Method D4530 shall be the referee method.

5.1.12 *Total Glycerin*—Test Method D6584.

5.1.13 *Free Glycerin*—Test Method D6584.

5.1.14 *Phosphorus Content*—Test Method D4951.

5.1.15 *Distillation Temperature, Reduced Pressure*—Test Method D1160.

5.1.16 *Calcium and Magnesium, combined*—Test Method EN 14538. Test Method UOP 389 may also be used. Test Method EN 14538 shall be the referee test method.

TABLE 1 Detailed Requirements for Biodiesel (B100) (All Sulfur Levels)

Property	Test Method[A]	Grade S15 Limits	Grade S500 Limits	Units
Calcium and Magnesium, combined	EN 14538	5 max	5 max	ppm (µg/g)
Flash point (closed cup)	D93	93 min	93 min	°C
Alcohol control				
One of the following must be met:				
1. Methanol content	EN 14110	0.2 max	0.2 max	mass %
2. Flash point	D93	130 min	130 min	°C
Water and sediment	D2709	0.050 max	0.050 max	% volume
Kinematic viscosity, 40°C	D445	1.9-6.0[B]	1.9–6.0[B]	mm^2/s
Sulfated ash	D874	0.020 max	0.020 max	% mass
Sulfur[C]	D5453	0.0015 max (15)	0.05 max (500)	% mass (ppm)
Copper strip corrosion	D130	No. 3 max	No. 3 max	
Cetane number	D613	47 min	47 min	
Cloud point	D2500	Report[D]	Report[D]	°C
Carbon residue[E]	D4530	0.050 max	0.050 max	% mass
Acid number	D664	0.50 max	0.50 max	mg KOH/g
Cold soak filterability	Annex A1	360 max[F]	360 max[F]	seconds
Free glycerin	D6584	0.020 max	0.020 max	% mass
Total glycerin	D6584	0.240 max	0.240 max	% mass
Phosphorus content	D4951	0.001 max	0.001 max	% mass
Distillation temperature, Atmospheric equivalent temperature, 90 % recovered	D1160	360 max	360 max	°C
Sodium and Potassium, combined	EN 14538	5 max	5 max	ppm (µg/g)
Oxidation stability	EN 14112	3 minimum	3 minimum	hours

[A] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 5.1.

[B] See X1.3.1. The 6.0 mm^2/s upper viscosity limit is higher than petroleum based diesel fuel and should be taken into consideration when blending.

[C] Other sulfur limits can apply in selected areas in the United States and in other countries.

[D] The cloud point of biodiesel is generally higher than petroleum based diesel fuel and should be taken into consideration when blending.

[E] Carbon residue shall be run on the 100 % sample (see 5.1.11).

[F] B100 intended for blending into diesel fuel that is expected to give satisfactory vehicle performance at fuel temperatures at or below –12°C shall comply with a cold soak filterability limit of 200 s maximum.

5.1.17 *Sodium and Potassium, combined*—Test Method EN 14538. Test Method UOP 391 may also be used. Test Method EN 14538 shall be the referee test method.

5.1.18 *Cold Soak Filterability*—The test method in Annex A1 shall be used to determine the cold soak filterability. B100 intended for blending into diesel fuels that is expected to give satisfactory vehicle performance at fuel temperatures at or below –12°C shall comply with a cold soak filtration limit of 200 s maximum. A cold soak filterability standard test method is under development.

NOTE 4—Interim precision information is provided in Annex A1 for the cold soak filterability test to give the user some indication of the repeatability and reproducibility expected.

6. Workmanship

6.1 The biodiesel fuel shall be visually free of undissolved water, sediment, and suspended matter.

7. Keywords

7.1 alternative fuel; biodiesel fuel; diesel fuel oil; fuel oil; renewable resource

ANNEX

(Mandatory Information)

A1. DETERMINATION OF FUEL FILTER BLOCKING POTENTIAL OF BIODIESEL (B100) BLEND STOCK BY COLD SOAK LABORATORY FILTRATION

A1.1 Scope

A1.1.1 This test method covers the determination by filtration time after cold soak of the suitability for a Biodiesel (B100) Blend Stock for blending with middle distillates to provide adequate low temperature operability performance to at least the cloud point of the finished blend.

A1.1.2 The interim precision of this test method has been determined.

A1.1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

A1.1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

A1.2 Referenced Documents

A1.2.1 *ASTM Standards*:[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems

D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration

A1.3 Terminology

A1.3.1 *Definitions*:

A1.3.1.1 *biodiesel, n*—fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.

A1.3.1.2 *bond, v*—to connect two parts of a system electrically by means of a conductive wire to eliminate voltage differences.

A1.3.1.3 *ground, v*—to connect electrically with earth.

A1.3.2 *Definitions of Terms Specific to this Standard*:

A1.3.2.1 *filtered flushing fluids, n*—either of two solvents, heptane or 2,2, 4–trimethylpentane, filtered through a nominal 0.45 µm glass fiber filter.

A1.3.2.2 *glass fiber filter, n*—the 0.7 µm glass fiber filters used in this test method.

A1.3.3 *Abbreviations*:

A1.3.3.1 *CSFT*—cold soak filtration test.

A1.4 Summary of Test Method

A1.4.1 In this test method, 300 mL of biodiesel (B100) is stored at 4.4°C (40°F) for 16 h, allowed to warm to 20 to 22°C (68 to 72°F), and vacuum filtered through a single 0.7 µm glass fiber filter.

A1.4.2 In this test method, the filtration time is reported in seconds.

A1.5 Significance and Use

A1.5.1 Some substances that are soluble or appear to be soluble in biodiesel at room temperature will, upon cooling or standing at room temperature for extended periods, come out of solution. These substances can cause filter plugging. This test method provides an accelerated means of assessing the propensity for these substances to plug filters.

A1.5.1.1 Fuels that give short filtration times are expected to give satisfactory operation down to the cloud point of biodiesel blends.

A1.5.2 The test method can be used in specifications as a means of controlling levels of minor filter plugging components in biodiesel and biodiesel blends.

A1.6 Apparatus

A1.6.1 *Filtration System*—Arrange the following components as shown in Fig. A1.1.

A1.6.1.1 *Funnel and Funnel Base*, with a stainless steel filter support for a 47–mm diameter glass fiber filter and a locking ring or spring action clip capable of receiving 300 mL.

NOTE A1.1—Sintered glass supports were found to give much higher filtration times during initial studies and should not be used.

A1.6.1.2 *Ground/Bond Wire*, 0.912 to 2.59 mm (No. 10 through No. 19) bare-stranded flexible stainless steel or copper installed in the flasks and grounded as shown in Fig. A1.1.

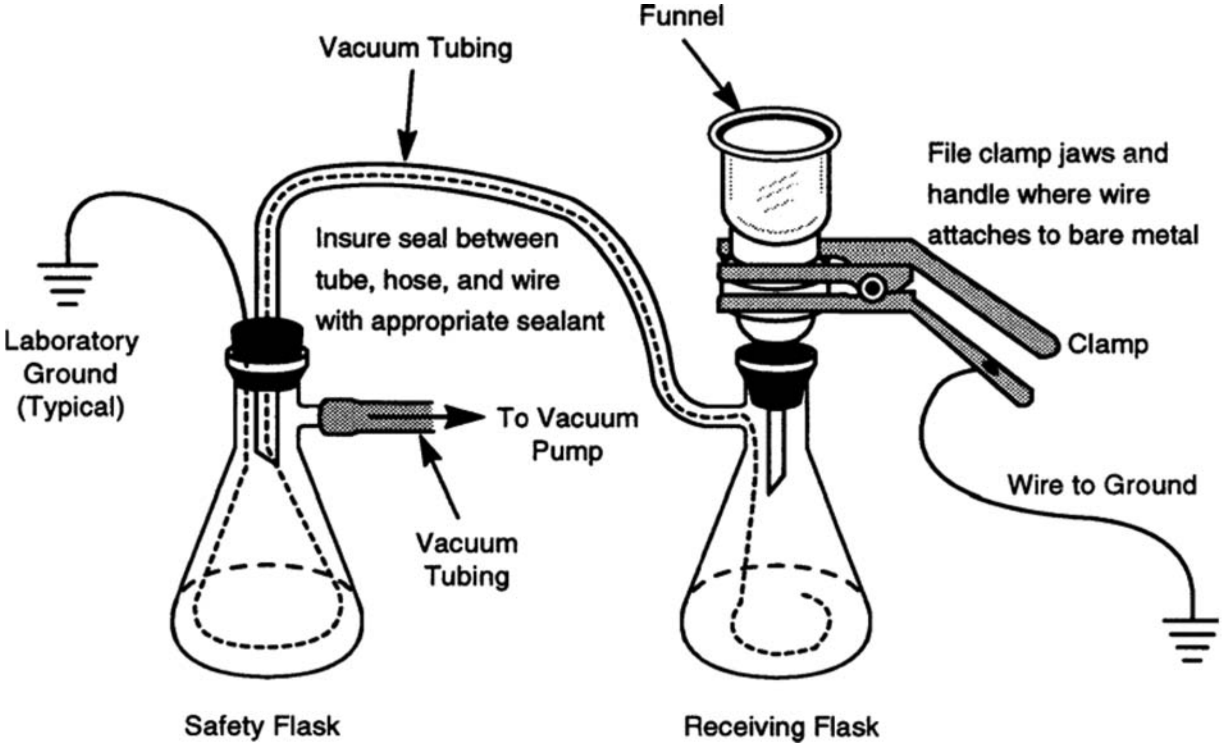

FIG. A1.1 Schematic of Filtration System

NOTE A1.2—The electrical bonding apparatus described in Test Method D5452 or other suitable means of electrical grounding which ensure safe operation of the filtration apparatus and flask can be used. If the filtrate is to be subsequently tested for stability it is advisable not to use copper as copper ions catalyze gum formation during the stability test.

A1.6.1.3 *Receiving Flask*, 1-L borosilicate glass vacuum filter flask, into which the filtration apparatus fits, equipped with a sidearm to connect to the safety flask.

A1.6.1.4 *Safety Flask*, 1-L borosilicate glass vacuum filter flask equipped with a sidearm to connect the vacuum system. A fuel and solvent resistant rubber hose, through which the grounding wire passes, shall connect the sidearm of the receiving flask to the tube passing through the rubber stopper in the top of the safety flask.

A1.6.1.5 *Vacuum System*, a vacuum system capable of producing a vacuum of 70 to 100 kPa below atmospheric pressure when measured at the receiving flask. A mechanical vacuum pump may be used if it has this capability.

NOTE A1.3—Water aspirated vacuum will not provide relative vacuum within the prescribed range.

A1.6.2 *Other Apparatus*:

A1.6.2.1 *Forceps*, approximately 12-cm long, flat-bladed, with non-serrated, non-pointed tips.

A1.6.2.2 *Graduated Cylinders*, to contain at least 0.5 L of fluid and marked at 10-mL intervals. Graduated cylinders, 100-mL, may be required for samples which filter slowly.

A1.6.2.3 *Petri Dishes*, approximately 12.5 cm in diameter, with removable glass supports for glass fiber filters.

NOTE A1.4—Small watch glasses, approximately 5 to 7 cm in diameter, have also been found suitable to support the glass fiber filters.

NOTE A1.5—B100 will dissolve some plastics. This can cause the filters to adhere to the plastic.

A1.6.2.4 *Glass Fiber Filters*, plain, 47-mm diameter, nominal pore size 0.7-µm.

A1.6.2.5 *Protective Cover*, polyethylene film or clean aluminum foil.

A1.6.2.6 *Liquid or Air Bath or Chamber*, capable of sustaining a temperature of 4.4 ± 1.1°C (40 ± 2°F) for 16 h.

A1.6.2.7 *Timer*, capable of displaying elapsed times of at least 900 s to the nearest 0.1 s.

A1.7 Reagents and Materials

A1.7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

A1.7.2 *Flushing Fluids*—Flushing fluids are not required for the test as the filter is not weighed. However, heptane or isooctane may be used to wash the apparatus after filtration to remove any residue. Alternatively soap and water may be used in accordance with A1.7.3.

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

A1.7.2.1 *Heptane*, (**Warning**—Flammable).

A1.7.2.2 *2,2,4-trimethylpentane (isooctane)*, (**Warning**—Flammable).

A1.7.3 *Liquid or Powder Detergent*, water-soluble, for cleaning glassware.

A1.8 Preparation of Apparatus and Sample Containers

A1.8.1 Clean all components of the filtration apparatus using the reagents described in A1.7.2 and A1.7.3.

A1.8.1.1 Remove any labels, tags, and so forth.

A1.9 Sampling

A1.9.1 The sample container should be 500 ± 15 mL in volume and have a screw-on cap with an inert liner. Glass containers are preferred to facilitate a visual inspection of the contents and the container before and after filling. Glass containers also allow for visual inspection of the container, after the sample is emptied, to confirm complete emptying of the container. Epoxy-lined sample cans, polytetrafluoroethylene (PTFE) bottles, and high density linear polyethylene bottles have also been found suitable as sample containers but are less desirable since visual inspection of the interior of the container is more difficult.

A1.9.2 Precautions to avoid sample contamination shall include selection of an appropriate sampling point. It is preferred to obtain samples dynamically from a sampling loop in a distribution line, or from the flushing line of a field sampling kit. Ensure that the line to be sampled is flushed with fuel before collecting the sample.

A1.9.2.1 Use clean sample containers.

A1.9.2.2 Keep a clean protective cover over the top of the sample container until the cap is installed. Similarly protect the funnel opening of the assembled filtration apparatus with a clean protective cover until ready for use.

A1.9.2.3 Where it is desirable or only possible to obtain samples from static storage, follow the procedures given in Practice D4057 or equivalent, taking precautions for cleanliness of all equipment used. The sample should pass through a minimum number of intermediate containers prior to placement in the prepared container.

A1.9.2.4 Samples obtained from static storage can give results that are not representative of the bulk contents of the tank because of particulate matter settling. Where possible, the contents of the tank should be circulated or agitated before sampling, or the sampling should be performed shortly after a tank has been filled.

A1.9.3 Visually inspect the sample container before taking the samples to verify that there are no visible particles present inside the container. Fill the sample container to contain 300 mL. Protect the fuel sample from prolonged exposure to light by wrapping the container in aluminum foil or storing it in the dark to reduce the possibility of particulate formation by light-promoted reactions. Do not transfer the fuel sample from its original sample container into an intermediate storage container. If the original sample container is damaged or leaking, then a new sample shall be obtained.

A1.9.3.1 If a 500-mL bottle is not available, or the sample has already been received in a container not suitable for this test, follow A1.9.5.

A1.9.4 Analyze fuel samples as soon as possible after sampling.

A1.9.4.1 Upon receipt of a Biodiesel Blend Stock (B100) sample, the entire sample shall be heated to 40°C for at least 3 h under an inert atmosphere to erase any thermal history and to dissolve any solids that might have precipitated during transit unless it is known that the sample has never been cooled below 20°C. If the sample has never been exposed to temperatures below 20°C then proceed to A1.9.5.

A1.9.4.2 After heating for the required time, allow the sample to sit for 24 h at a temperature no lower than 20°C.

A1.9.5 Shake the sample vigorously for 1 min, and transfer 300 mL to a clean fresh 500 ± 15 mL bottle.

A1.10 Preparation of Glass Fiber Filter

A1.10.1 Each filtration uses one filter. The glass fiber filter used for each individual test shall be identified by marking the petri dishes used to hold and transport the filters.

A1.10.2 Clean all glassware used in preparation of glass fiber filter as described in A1.8.1.

A1.10.3 Using forceps, place the filters on clean glass support rods or watch glasses in petri dish.

A1.10.4 Place the petri dish with its lid slightly ajar in a drying oven at 90 ± 5°C, and leave it for 30 min.

A1.10.5 Remove the petri dish from the drying oven. Keep the petri dish cover ajar, such that the filter is protected from contamination from the atmosphere. Allow 30 min for the filter to come to equilibrium with room air temperature and humidity.

A1.10.6 Using clean forceps, place the filter centrally on the filter support of the filtration apparatus (see Fig. A1.1). Install the funnel and secure with locking ring or spring clip. Do not remove the plastic film from the funnel opening until ready to start filtration.

A1.11 Procedure

A1.11.1 Place 300 mL of sample in a glass 500–mL bottle, and set in a liquid or air bath or chamber at 4.4 ± 1.1°C (40 ± 2°F) for 16 ± 0.5 h.

A1.11.2 After the 16-h cold soak is completed, allow the sample to come back to room temperature at 20 to 22°C (68 to 72°F) on its own without external heating. The sample shall be completely liquid before filtration. The sample shall be filtered within 1 h after reaching 20 to 22°C (68 to 72°F).

A1.11.3 Complete assembly of the receiving flask, 0.7 µm glass fiber filter and funnel as a unit (see Fig. A1.1) before swirling the sample. To minimize operator exposure to fumes, the filtering procedure should be performed in a fume hood.

A1.11.4 Start the vacuum system. Record the pressure in the system after 1 min of filtration. The vacuum shall be between 71.1 and 84.7 kPa (21 and 25 in. Hg) below atmospheric pressure. If the vacuum is not within the specified range, make adjustments to the vacuum system.

A1.11.5 Thoroughly clean the outside of the sample container in the region of the cap by wiping it with a damp, lint-free cloth. Swirl the container vigorously for about 2 to 3 s to dislodge any particles that may have adhered to the walls of the container.

A1.11.6 Immediately after swirling, pour the entire contents of the sample container into the filtration funnel and simultaneously start the timer. The entire contents of the sample container shall be filtered through the glass fiber filter to ensure a correct measure of the contamination in the sample.

NOTE A1.6—Take care not to shake the sample vigorously, as this could cause some of the solids to go back into solution.

A1.11.7 If the filtration is not complete when 720 s (12 min) has elapsed, turn off the vacuum system and record the duration of the filtration to the nearest second. Record the pressure in the system and the volume filtered just before the termination of the filtration.

A1.12 Reporting

A1.12.1 Report the time for the 300–mL B100 to be completely filtered as B100 filtration time in seconds.

A1.12.2 If the filtration of the 300 mL failed to be completed after 720 s, report the volume that was filtered after 720 s.

A1.13 Precision and Bias

A1.13.1 *Precision*—The precision of this test method for B100 filtration has not yet been determined.

A1.13.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material for B100 filtration has not yet been determined.

A1.13.1.2 *Reproducibility*—The difference between the two single and independent results obtained by different operators working in different laboratories on identical test material for B100 filtration has not yet been determined.

A1.13.1.3 *Interim Precision*—Repeatability and reproducibility determinations were made using data from the ASTM Biodiesel Low Temperature Operability Task Force. The analysis of the data is the subject of a research report, RR:D02-1649.[7] The report is an attempt to supply such an analysis based on well-established methodologies. Subsequent to test method publication a more thorough round robin is planned.

	200 s	360 s
Repeatability	34.0	61.0
Reproducibility	115.9	208.1

NOTE A1.7—The degree of freedom associated with the repeatability estimate from this round robin study is 25 for repeatability which is below 30 but acceptable. The degree of freedom associated with the reproducibility estimate from this round robin study is 10 and below acceptable limits. For that reason only the repeatability is included in A1.13.1.4. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual repeatability/reproducibility may be significantly different than these estimates. An ASTM ILS will be conducted in the future.

A1.13.1.4 The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material for B100 filtration would in the long run, in the normal and correct operation of this test method, exceed 0.1689(X + 1.2018) time(s) only in one case in twenty.

A1.13.2 *Bias*—The procedure given for the determination of B100 filtration time has no bias because the value of the filtration time is defined in terms of this test method.

A1.14 Keywords

A1.14.1 biodiesel; diesel fuel; glass fiber filter; biodiesel; filter blocking potential, cold soak filtration test, CSFT, biodiesel blend; laboratory filtration; glass fiber filter; low temperature operability, middle distillate fuel.

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1649.

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF PROPERTIES SPECIFIED FOR BIODIESEL FUEL

X1.1 Introduction

X1.1.1 The properties of commercial biodiesel fuel depends upon the refining practices employed and the nature of the renewable lipids from which it is produced. Biodiesel, for example, can be produced from a variety of vegetable oils or animal fats which produce similar volatility characteristics and combustion emissions with varying cold flow properties.

X1.1.2 The significance of the properties in this appendix are based primarily on the commercial use of biodiesel in on-road and off-road diesel engine applications. Some of the properties may take on other significance if biodiesel is used as a fuel or blending component in other applications. See the respective finished product specifications for additional information on significance of properties of those applications.

X1.2 Flash Point

X1.2.1 The flash point, as specified, is not directly related to engine performance. It is, however, of importance in connection with legal requirements and safety precautions involved in fuel handling and storage that are normally specified to meet insurance and fire regulations.

X1.2.2 The flash point for biodiesel has been set at 93°C (200°F) minimum, so biodiesel falls under the non-hazardous category under National Fire Protection Association codes.

X1.3 Viscosity

X1.3.1 For some engines it may be advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum allowable viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system. The upper limit for the viscosity of biodiesel (6.0 mm^2/s at 40°C) is higher than the maximum allowable viscosity in Specification D975 Grade 2-D and 2-D low sulfur (4.1 mm/s at 40°C). Blending biodiesel with diesel fuel close to its upper limit could result in a biodiesel blend with viscosity above the upper limits contained in Specification D975.

X1.4 Sulfated Ash

X1.4.1 Ash-forming materials may be present in biodiesel in three forms: (*1*) abrasive solids, (*2*) soluble metallic soaps, and (*3*) unremoved catalysts. Abrasive solids and unremoved catalysts can contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear but may contribute to filter plugging and engine deposits.

X1.5 Sulfur

X1.5.1 The effect of sulfur content on engine wear and deposits appears to vary considerably in importance and depends largely on operating conditions. Fuel sulfur can also affect emissions control systems performance and various limits on sulfur have been imposed for environmental reasons. B100 is essentially sulfur-free.

NOTE X1.1—Test Method D5453 should be used with biodiesel. Use of other test methods may provide falsely high results when analyzing B100 with extremely low sulfur levels (less than 5 ppm). Biodiesel sulfur analysis from RR:D02-1480[8], *Biodiesel Fuel Cetane Number Testing Program, January-April, 1999*, using Test Method D2622 yielded falsely high results due to the presence of the oxygen in the biodiesel. Sulfur results using Test Method D2622 were more accurate with B20 than with B100 due to the lower oxygen content of B20. Potential improvements to Test Method D2622 may provide more accurate values in the future.

X1.6 Copper Strip Corrosion

X1.6.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system. The presence of acids or sulfur-containing compounds can tarnish the copper strip, thus indicating the possibility for corrosion.

X1.7 Cetane Number

X1.7.1 Cetane number is a measure of the ignition quality of the fuel and influences white smoke and combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions.

X1.7.2 The calculated cetane index, Test Methods D976 or D4737, may not be used to approximate the cetane number with biodiesel or its blends. There is no substantiating data to support the calculation of cetane index with biodiesel or biodiesel blends.

X1.8 Cloud Point

X1.8.1 Cloud point is of importance in that it defines the temperature at which a cloud or haze of crystals appears in the fuel under prescribed test conditions which generally relates to the temperature at which crystals begin to precipitate from the fuel in use. Biodiesel generally has a higher cloud point than petroleum based diesel fuel. The cloud point of biodiesel and its impact on the cold flow properties of the resulting blend should be monitored by the user to ensure trouble-free operation in cold climates. For further information, consult Appendix X4 of Specification D975.

X1.9 Carbon Residue

X1.9.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil. While not directly correlating with engine deposits, this property is considered an approximation. Although biodiesel is in the distillate boiling range, most biodiesels boil at approximately the same temperature and it is difficult to leave a 10 % residual upon distillation. Thus, a 100 % sample is used to replace the 10 % residual sample, with the calculation executed as if it were the 10 % residual. Parameter E (final weight flask charge/original weight flask charge) in 8.1.2 of Test Method D4530-93 is a constant 20/200.

X1.10 Acid Number

X1.10.1 The acid number is used to determine the level of free fatty acids or processing acids that may be present in biodiesel. Biodiesel with a high acid number has been shown to increase fueling system deposits and may increase the likelihood for corrosion.

NOTE X1.2—Acid number measures a different phenomenon for biodiesel than petroleum based diesel fuel. The acid number for biodiesel measures free fatty acids or degradation by-products not found in petroleum based diesel fuel. Increased recycle temperatures in new fuel system designs may accelerate fuel degradation which could result in high acid values and increased filter plugging potential.

X1.11 Free Glycerin

X1.11.1 The free glycerin method is used to determine the level of glycerin in the fuel. High levels of free glycerin can cause injector deposits, as well as clogged fueling systems, and result in a buildup of free glycerin in the bottom of storage and fueling systems.

X1.12 Total Glycerin

X1.12.1 The total glycerin method is used to determine the level of glycerin in the fuel and includes the free glycerin and the glycerine portion of any unreacted or partially reacted oil or fat. Low levels of total glycerin ensure that high conversion of the oil or fat into its mono-alkyl esters has taken place. High levels of mono-, di-, and triglycerides can cause injector deposits and may adversely affect cold weather operation and filter plugging.

X1.13 Phosphorus Content

X1.13.1 Phosphorus can damage catalytic converters used in emissions control systems and its level must be kept low. Catalytic converters are becoming more common on diesel-powered equipment as emissions standards are tightened, so low phosphorus levels will be of increasing importance. Biodiesel produced from U.S. sources has been shown to have

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1480.

low phosphorus content (below 1 ppm) and the specification value of 10 ppm maximum is not problematic. Biodiesel from other sources may or may not contain higher levels of phosphorus and this specification was added to ensure that all biodiesel, regardless of the source, has low phosphorus content.

X1.14 Reduced Pressure Distillation

X1.14.1 Biodiesel exhibits a boiling point rather than a distillation curve. The fatty acids chains in the raw oils and fats from which biodiesel is produced are mainly comprised of straight chain hydrocarbons with 16 to 18 carbons that have similar boiling temperatures. The atmospheric boiling point of biodiesel generally ranges from 330 to 357°C, thus the specification value of 360°C is not problematic. This specification was incorporated as an added precaution to ensure the fuel has not been adulterated with high boiling contaminants.

NOTE X1.3—The density of biodiesel meeting the specifications in Table 1 falls between 0.86 and 0.90, with typical values falling between 0.88 and 0.89. Since biodiesel density falls between 0.86 and 0.90, a separate specification is not needed. The density of raw oils and fats is similar to biodiesel, therefore use of density as an expedient check of fuel quality may not be as useful for biodiesel as it is for petroleum based diesel fuel. This section has been added to provide users and engine interests with this information.

NOTE X1.4—In certain items of fuel injection equipment in compression ignition engines, such as rotary/distributor fuel pumps and injectors, the fuel functions as a lubricant as well as a source for combustion. Blending biodiesel fuel with petroleum based compression-ignition fuel typically improves fuel lubricity.

X1.15 Alcohol Control

X1.15.1 Alcohol control is to limit the level of unreacted alcohol remaining in the finished fuel. This can be measured directly by the volume percent alcohol or indirectly through a high flash point value.

X1.15.2 The flash point specification, when used for alcohol control for biodiesel, is intended to be 100°C minimum, which has been correlated to 0.2 vol % alcohol. Typical values are over 160°C. Due to high variability with Test Method D93 as the flash point approaches 100°C, the flash point specification has been set at 130°C minimum to ensure an actual value of 100°C minimum. Improvements and alternatives to Test Method D93 are being investigated. Once complete, the specification of 100°C minimum may be reevaluated for alcohol control.

X1.16 Calcium and Magnesium

X1.16.1 Calcium and magnesium may be present in biodiesel as abrasive solids or soluble metallic soaps. Abrasive solids can contribute to injector, fuel pump, piston, and ring wear, as well as to engine deposits. Soluble metallic soaps have little effect on wear, but they may contribute to filter plugging and engine deposits. High levels of calcium and magnesium compounds may also be collected in exhaust particulate removal devices, are not typically removed during passive or active regeneration, and can create increased back pressure and reduced time to service maintenance.

X1.17 Sodium and Potassium

X1.17.1 Sodium and potassium may be present in biodiesel as abrasive solids or soluble metallic soaps. Abrasive solids can contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear, but they may contribute to filter plugging and engine deposits. High levels of sodium or potassium compounds may also be collected in exhaust particulate removal devices, are not typically removed during passive or active regeneration, and they can create increased back pressure and reduced period to service maintenance.

X1.18 Oxidation Stability

X1.18.1 Products of oxidation in biodiesel can take the form of various acids or polymers, which, if in high enough concentration, can cause fuel system deposits and lead to filter clogging and fuel system malfunctions. Additives designed to retard the formation of acids and polymers can significantly improve the oxidation stability performance of biodiesel. See Appendix X2 for additional information on long-term storage.

X2. LONG-TERM STORAGE OF BIODIESEL

X2.1 Scope

X2.1.1 This appendix provides guidance for consumers of biodiesel (B100) who may wish to store quantities of fuels for extended periods. Consistently successful long-term fuel storage requires attention to fuel selection, storage conditions, and monitoring of properties prior to and during storage. This appendix is directed toward biodiesel (B100) and may be more or less applicable to blends of biodiesel with petroleum based diesel fuel.

X2.1.2 Normally produced biodiesel has adequate stability properties to withstand normal storage without the formation of troublesome amounts of insoluble degradation products, although data suggests some biodiesel may degrade faster than petroleum based diesel fuel. Biodiesel that is to be stored for prolonged periods should be selected to avoid formation of sediments, high acid numbers, and high viscosities that can clog filters, affect fuel pump operation or plug combustor nozzles or injectors. The selection of biodiesel should result from supplier-user discussions.

X2.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirement imposed by the warranty of the distillate fuel equipment manufacturers or by federal, state, or local government regulations. Although they cannot replace knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the biodiesel fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when, and how fuel quality should be monitored.

X2.2 Terminology

X2.2.1 *bulk fuel*—fuel in the storage facility in quantities over 50 gallons.

X2.2.2 *combustor fuel*—fuel entering the combustion zone of the burner or engine after filtration or other treatment of bulk fuel.

X2.2.3 *fuel contaminants*—foreign materials that make fuel less suitable or unsuitable for the intended use. Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X2.2.4 *fuel-degradation products*—those materials formed in fuel after it is produced. Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (acids and gums) may be more or less volatile than the fuel and may cause an increase in injector and nozzle deposits. The formation of degradation products may be catalyzed by contact with metals, especially those containing copper and, to a lesser extent, iron.

X2.2.5 *long-term storage*—storage of fuel for longer than 6 months after it is received by the user.

X2.3 Fuel Selection

X2.3.1 The stability properties of biodiesel are not fully understood and appear to depend on the vegetable oil and animal fat sources, severity of processing, and whether additional production plant treatment has been carried out or stability additives are present.

X2.3.2 The composition and stability properties of biodiesel produced at specific production plants may be different. Any special requirements of the user, such as long-term storage, should be discussed with the supplier.

X2.4 Fuel Additives

X2.4.1 Available fuel additives appear to improve the long term storage of biodiesel. Most additives should be added as close to the production site as possible to obtain maximum benefits.

X2.4.2 Biocides or biostats destroy or inhibit the growth of fungi and bacteria which can grow at fuel-water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in the fuel phase or the water phase, or both. Refer to Guide D6469 for a more complete discussion.

X2.5 Tests for Fuel Quality

X2.5.1 Test methods for estimating the storage stability of biodiesel (B100) are being developed. Modifications of Test Method D2274 to use glass fiber filters, varying times and temperatures, and the measurement of pre-test and post-test acid number and viscosity appear promising. However, correlation of this test with actual storage stability is unknown, and may depend upon field conditions and fuel composition.

X2.5.2 Performance criteria for accelerated stability tests that ensure satisfactory long-term storage of biodiesel (B100) have not been established.

X2.6 Fuel Monitoring

X2.6.1 A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful monitoring program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

X2.6.2 Stored fuel should be periodically sampled and its quality assessed. Practice D4057 provides guidance for sampling. Fuel contaminants and degradation products may settle to the bottom of a quiescent tank although detrimental changes to biodiesel can occur (rising acid value) without causing sediment formation. A *Bottom* or *Clearance* sample, as defined in Practice D4057, should be included in the evaluation along with an *All Level* sample.

X2.6.3 The quantity of insoluble fuel contaminants present in biodiesel can be determined using Test Method D6217 with glass fiber filters and abundant washing although no precision or bias testing has been performed with biodiesel using Test Method D6217.

X2.6.4 The acid value of biodiesel appears to exceed its specified maximum before other deleterious fuel property changes occur. A conscientious program of measuring the acid value of biodiesel may be sufficient for monitoring biodiesel stability.

X2.7 Fuel Storage Conditions

X2.7.1 Contamination levels in fuel can be reduced by storage in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface. Refer to Guide D6469 for a more complete discussion. Underground or isothermal storage is preferred to avoid temperature extremes; above-ground storage tanks should be sheltered or painted with reflective paint. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank breathing. The use of airtight sealed containers, such as drums or totes, can enhance the storage life of biodiesel.

X2.7.2 Copper and copper-containing alloys should be avoided with biodiesel due to increased sediment and deposit formation. Contact with lead, tin, and zinc can also cause increased sediment levels that can rapidly plug filters and should be avoided.

X2.7.3 Appendix X3 of Specification D2880 discusses fuel contaminants as a general topic. The discussion in Specification D2880 pertains to gas turbine combustion which may or may not be applicable to diesel engine combustion.

SUMMARY OF CHANGES

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751–09) that may impact the use of this standard. (Approved Dec. 1, 2009.)

(*1*) Added Test Method D5771 and D5772 to 5.1.9 and to the Referenced Documents.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751–08) that may impact the use of this standard. (Approved April 15, 2009.)

(*1*) Added Test Method D7397 cloud point as an option to Test Method D2500 in 5.1.9 and to the Referenced Documents.

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D6751–07b$^{\varepsilon 1}$) that may impact the use of this standard. (Approved Oct. 1, 2008.)

(*1*) Added Annex A1.
(*2*) Revised Table 1.
(*3*) Added 5.1.18.
(*4*) Added Note 4.

Designation: D 445 – 09

British Standard 2000: Part 71:1990

Designation: 71/1/97

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

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

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

1. Scope*

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

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

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

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

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

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

2.2 *ISO Standards:*[3]

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

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

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

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

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

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

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

ISO 3105 Glass Capillary Kinematic Viscometers—Specification and Operating Instructions

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

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

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

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

2.3 *NIST Standards:*[4]

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

NIST GMP 11

NIST Special Publication 819

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

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

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

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

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

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

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

4. Summary of Test Method

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

5. Significance and Use

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

6. Apparatus

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

7. Reagents and Materials

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

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

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

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

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

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

8. Certified Viscosity Reference Standards

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

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

9. Calibration and Verification

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

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

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

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

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

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

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

$$C_2 = (g_2/g_1) \times C_1 \quad (1)$$

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

10. General Procedure for Kinematic Viscosity

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

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

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

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

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

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

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

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.

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%

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

11. Procedure for Transparent Liquids

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

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

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

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

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

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

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

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

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

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

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

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

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

12. Procedure for Opaque Liquids

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

13. Cleaning of Viscometer

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

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

14. Calculation

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

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

where:

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

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

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

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

where:

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

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

15. Expression of Results

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

16. Report

16.1 Report the following information:

16.1.1 Type and identification of the product tested,

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

16.1.3 Result of the test (see Section 15),

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

16.1.5 Date of the test, and

16.1.6 Name and address of the test laboratory.

17. Precision

17.1 *Comparison of Determined Values*:

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

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

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

17.2 *Comparison of Results*:

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

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

where: x is the average of results being compared.

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

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

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

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

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

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

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

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

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

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

where: x is the average of results being compared.

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

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

18. Keywords

18.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

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

ANNEXES

(Mandatory Information)

A1. VISCOMETER TYPES AND CERTIFIED VISCOSITY REFERENCE STANDARDS

A1.1 Viscometer Types

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

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

TABLE A1.1 Viscometer Types

Viscometer Identification	Kinematic Viscosity Range,[A] mm^2/s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine[B]	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube[B]	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL[B]	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch[B]	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL[B]	3.5 to 100 000
BS/IP/SL(S)[B]	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde[B]	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic[B]	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution[B](B)	0.5 to 100 000
Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow Types for Transparent and Opaque Liquids	
Cannon-Fenske opaque	0.4 to 20 000
Zeitfuchs cross-arm	0.6 to 100 000
BS/IP/RF U-tube reverse-flow	0.6 to 300 000
Lantz-Zeitfuchs type reverse-flow	60 to 100 000

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Specifications D 446.

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

TABLE A1.2 Certified Viscosity Reference Standards

Designation	Approximate Kinematic Viscosity, mm^2/s					
	20°C	25°C	40°C	50°C	80 °C	100°C
S3	4.6	4.0	2.9	...	...	1.2
S6	11	8.9	5.7	...	...	1.8
S20	44	34	18	...	...	3.9
S60	170	120	54	...	...	7.2
S200	640	450	180	...	...	17
S600	2400	1600	520	280	67	32
S2000	8700	5600	1700	...	...	75
S8000	37 000	23 000	6700	...	...	...
S30 000	...	81 000	23 000	11 000	...	...

A2. KINEMATIC VISCOSITY TEST THERMOMETERS

A2.1 Short-Range Specialized Thermometer

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

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

A2.2 Calibration

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

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

TABLE A2.1 General Specification for Thermometers

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

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

TABLE A2.2 Complying Thermometers

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

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

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

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

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

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

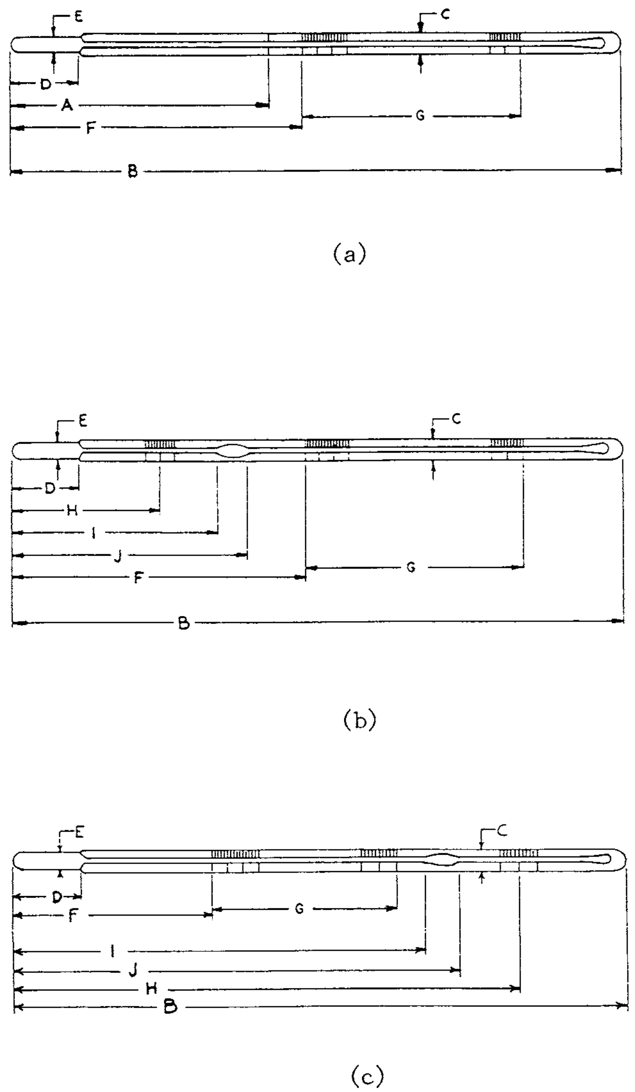

FIG. A2.1 Thermometer Designs

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

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

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

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

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

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

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

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

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

A3. TIMER ACCURACY

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

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

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

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

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

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

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

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

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

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

A4.4 Construct the acceptable tolerance zone:

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

SUMMARY OF CHANGES

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

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

Designation: D 2500 – 09

An American National Standard
British Standard 4458

Designation: 219/82

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

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

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

1. Scope*

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

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

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standard:*[3]

Specifications for IP Standard Thermometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

4. Summary of Test Method

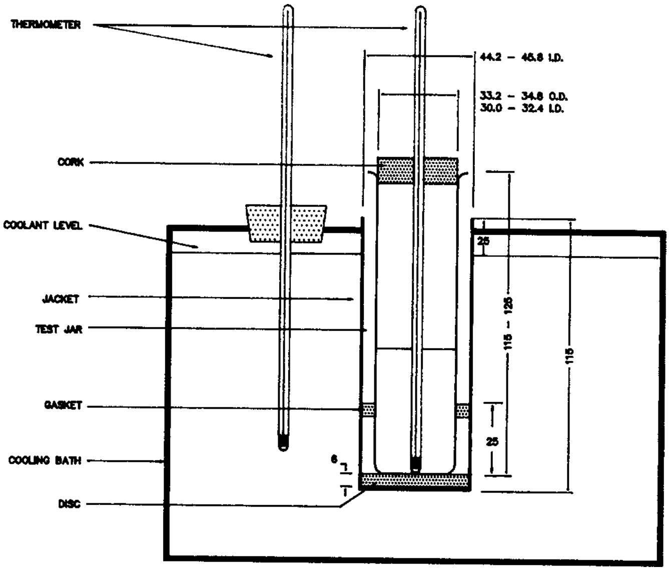
4.1 The specimen is cooled at a specified rate and examined periodically. The temperature at which a cloud is first observed at the bottom of the test jar is recorded as the cloud point.

5. Significance and Use

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

6. Apparatus (see Fig. 1)

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



NOTE—All dimensions are in milllimetres.

FIG. 1 Apparatus for Cloud Point Test

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

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

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

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

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

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

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

7. Reagents and Materials

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

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

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

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

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

TABLE 1 Cooling Mixtures and Bath Temperatures

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

8. Procedure

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

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

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

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

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

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

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

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

TABLE 2 Bath and Sample Temperature Ranges

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

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

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

9. Report

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

10. Precision and Bias

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

11. Keywords

11.1 cloud point; petroleum products; wax crystals

SUMMARY OF CHANGES

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

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

Designation: D 97 – 05

An American National Standard

Designation: 15/95

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

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

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

1. Scope*

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

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

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

2. Referenced Documents

2.1 *ASTM Standards:* [3]

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

2.2 *Energy Institute Standards:*

Specifications for IP Standard Thermometers [5]

3. Terminology

3.1 *Definitions:*

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

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

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

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

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

4. Summary of Test Method

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

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

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

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

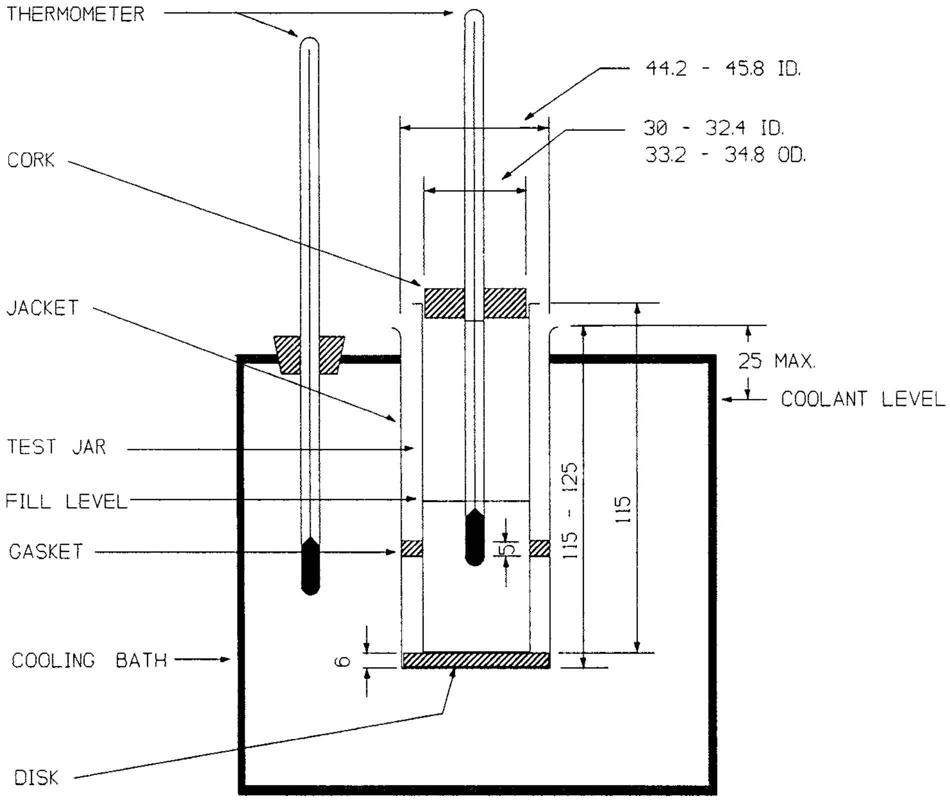
[2] Statements defining this test and its significance when applied to electrical insulating oils of mineral origin will be found in Guide D 117.

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

[4] Withdrawn.

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

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



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

FIG. 1 Apparatus for Pour Point Test

5. Significance and Use

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

6. Apparatus

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

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

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

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

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

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

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

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

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

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

7. Reagents and Materials

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

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

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

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

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

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

8. Procedure

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

9. Calculation and Report

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

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

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

10. Precision and Bias

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

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

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

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

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

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

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

APPENDIX

(Nonmandatory Information)

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

X1.1 General

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

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

X1.2 Scope

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

X1.3 Definition

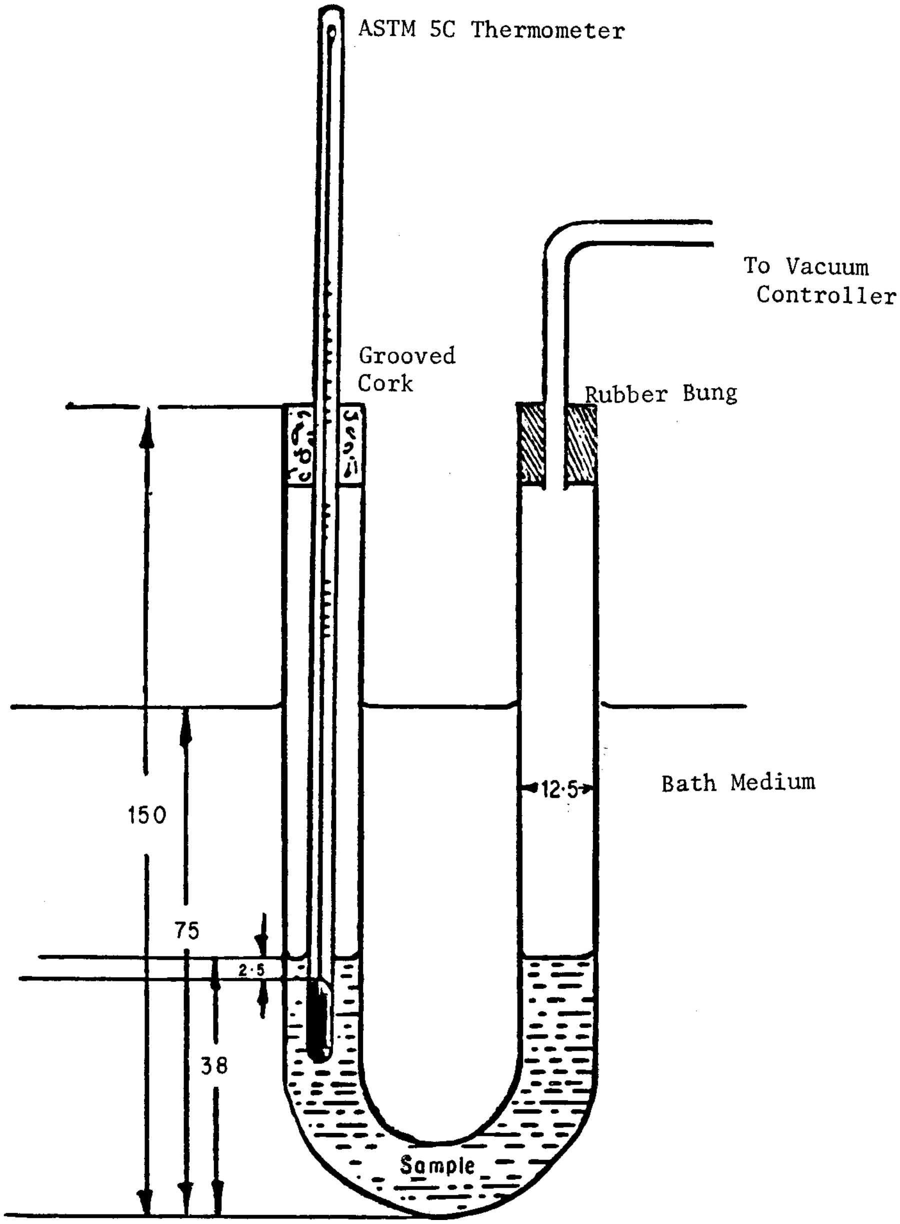
X1.3.1 *fluidity temperature*—the sample when tested in an as-received condition is considered "fluid at the temperature of the test" if it will flow 2 mm in 1 min in a 12.5 mm U-tube under a maximum pressure of 152 mm of mercury.

X1.4 Summary of Test Method

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

X1.5 Significance and Use

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



NOTE—All dimensions are in millimetres

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

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

X1.6 Apparatus

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

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

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

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

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[8] A kinematic viscosity bath is usually satisfactory.

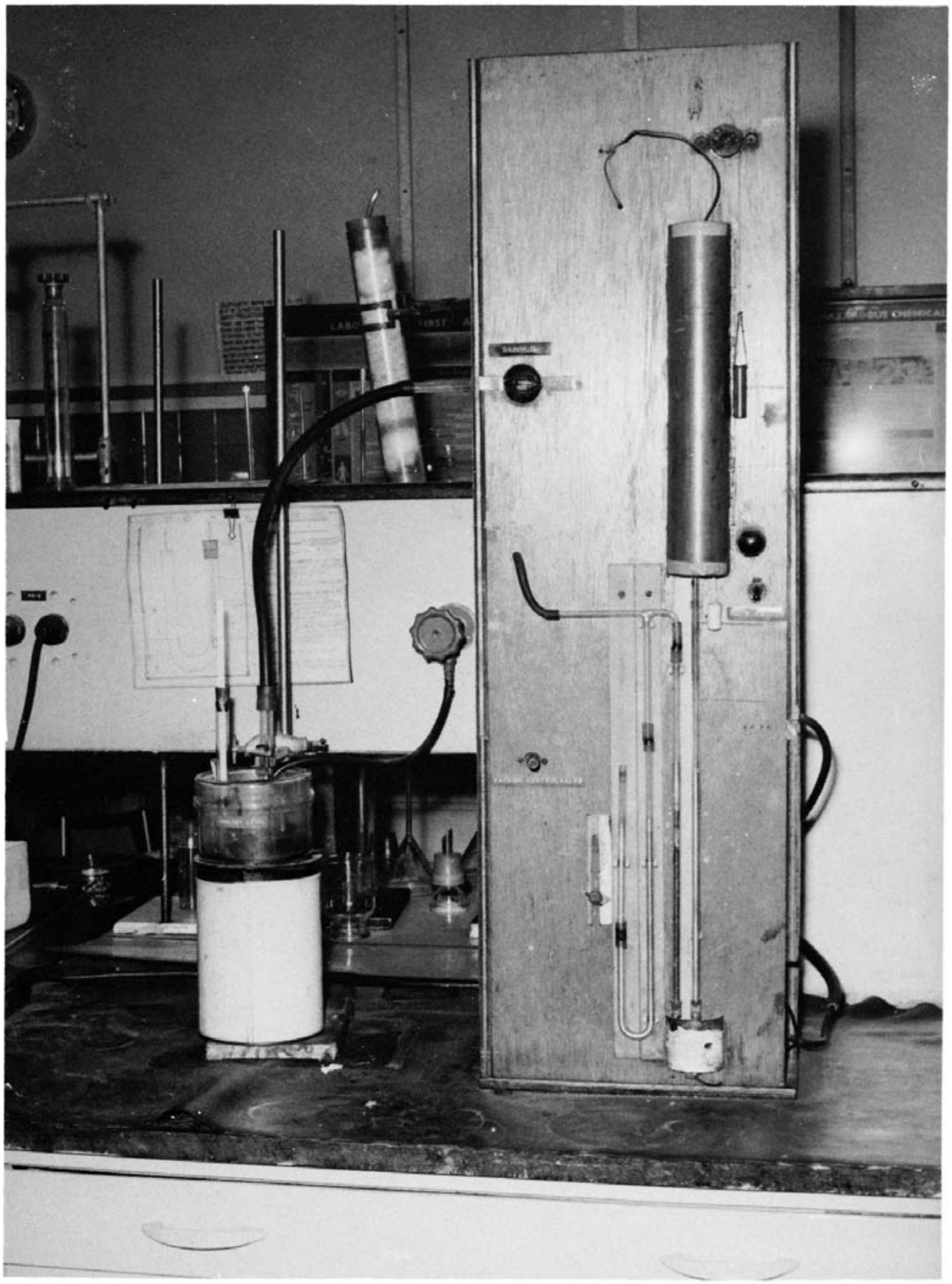

FIG. X1.2 Fluidity Temperature Apparatus

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

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

X1.7 Preparation of Apparatus

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

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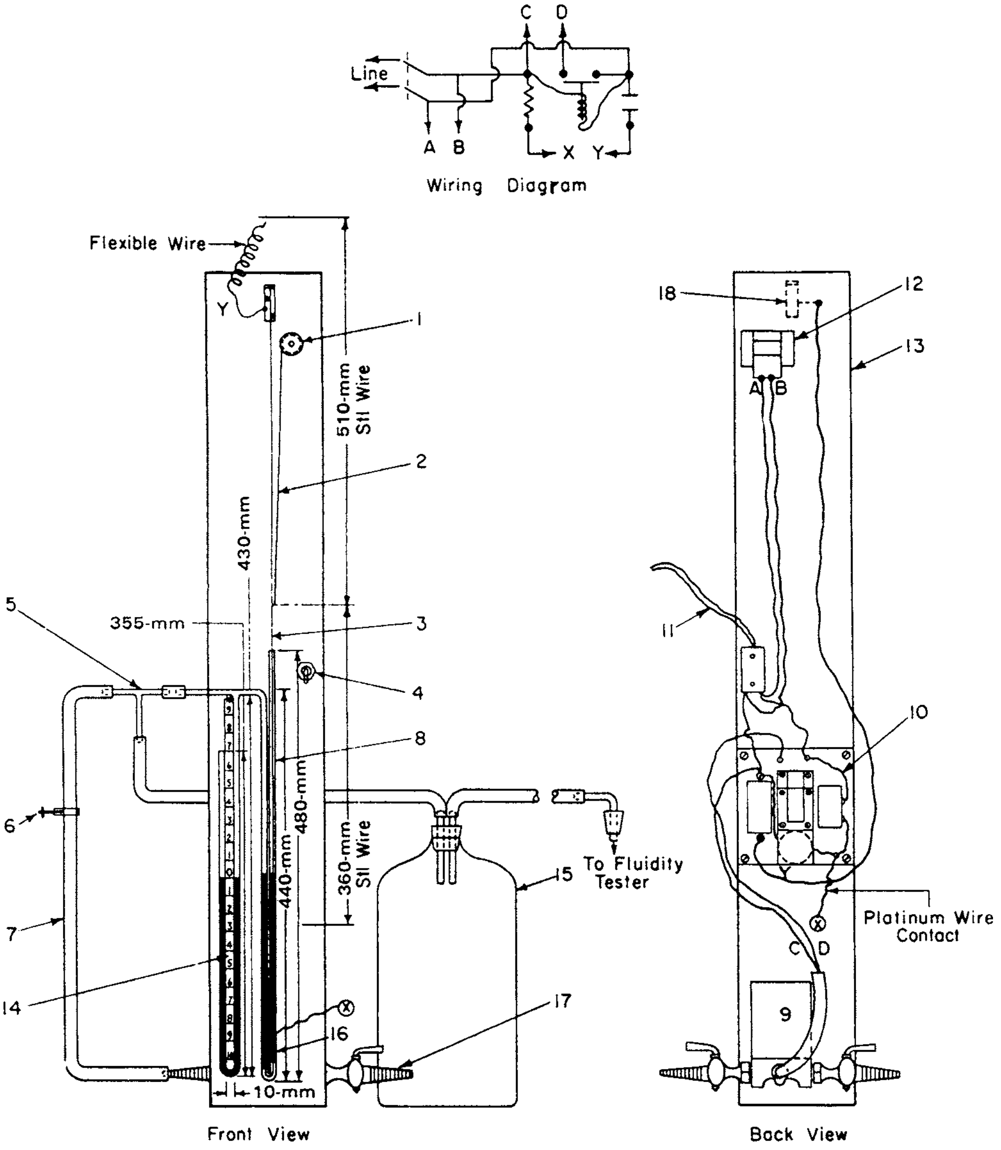
[9] This apparatus may be shop fabricated. Details of special parts are indicated in Figs. X1.3 and X1.4. Alternatively the apparatus can be purchased.



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

FIG. X1.3 Assembly Automatic Vacuum Controller Apparatus

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

X1.8 Procedure

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

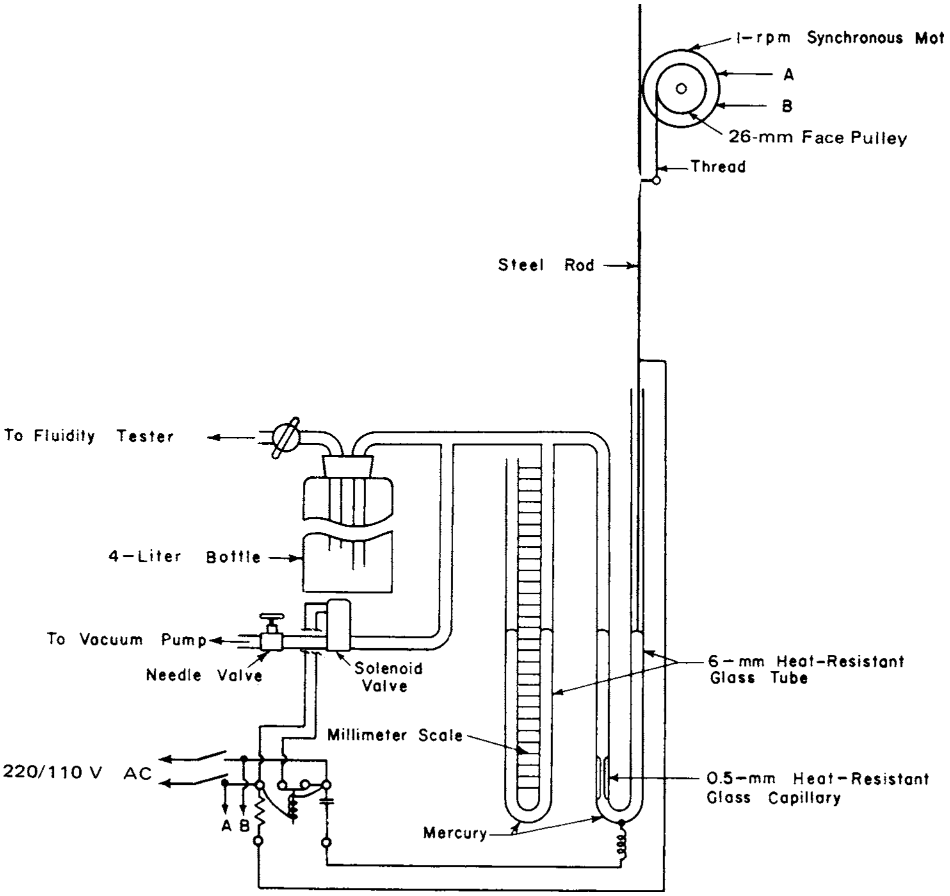


FIG. X1.4 Detail of Automatic Vacuum Controller

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

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

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

X1.9 Report

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

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

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

X1.10 Precision and Bias

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

SUMMARY OF CHANGES

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

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

H-26-30

Designation: D 941 – 88

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An American National Standard

Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer[1]

This standard is issued under the fixed designation D 941; 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 to replace Method 402 of Test Method Standard No 791b. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the measurement of the density of any hydrocarbon material that can be handled in a normal fashion as a liquid at the specified test temperatures of 20 to 25°C. Its application is restricted to liquids having vapor pressures less than 80 kPa (600 mm Hg) and having viscosities less than 15 mm^2/s (cst) at 20°C.

1.2 Two procedures are covered as follows:

1.2.1 *Procedure A*, for pure compounds and mixtures which are not highly volatile.

1.2.2 *Procedure B*, for highly volatile mixtures.

1.3 This test method provides a calculation procedure for converting density to relative density.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Notes 1, 2, 6 and Annex A1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1250 Petroleum Measurement Tables[2]

E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 *density*—mass per unit volume.

3.1.1 *Discussion*— In this test method, the measurement is at any given temperature and the units are grams per millilitre.

3.1.2 *relative density*—the ratio of the density of a material at a stated temperature to the density of water at a stated temperature.

4. Summary of Test Method[4]

4.1 The liquid sample is drawn into the pycnometer and weighed. It is then equilibrated at the test temperature, and the positions of the liquid levels are observed. The density or relative density of the sample is then calculated from its weight, a calibration factor proportional to an equal volume of water, and a term that corrects for the buoyancy of air.

5. Significance and Use

5.1 Density is a fundamental physical property which can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and to assess the quality of crude oils.

5.2 Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperatures of 15°C or 60°F.

6. Apparatus

6.1 *Pycnometer*—A pycnometer conforming to the dimensions given in Fig. 1, constructed of borosilicate glass, and having a total weight not exceeding 30 g.

6.2 *Constant-Temperature Bath*—A water bath having a depth of at least 12 in. (305 mm), provided with means for maintaining a temperature of 20 ± 0.02°C or 25 ± 0.02°C.

6.3 *Bath Thermometer*—No suitable ASTM Celsius thermometers are available; ASTM Kinematic Viscosity Thermometers 44F and 45F designed for tests at 68°F (20°C) and 77°F (25°C) and conforming to the requirements prescribed in Specification E 1 are therefore specified. Ice point and bore corrections must be known to the nearest 0.02°F. In use, the thermometers must be immersed to a point at least 2°F above the test temperature.

6.4 *Pycnometer Holder*—Figure 2 shows the structural details of the holder proper. It can be made of brass or any other available metal that can be hard- or soft-soldered and that will not corrode in the thermostat liquid. Figure 3 illustrates a convenient mounting for suspending the holders in the thermostat. It consists of a brass bar ⅛ in. (3.2 mm) in thickness by 1 in. (25 mm) in width, of a length suitable for

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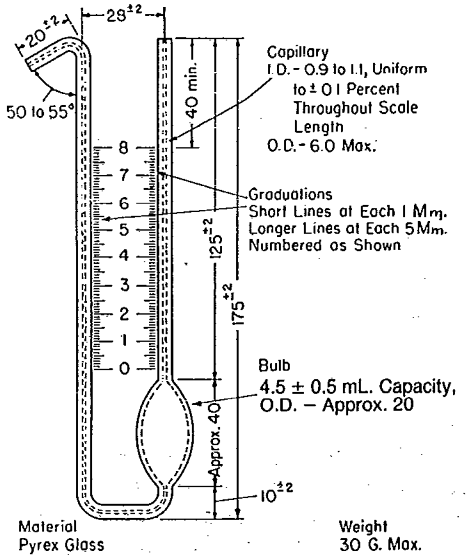
[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04 on Hydrocarbon Analysis.

Current edition approved Oct 31, 1988. Published December 1988. Originally published as D 941 – 47. Last previous edition D 941 – 83.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vols 05.03 and 14.01.

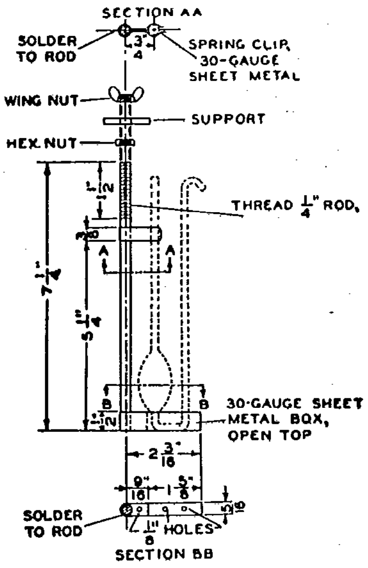
[4] For a more complete discussion of this method, see Davidson, J. A., Harvey, T., Kurtz, S. S., Jr., Lipkin, M. R., "Pycnometer for Volatile Liquids," *Industrial and Engineering Chemistry*, Analytical Edition, IENAA Vol 16, No. 1, 1944, p. 55 and H. M. Smith, and Cooperators, "Measurement of Density of Hydrocarbon Liquids by the Pycnometer," *Analytical Chemistry*, ANCHA Vol 22, Nov. 1952, p. 1452.



All Dimensions in Millimetres

NOTE—The graduation lines shall extend around the entire circumference of the pycnometer at the integral numbers 0, 1, 2 cm, etc., half way around at the half divisions 0.5, 1.5, etc., and shorter lines for the intermediate subdivisions.

FIG. 1 Pycnometer



Metric Equivalents

in.	mm	in.	mm
5/16	7.94	3/4	19
9/16	14.3	1½	38
1/8	3.2	1⅝	41.3
3/8	9.53	2 3/16	55.6
1/4	6.4	5¼	133.4
1/2	12.7	7¼	184.2

FIG. 2 Pycnometer Holder

the bath used, and with seven 9/32-in. (7.144 mm) holes drilled 1½ in. (38.1 mm) apart to accommodate the threaded ends of the holders. Two nuts support each holder and permit regulation of the depth of immersion of the pycnometers.

6.5 *Balance*—A balance able to reproduce weighings within 0.1 mg when carrying a load of 30 g or less on each pan. The balance is to be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. Otherwise weighings must be made by the substitution method in which the calibrated weights and pycnometer are alternately weighed on the same balance pan. The same balance shall be used for all related weighings.

6.6 *Weights*—Weights are to be used whose relative values are known to the nearest 0.05 mg, or better. The same set of weights shall be used for the calibration of the pycnometer and the determination of the densities, or the sets of weights shall be calibrated relative to each other.

7. Preparation of Apparatus

7.1 Thoroughly clean the pyncometer with hot chromic acid. (**Warning**—See Note 1.) Chromic acid solution is the most effective cleaning agent. However, surfactant cleaning fluids have also been used successfully. Rinse well with distilled water and dry at 105 to 110°C for at least 1 h, preferably with a slow current or filtered air passing through the pycnometer. Cleaning is to be done in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pyncometer can be cleaned between determinations by washing with a suitable solvent, such as isopentane or acetone (**Warning**—See Note 2.) and vacuum drying. If acetone is used as the wash liquid, the pycnometer is then to be rinsed with isopentane.

NOTE 1: **Warning**—Causes severe burns. A recognized carcinogen. See Annex A1.1.

NOTE 2: **Warning**—Extremely flammable. See Annexes A1.2, A1.3.

8. Calibration of Apparatus

8.1 Proceeding as directed in Section 9, determine the weight of freshly boiled distilled water held by the pycnometer when equilibrated at the test temperature (20 or 25°C) with the water level at each of three different scale points on the graduated arms, two of which are to be at opposite ends of the scale. Prepare a calibration curve by plotting the sum of the scale readings on the two arms of the pycnometer against the corresponding apparent volume. If this curve is not a straight line, and subsequent checks do not correct the curvature, discard the pycnometer as imperfect, unless a line conforming to 8.2 can be obtained. Obtain the apparent volume in millilitres by dividing the weight of the water held in the pycnometer by the density of water at 20°C (0.99823 g/mL), or at 25°C (0.99707 g/mL).

NOTE 3—The apparent volume differs from the true volume by the amount of the air buoyancy correction on the weight of water contained in the pycnometer.

8.2 If a straight line cannot be drawn through the three points, determine enough additional points so that a straight line calibration can be drawn which does not lie more than 0.0002 mL in units from the points used to determine the line.

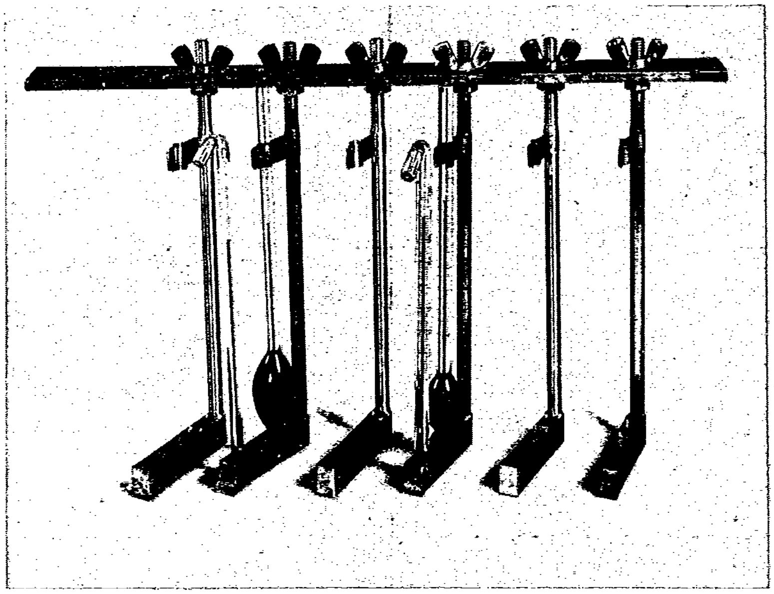


FIG. 3 Holder Mounting

9. Procedure A

9.1 Procedure A is intended for pure compounds and mixtures that are not highly volatile, that is, which are essentially free from materials boiling below 20°C.

9.2 Weigh the clean, dry pycnometer to 0.1 mg and record the weight.

9.3 Fill the pycnometer with the sample at approximately the test temperature by holding it in an upright position and placing the hooked tip in the sample, allowing the liquid to be drawn over the bend in the capillary by surface tension. Allow the pycnometer to fill by siphoning (requiring about 1 min) and break the siphon when the liquid level in the bulb arm of the pycnometer reaches the lowest calibration mark.

TABLE 1 Air Buoyancy Corrections

W/V	Correction,[A] plus	*W/V*	Correction,[A] plus
0.70	0.00036	0.85	0.00018
0.71	0.00035	0.86	0.00017
0.72	0.00033	0.87	0.00016
0.73	0.00032	0.88	0.00014
0.74	0.00031	0.89	0.00013
0.75	0.00030	0.90	0.00012
0.76	0.00029	0.91	0.00011
0.77	0.00028	0.92	0.00010
0.78	0.00026	0.93	0.00009
0.79	0.00025	0.94	0.00007
0.80	0.00024	0.95	0.00006
0.81	0.00023	0.96	0.00005
0.82	0.00022	0.97	0.00004
0.83	0.00020	0.98	0.00003
0.84	0.00019	0.99	0.00001

[A] This table applies for all air density values between 0.0011 and 0.0013 g/mL. For air densities outside this range, the air buoyancy correction, C, should be calculated as follows:

$$C = (d_a/0.99823) \times [0.99823 - (W/V)]$$

where:

C = air buoyancy correction,
d_a = density of air in the balance case, g/mL
W = weight of sample in pycnometer, and
V = volume of sample in pycnometer.

9.4 Wipe off the wet tip thoroughly (Note 4) with a chemically clean, lint-free cloth slightly damp with water and weigh to the nearest 0.1 mg.

NOTE 4—In atmospheres of low humidity (60 % or lower) drying the pycnometer by rubbing with dry cotton cloth will induce static charges equivalent to a loss of about 1 mg or more in the weight of the pycnometer. If this charge is not dissipated in less than ½ h it can be detected by touching the pycnometer to the wire hook on the balance and then drawing it away slowly. If the pycnometer exhibits an attraction for the wire hook, it may be considered to have a static charge.

9.5 Place the pycnometer in the holder in a constant temperature bath adjusted to the test temperature (20 to 25°C) within ±0.02°C. When the liquid level has reached equilibrium (usually in about 10 min), read the scale to the nearest 0.2 small division at the liquid level in each arm.

10. Procedure B

10.1 Procedure B is intended for highly volatile mixtures that contain appreciable amounts of material boiling below 20°C, or for any material where there is uncertainty concerning loss due to evaporation during the density determination.

10.2 Weigh the pycnometer as described in 9.2.

10.3 Cool the sample and pycnometer to a temperature of 0 to 5°C before filling. If the determination must be made when the dew point is high enough to cause condensation of moisture in the pycnometer, proper precautions should be taken to avoid this. Fill the pycnometer according to the procedure described in 9.3.

10.4 Place the pycnometer in the bath and read the volume as described in 9.5.

NOTE 5—If at any time during equilibration the level of the liquid rises above the scale graduations, cautiously apply air pressure to the opening of the bulb arm of the pycnometer and force a few drops of the sample from the bent arm.

10.5 Remove the pycnometer from the bath, rinse the outside with acetone, then with clean isopentane (**Warning—**

TABLE 2 Density of Water

Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.99987	21	0.99802	40	0.99224
3	0.99999	22	0.99780	45	0.99025
4	1.00000	23	0.99756	50	0.98807
5	0.99999	24	0.99732	55	0.98573
10	0.99973	25	0.99707	60	0.98324
15	0.99913	26	0.99681	65	0.98059
15.56	0.99904	27	0.99654	70	0.97781
16	0.99897	28	0.99626	75	0.97489
17	0.99880	29	0.99597	80	0.97183
18	0.99862	30	0.99567	85	0.96865
19	0.99843	35	0.99406	90	0.96534
20	0.99823	37.78	0.99307	100	0.95838

See Note 6.) and dry thoroughly (see Note 4) with a chemically clean, lint-free cloth, slightly damp with water. Weigh to the nearest 0.1 mg.

NOTE 6: **Warning**—Extremely flammable. See Annex A1.2, A1.3.

11. Calculation

11.1 Calculate the density of the sample as follows:

$$D = (W/V) + C$$

where:

D = density, g/mL at 20 or 25°C,
W = weight, g, in air of sample contained in the pycnometer at the test temperature (20 or 25°C),
V = apparent volume, mL, corresponding to the sum of the scale readings on the two arms of the pycnometer, as obtained from the calibration curve, and
C = air buoyancy correction, as obtained from Table 1.

11.2 Calculate the relative density of the sample at t_1/t_2 by dividing the density as calculated in 11.1 by the density of water at the reference temperature, t_2, as obtained from Table 2. Relative density at t_1/15.56°C (t_1/60°F where t is expressed in degrees F) can be changed to the conventional 15.56/15.56°C (60/60°F) by use of the appropriate relative density Table 23 in Standard D 1250, provided that the glass expansion factor has been excluded.

12. Report

12.1 In reporting density, give the test temperature and the units (For example: Density at 20°C = x.xxxx g/mL). In reporting relative density, give both the test temperature and the reference temperature, but no units (For example: relative density, 15.56/15.56°C = x.xxxx). Carry out all calculations to five figures, and round off the final result to four figures.

13. Precision and Bias

13.1 The precision and bias of the test method as obtained by statistical examination of interlaboratory test results is as follows.

13.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed 0.0001 g/mL only in one case in twenty.

13.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 0.0002 g/mL only in one case in twenty.

13.1.3 *Bias*—The subcommittee is presently working on the development of a bias statement.

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 Chromic Acid (Cleaning Solution)

Do not get in eyes, on skin, or on clothing.
Avoid breathing vapor or mist.
Keep container closed.
Use with adequate ventilation.
Do not take internally.
Wash thoroughly after handling.
Use protective clothing and goggles when handling.

A1.2 Isopentane

Harmful if inhaled. Vapors may cause flash fire.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid build-up of vapors and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A1.3 Acetone

Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Vapors may spread long distances and ignite explosively.
Avoid build-up of vapors, and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid contact with eyes or skin.

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INTERNATIONAL

Designation: D 6371 – 05

An American National Standard

Standard Test Method for
Cold Filter Plugging Point of Diesel and Heating Fuels[1]

This standard is issued under the fixed designation D 6371; 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.

1. Scope*

1.1 This test method covers the determination of the cold filter plugging point (CFPP) temperature of diesel and domestic heating fuels using either manual or automated apparatus.

NOTE 1—This test method is technically equivalent to test methods IP 309 and EN 116.

1.2 The manual apparatus and automated apparatus are both suitable for referee purposes.

1.3 This test method is applicable to distillate fuels, including those containing a flow-improving or other additive, intended for use in diesel engines and domestic heating installations.

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

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

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 2500 Test Method for Cloud Point of Petroleum Products
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D 5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)
D 5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)
D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)
E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *IP Standards:*[3]

IP 309 Diesel and domestic heating fuels - Determination of cold filter plugging point
Specifications for IP Standard Thermometers

2.3 *ISO Standards:*[4]

IP 3310 Test sieves - Technical requirements and testing - Part 1: Metal cloth

2.4 *European Standards:*[5]

EN 116 Diesel and domestic heating fuels - Determination of cold filter plugging point

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *certified reference material*, *n*—a stable petroleum product with a method-specific nominal CFPP value established by a method-specific interlaboratory study following RR:D02-1007[6] guidelines or ISO Guides 34 and 35.[4]

3.1.2 *cold filter plugging point*, *n*—highest temperature, expressed in multiples of 1°C, at which a given volume of fuel fails to pass through a standardized filtration device in a specified time when cooled under the conditions prescribed in this test method.

4. Summary of Test Method

4.1 A specimen of the sample is cooled under specified conditions and, at intervals of 1°C, is drawn into a pipet under a controlled vacuum through a standardized wire mesh filter. The procedure is repeated, as the specimen continues to cool, for each 1°C below the first test temperature. Testing is continued until the amount of wax crystals that have separated out of solution is sufficient to stop or slow down the flow so that the time taken to fill the pipet exceeds 60 s or the fuel fails to return completely to the test jar before the fuel has cooled by a further 1°C.

4.2 The indicated temperature at which the last filtration was commenced is recorded as the CFPP.

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

Current edition approved May 1, 2005. Published May 2005. Originally approved in 1999. Last previous edition approved in 1999 as D 6371–99.

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

[3] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K.

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

[5] Available from European Committee for Standardization, Central Secretariat, Rue Bréderode 2, B-1000, Brussels, Belgium.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1007.

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

5. Significance and Use

5.1 The CFPP of a fuel is suitable for estimating the lowest temperature at which a fuel will give trouble-free flow in certain fuel systems.

5.2 In the case of diesel fuel used in European light duty trucks, the results are usually close to the temperature of failure in service except when the fuel system contains, for example, a paper filter installed in a location exposed to the weather or if the filter plugging temperature is more than 12°C below the cloud point value in accordance with Test Method D 2500, D 5771, D 5772, or D 5773. Domestic heating installations are usually less critical and often operate satisfactorily at temperatures somewhat lower than those indicated by the test results.

5.3 The difference in results obtained from the sample *as received* and after heat treatment at 45°C for 30 min can be used to investigate complaints of unsatisfactory performance under low temperature conditions.

6. Apparatus

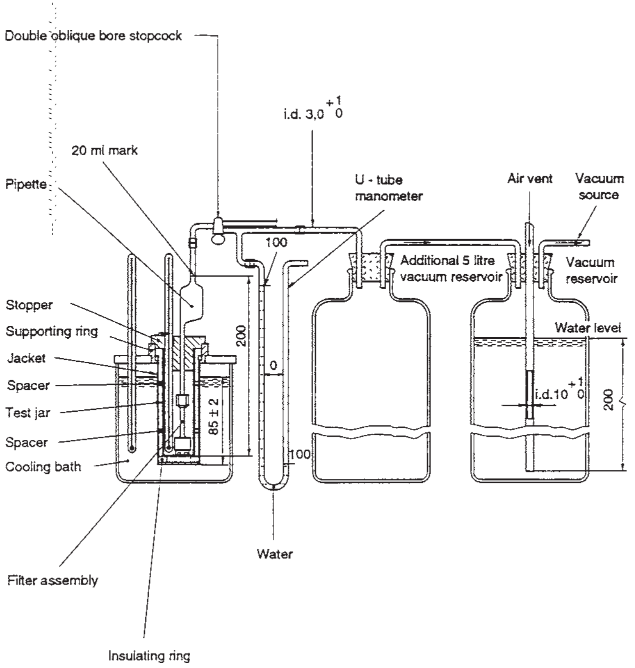
6.1 *Manual Apparatus*:

6.1.1 The apparatus, as detailed in 6.1.2-6.1.13, shall be arranged as shown in Fig. 1.

6.1.2 *Test Jar*, cylindrical, of clear glass, flat bottomed, with an internal diameter of 31.5 ± 0.5 mm, a wall thickness of 1.25 ±0.25 mm and a height of 120 ± 5 mm. The jar shall have a permanent mark at the 45 ± 1 mL level.

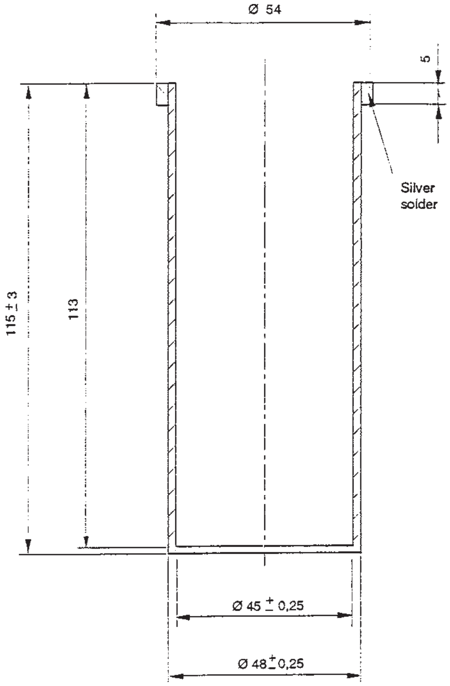
NOTE 2—Test jars of the required dimensions may be obtained by selection from jars conforming to Test Method D 2500, which specifies a wider diameter tolerance.

6.1.3 *Jacket*, brass, watertight, cylindrical, flat bottomed, to be used as an air bath. It shall have an inside diameter of 45 ±0.25 mm, outside diameter of 48 ± 0.25 mm, and a height of 115 ± 3 mm (see Fig. 2).



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

FIG. 1 Arrangement of Manual CFPP Apparatus



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

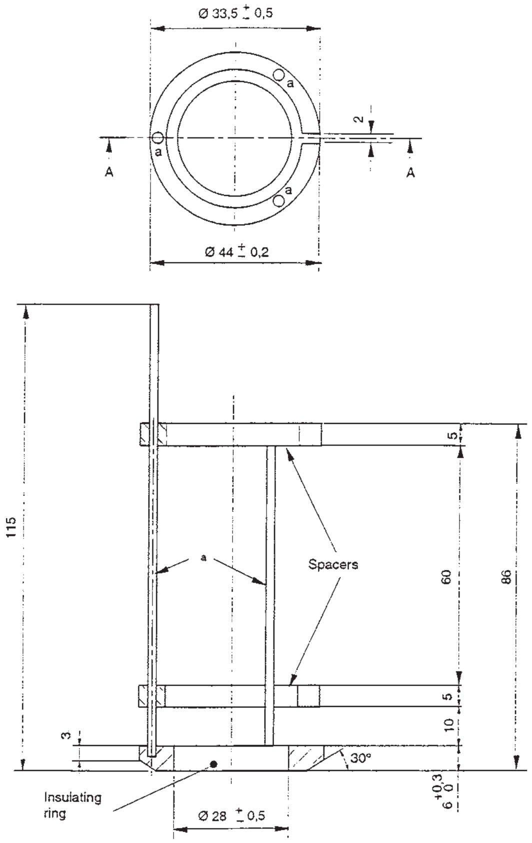
FIG. 2 Watertight Brass Jacket

6.1.4 *Insulating Ring*, made from oil-resistant plastics or other suitable material, to be placed in the bottom of the jacket (see 6.1.3) to provide insulation for the bottom of the test jar. It shall fit closely inside the jacket and have a thickness of 6 + 0.3 - 0.0 mm.

6.1.5 *Spacers (two)*, approximately 5-mm thick, made of oil-resistant plastics or other suitable material, to be placed as shown in Fig. 1 around the test jar (see 6.1.2) to provide insulation for the test jar from the sides of the jacket. The spacers shall fit closely to the test jar and closely inside the jacket. The use of incomplete rings, each with a 2-mm circumferential gap, will accommodate variations in test jar diameter. The spacers and insulating ring may be made as a single part as shown in Fig. 3.

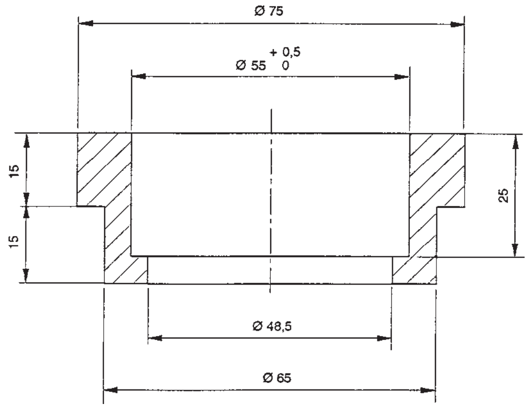
6.1.6 *Supporting Ring*, of oil resistant plastics or other suitable non-metallic, non-absorbent, oil-resistant material, used to suspend the jacket (see 6.1.3) in a stable and upright position in the cooling bath and to provide a concentric location for the stopper (see 6.1.7). A design is shown in Fig. 4 for guidance, but this design may be modified to suit the cooling bath.

6.1.7 *Stopper*, of oil-resistant plastics or other suitable nonmetallic, nonabsorbent, oil-resistant material, to fit the test jar and the support ring as shown in Fig. 5. It shall have three holes to accommodate the pipet (see 6.1.8) and the thermometer (see 6.1.9) and to allow venting of the system. If necessary, when using the high-range thermometer (see 6.1.9), the upper part of the stopper shall have an indentation to permit the thermometer (see 6.1.9) to be read down to a temperature of -30°C. A pointer shall be fitted to the upper surface of the stopper to facilitate location of the thermometer in relation to



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

FIG. 3 Spacers



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

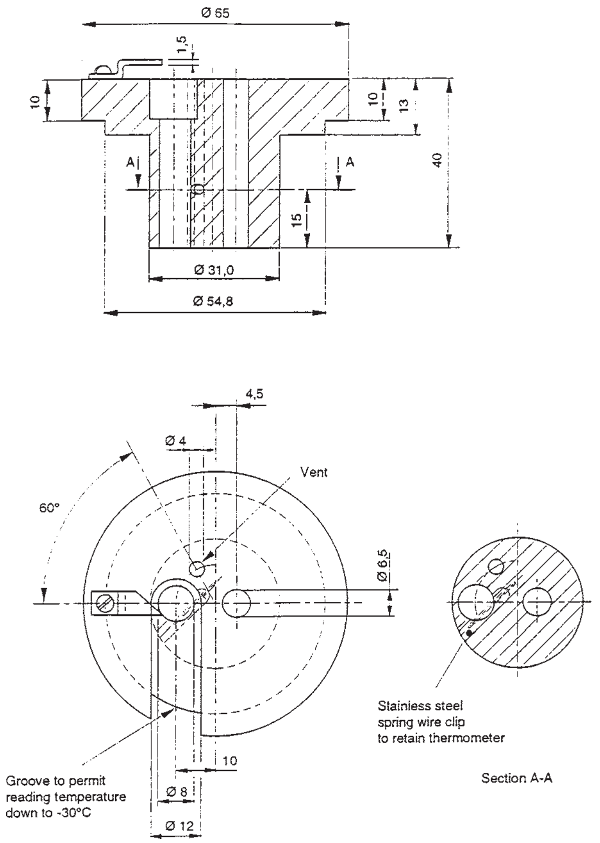
FIG. 4 Supporting Ring

the bottom of the test jar. A spring wire clip shall be used to retain the thermometer in the correct position.

6.1.8 *Pipet with Filter Unit*:

6.1.8.1 *Pipet*, of clear glass with a calibration mark corresponding to a contained volume of 20 ± 0.2 mL at a point 149 ± 0.5 mm from the bottom of the pipet (see Fig. 6). It shall be connected to the filter unit (see 6.1.8.2).

6.1.8.2 *Filter Unit (see* Fig. 7*)*, containing the following elements:



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

FIG. 5 Stopper with Holes for Thermometer, Pipet, and Vent

(1) Brass Body, with a threaded cavity that houses the wire mesh holder. The cavity shall be fitted with an O-ring of oil-resistant plastics. The internal diameter of the central tube shall be 4 ± 0.1 mm.

(2) Brass Screw Cap, to connect the upper part of the body of the filter unit (see 6.1.8.2) to the lower part of the pipet (see 6.1.8.1) to ensure a leak-free joint. An example of satisfactory connection is shown in Fig. 7.

(3) Disc, 15 ± 0.1-mm diameter, of plain weave stainless steel wire mesh gauze with a nominal aperture size of 45 µm. The nominal diameter of the wire shall be 32 µm, and the tolerance for the size of an individual aperture shall be as follows:

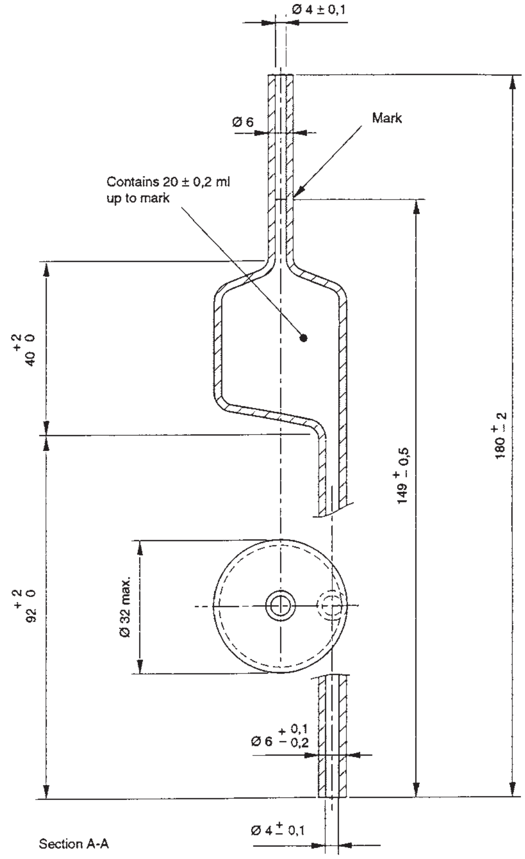
No aperture size shall exceed the nominal size by more than 22 µm.

The average aperture size shall be within ± 3.1 µm of the nominal size.

Not more than 6 % of the apertures shall be above the nominal size by more than 13 µm.

(4) Filter Holder of Brass, in which the disc of wire mesh gauze (see 6.1.8.2 (*3*)) is firmly clamped by a retaining ring pressed into the filter holder. The diameter of the exposed part of the gauze shall be 12 + 0.1 - 0.0 mm (see Fig. 8).

(5) Brass Cylinder, threaded on the outside, that can be screwed into the cavity of the body (see 6.1.8.2 (*1*)) to clamp the filter holder (see 6.1.8.2 (*4*)) against the O-ring (6.1.8.2



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

FIG. 6 Pipet

(*1*)), The lower end shall have four slots to allow the specimen to flow into the filter unit.

NOTE 3—The requirements for the wire mesh are taken from ISO 3310, to which reference may be made for methods for testing the gauze.

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

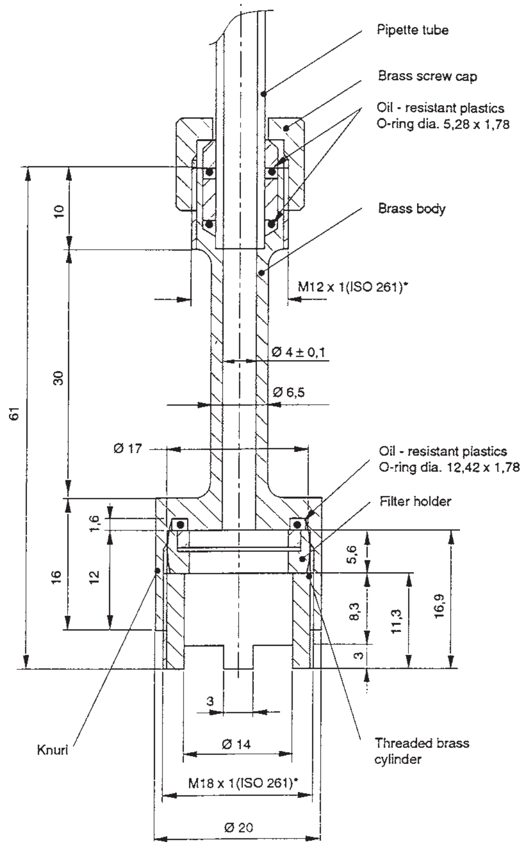
Thermometer	Temperature Range	Thermometer Number ASTM	IP
High-range for CFPP down to −30°C	−38°C to +50°C	5C	1C
Low-range from CFPP below −30°C	−80°C to +20°C	6C	2C
Cooling bath	−80°C to +20°C	6C	2C

6.1.10 *Cooling Bath*:

6.1.10.1 The type of cooling bath is optional, but it shall be of a shape and size suitable for containing the jacket (see 6.1.3) in a stable and upright position at the required depth.

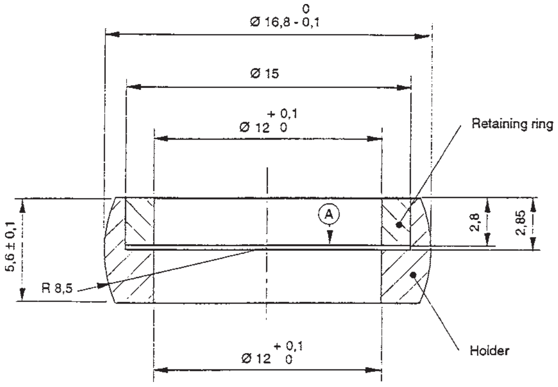
6.1.10.2 The bath shall be fitted with a cover with one or more holes in it to accommodate the supporting ring (see 6.1.6). The jacket (see 6.1.3) may be permanently mounted in the cover.

6.1.10.3 The bath temperature shall be maintained at the required value and tolerance by a refrigeration unit or by the use of suitable freezing mixtures, ensuring a homogenous temperature in the bath by stirring or other means of agitation.



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

FIG. 7 Filter Unit



NOTE—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

FIG. 8 Brass Filter Holder

Table 1 lists the bath temperature set-points required in the CFPP procedure. If only one bath is utilized, it must have the

TABLE 1 Cooling Bath Temperatures

Expected CFPP	Required Cooling Bath Temperature(s)
Down to −20°C	−34 ± 0.5°C
Between −20°C and −35°C	−34 ± 0.5°C then −51 ± 1°C
Below −35°C	−34 ± 0.5°C then −51 ± 1°C then −67 ± 2°C

ability to change down to the next lower set-point temperature in a time period not exceeding 2 min 30 s.

6.1.11 *Stopcock*, glass, with double oblique bore of 3-mm diameter.

6.1.12 *Vacuum Source*, vacuum pump or water pump powerful enough to ensure an air flow rate in the vacuum regulator of 15 ± 1 L/h for the duration of the test.

6.1.13 *Vacuum Regulator*, consisting of a glass bottle, at least 350-mm high, not less than 5 L capacity, partially filled with water. It shall be closed by a stopper with three holes of convenient diameters for glass tubes. Two tubes shall be short and shall not go below the water level. The third tube, with an internal diameter of 10 ± 1 mm, shall be long enough for one end to be approximately 200 mm beneath the surface of the water while the other end reaches a few centimetres above the stopper. The depth of the immersed part shall then be adjusted to obtain a depression of 200 ± 1 mm of water (2 ± 0.05 kPa) on the manometer, which shall contain water. A second empty 5 L bottle shall be fitted in the line to serve as a vacuum reservoir to ensure a constant depression. The arrangement is shown in Fig. 1.

6.1.14 *Stopwatch*, with a graduation or reading of 0.2 s or lower, with an accuracy of 0.1 % over a period of 10 min.

6.2 *Automated Apparatus*:

6.2.1 The automated apparatus shall include elements conforming to 6.1.1-6.1.8, platinum resistance thermometers, cooling bath(s), vacuum pump, and suitable electronic control and measurement devices.

6.2.2 *Cooling Bath*, a refrigeration unit capable of maintaining the cooling bath at the required temperature and also of automatically changing the bath temperature within 2 min 30 s at the appropriate stage (see 12.2.5).

6.2.3 *Vacuum Pump*, powerful enough to ensure an air flow rate in the vacuum regulator of a minimum of 15 ± 1 L/h, and to maintain a constant vacuum of 200 ± 1 mm (2 ± 0.05 kPa) for the duration of the test. For multi-position testers using the same vacuum pump, the flow rate shall be checked when several positions are operating simultaneously.

7. Reagents and Materials

7.1 *Heptane*, clean commercial or reagent grade. (**Warning—**Flammable. Harmful if inhaled.)

7.2 *Acetone*, clean commercial or reagent grade. (**Warning—**Extremely flammable.)

7.3 *Filter Paper*, (approximately 4 to 6 µm retention).

7.4 *Certified Reference Materials*.

8. Sampling

8.1 Unless otherwise specified in the commodity specification, samples shall be taken as described in Practice D 4057 or D 4177 in accordance with the requirements of national standards or regulation for the sampling of the product under test, or both.

9. Preparation of Test Specimen

9.1 Filter approximately 50 mL of the sample (see 8.1) at laboratory ambient temperature, but in any case not at a temperature less than 15°C, through dry filter paper (see 7.3).

10. Preparation of Apparatus

10.1 Prepare the manual apparatus or the automated apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment. See Fig. 1 for manual apparatus.

10.2 Before each test, dismantle the filter unit (see 6.1.8.2) and wash the pieces and the test jar (see 6.1.2), the pipet (see 6.1.8.1) and the thermometer (see 6.1.9 for manual apparatus and 6.2 for platinum resistance used in automated equipment) with heptane (see 7.1), then rinse with acetone (see 7.2) and dry in a stream of filtered air. Check the cleanliness and dryness of all elements, including the jacket (see 6.1.3). Examine the wire mesh (see 6.1.8.2(*3*)) and the joints (see 6.1.8.2(*1*) and 6.1.8.2(*2*) for damage; if necessary renew them.

10.3 Check that the screw cap (see 6.1.8.2(*2*) is tight enough to prevent leakage.

11. Calibration and Standardization

11.1 Adjust the automated CFPP apparatus (when used) in accordance with the manufacturer's instructions.

11.2 Calibrate the temperature measuring device in accordance with the manufacturer's instructions.

11.3 Periodically verify the correct functioning of manual and automated apparatus using a certified reference material or in-house secondary reference material, such as fuel of known CFPP value.

NOTE 4—It is preferable that verification be carried out at least two times a year, where possible, using certified reference materials. The apparatus should be checked more frequently (for example, weekly) using a secondary verification material.

11.4 When the CFPP values obtained using a verification material deviate by more than the test repeatability (see 15.2), or an unacceptable statistical quality control bias is observed, check the condition and operation of the apparatus to ensure conformity with the specification as stated in this test method. The manufacturer's instruction manual should provide guidance on ensuring that the apparatus is correctly set up and calibrated.

12. Procedure

12.1 *Manual Apparatus*:

12.1.1 Establish the cooling bath temperature at –34 ± 0.5°C

12.1.2 Place the insulating ring (see 6.1.4) on the bottom of the jacket (see 6.1.3). If spacers (see 6.1.5) are not mounted on the insulating ring (see 6.1.4), position them approximately 15 and 75 mm above the bottom of the test jar (see 6.1.2).

12.1.3 Pour the filtered specimen (see Section 9) into the clean and dry test jar to the mark (45 mL).

12.1.4 Close the test jar with the stopper (see 6.1.7) carrying the pipet with filter unit (see 6.1.8) and the appropriate thermometer (see 6.1.9). Use a low-range thermometer if the expected CFPP is below –30°C. Thermometers shall not be changed during the test. Adjust the apparatus in such a way that the bottom of the filter unit (see 6.1.8.2(*5*)) rests on the bottom of the test jar, and position the thermometer so that its lower end is 1.5 ± 0.2 mm above the bottom of the test jar. Take care to ensure that no part of the thermometer is not in contact with the side of the test jar or the filter body.

NOTE 5—The precise positioning of the thermometer in the test jar is a critical parameter of this test method. The position of the lower end of the thermometer above the bottom of the test jar can be indirectly measured by marking the stem of the thermometer flush with the stopper (see 6.1.7) when the lower end of the thermometer is just touching the bottom of the test jar, and then pulling the thermometer up such that the reference line is 1.5 ± 0.2 mm above the top of the stopper.

12.1.5 If the jacket is not an integral part of the cooling bath, place the jacket vertically to a depth of 85 ± 2 mm in the cooling bath (see 6.1.10), which is maintained at the temperature of –34 ± 0.5°C.

12.1.6 Insert the test jar assembly in a stable vertical position into the jacket.

12.1.7 With the stopcock (see 6.1.11) open to atmosphere, connect the pipet to the vacuum system (see 6.1.12 and 6.1.13) by means of flexible tubing attached to the stopcock (see Fig. 1). Switch on the vacuum source and regulate to ensure an air flow rate of 15 L/h in the vacuum regulator (see 6.1.13). Before starting a test, check that the U-tube manometer indicates a 200 ± 1 mm of water depression (2 ± 0.05 kPa).

12.1.8 Start the test immediately after inserting the test jar assembly into the jacket, but if the cloud point of the sample is known, it is permitted to wait until the specimen has cooled to a temperature of not less than 5°C above its cloud point.

12.1.9 When the specimen temperature reaches a suitable integer value, turn the stopcock (see 6.1.11) so that the filter assembly is connected to the vacuum source, causing the specimen to be drawn through the wire mesh into the pipet; simultaneously start the stopwatch.

12.1.10 When the specimen reaches the mark on the pipet, stop the stopwatch and turn the stopcock to its initial position to vent the pipet and so allow the specimen to return to the test jar.

12.1.11 If the time taken to reach the mark exceeds 60 s on the first filtration, abandon the test and repeat it on a fresh portion, starting at a higher temperature.

12.1.12 Repeat the operations (see 12.1.9 to 12.1.10) for each 1°C decrease of the specimen temperature until the temperature is reached at which the pipet is not filled to the 20 mL mark within 60 s. Record the temperature at which this last filtration was commenced as CFPP (see Section 13).

NOTE 6—A small minority of samples may exhibit anomalous aspiration behavior, which can be detected by examining the observed aspiration times. This behavior is marked by an unexpected reduction in the time taken to fill the pipet, after which aspiration time again continues to increase progressively, until the failure limit of 60 s is reached.

12.1.13 If the filter has not plugged when the temperature of the specimen reaches –20°C, continue the test by using a second cooling bath maintained at –51 ± 1°C, quickly transferring the test jar and filtration assembly to a new jacket placed on the second cooling bath. Alternatively, for single bath apparatus, adjust the refrigeration unit to –51 ± 1°C. The new temperature must be reached within 2 min 30 s of the adjustment. Repeat the operations 12.1.9 to 12.1.10 to each 1°C decrease of the specimen temperature.

12.1.14 If the filter has not plugged when the temperature of the specimen reaches –35°C, continue the test by using a third cooling bath maintained at –67 ± 2°C by quickly transferring the test jar and filtration assembly to a new jacket placed on the second cooling bath. Alternatively, for single bath apparatus, adjust the refrigeration unit to –67 ± 2°C. The new temperature must be reached within 2 min 30 s of the adjustment. Repeat the operations 12.1.9 to 12.1.10 at each 1°C decrease of the specimen temperature.

12.1.15 If the filter has not plugged when the temperature of the specimen reaches –51°C, discontinue the test (see Section 13).

12.1.16 If, after cooling in accordance with 12.1.12, 12.1.13, and 12.1.14, the specimen fills the pipet to the mark in less than 60 s, but does not flow back completely into the test jar when the pipet is vented to atmosphere through the stopcock (see 6.1.11) before the start of the next aspiration, record the temperature at the commencement of the filtration as the CFPP (see Section 13).

12.2 *Automated Apparatus*:

12.2.1 Check that the cooling bath is operating and has reached the temperature required as specified in the manufacturer's instructions.

12.2.2 Pour the filtered specimen (see Section 9) into the clean and dry test jar to the 45 mL mark.

12.2.3 Close the test jar with the stopper (see 6.1.7) carrying the pipet with filter unit (see 6.1.8) and the platinum resistance thermometer. Adjust the apparatus in such a way that the bottom of the filter unit (see 6.1.8.2(*5*)) rests on the bottom of the test jar, and position the thermometer so that its lower end is 1.5 ± 0.2 mm above the bottom of the test jar. Take care to ensure that no part of the thermometer is in contact with the side of the test jar or the filter body.

NOTE 7—The precise positioning of the thermometer in the test jar is a critical parameter of this test method. The position of the lower end of the thermometer above the bottom of the test jar can be indirectly measured by marking the stem of the thermometer flush with the stopper (see 6.1.7) when the lower end of the thermometer is just touching the bottom of the test jar, and then pulling the thermometer up such that the reference line is 1.5 ± 0.2 mm above the top of the stopper.

12.2.4 If necessary, reconnect the pipet to the vacuum system. Switch on the vacuum source and regulate to ensure an air flow rate of 15 L/h in the vacuum regulator. Check that the U-tube manometer (if used) indicates a 200 ± 1 mm depression (2 ± 0.05 kPa) or that the electronic vacuum regulator indicates a pressure of 2 ± 0.05 kPa.

12.2.5 Press the start button immediately after insertion of the test jar assembly. If the cloud point is known, aspiration of the specimen through the filter may be set to start when it has cooled to a temperature not less than 5°C above the cloud point. The apparatus will carry out the test procedure filtering the specimen at each 1°C decrease if temperature and measuring the filtering time. If the time to reach the 20 mL mark exceeds 60 s on the first filtration, the test is to be abandoned and repeated on a fresh specimen starting at a higher temperature. The apparatus will record the first temperature at which the specimen fails to reach the 20 mL mark in less than 60 s or fails to flow back into the test jar when the vacuum is cut off as CFPP (see Section 13). The test will be discontinued if the specimen reaches −51°C without plugging (see Section 13). During the procedure, the apparatus will automatically change the cooling bath temperature as indicated below.

	Bath Temperature
Start of test	−34 ± 0.5°C
When (if) specimen reaches −20°C	−51 ± 1°C
When (if) specimen reaches −35°C	−67 ± 2°C

NOTE 8—A small minority of samples may exhibit anomalous aspiration behavior, which can be detected by examining the aspiration times recorded in the test printout for signs of an unexpected reduction in the time taken to fill the pipet, after which aspiration time again continues to increase progressively until the failure limit of 60 s is reached.

12.2.6 If the automated CFPP apparatus used does not incorporate a lower light sensor, it shall only be used if the test sequence is observed as in the manual procedure (see 12.1.16), so that any fuels not flowing back into the test jar as described are detected and reported accordingly.

13. Report

13.1 Report the temperature read or indicated at the beginning of the last filtration to the nearest 1°C (see 12.1.12, 12.1.16, and 12.2.5) as the CFPP.

13.2 If the specimen has reached −51°C without plugging (see 12.1.15 and 12.2.5) report as "Not plugged at −51°C."

13.3 The report shall contain at least the following information:

13.3.1 The type and identification of the product under test;

13.3.2 A reference to this test method;

13.3.3 The sampling procedure used (see Section 8);

13.3.4 The result of the test (13.1 or 13.2);

13.3.5 Any deviation from the procedure described (see Note 6 and Note 8); and

13.3.6 the date of the test.

14. Precision and Bias

14.1 The precision of this procedure as determined by the statistical examination of the interlaboratory test results is as follows:

14.2 *Repeatability*—The difference between results obtained on the same day by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, with normal and correct operation of the test method, exceed 1.76°C only in one case in twenty.

14.3 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values indicated by the formula:

$$0.102\ (25-X)°C$$

where: X is the average of the two results being compared, only in one case in twenty.

NOTE 9—The interlaboratory test program used to determine the precision of this test method was carried out in 1988 by the IP. The program involved 46 laboratories and 5 samples, ranging in CFPP values from 0°C to −33°C. Extrapolations to measurements more than a few degrees outside this range are unsupported by the data. The raw data from the 1988 program was reanalyzed in 1997 using the ASTM D2PP program. The report of the reevaluation is available from ASTM Headquarters.[7]

14.4 *Bias*—The procedure in this test method has no bias because the value of CFPP can be defined only in terms of a test method.

14.5 *Relative Bias*—The current interlaboratory tests confirm that there is no relative bias between the manual and automated apparatuses. Both apparatuses are suitable for reference purposes.

15. Keywords

15.1 automated cold filter plugging point; cold filter plugging point (CFPP); diesel; domestic heating fuels; filterability; manual cold filter plugging point

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1452.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 6371–99) that may impact the use of this standard.

(*1*) Modified 7.3 to remove the reference to "lintless."

Total Acid Number (TAN) (ASTM D664)

DOC316.52.93095

Potentiometric titration
Application: Petrochemical oils

1. Introduction

This working procedure deals with the titration of Total Acid Number (TAN) in oil.

2. Principle

The working procedure determines the sum of all acid compounds present in petrochemical samples by an acid-base titration using KOH as titrant. TAN is expressed in mg of KOH per g of sample.

Since samples are non-aqueous, they are diluted in a mix of chloroform and isopropyl alcohol. The solvent for KOH is isopropyl alcohol.

3. Electrode and reagents

Electrodes: Glass pH electrode, PHG201-8

Ag/AgCl reference electrode, REF361: the reference electrode has a sleeve junction with important diffusion in order to prevent clogging from oily samples. The REF361 is delivered filled with aqueous KCl solution. Empty this solution, rinse the electrode with deionized water then with isopropyl alcohol and fill it with a 1M LiCl alcoholic solution

Legacy adapter: the working electrode PHG201-8 is connected to the BNC socket, and the reference electrode REF361 is connected using a BNC-to-banana connector

Titrant: KOH 0.1 eq/L in isopropyl alcohol. It is recommended to buy a commercial 0.1 N alcoholic potassium hydroxide solution. Titer is not stable so a titrant calibration is recommended before use

Solvent: Mix 5 mL of water with 495 mL of isopropyl alcohol and add 500 mL of chloroform

Filling solution for the reference electrode: Dissolve 4.2 g of LiCl in 100 mL of isopropyl alcohol

Basic buffer:

1. **Prepare a stock solution**: weigh 27.8 g of m-nitrophenol, add 100 mL of isopropyl alcohol and 500 mL of KOH 0.1 N (in isopropyl alcohol), dilute to 1000 mL with isopropyl alcohol in a volumetric flask. Store the solution in a brown glass bottle and use within 2 weeks.
2. **Prepare the basic buffer solution**: add 10 mL of the stock solution to 100 mL of solvent. Use this solution within 1 hour.

Standard for titrant calibration: Potassium hydrogen phthalate, molar weight = 204.22 g/mol

pH standards: Colored 4.01, 7.00, 10.01 (part numbers 2283449, 2283549, 2283649)

Deionized water

4. Ranges and settings

4.1. Default parameters

The working procedure is described using the following parameters:

- m sample = 2.000 g
- Syringe volume = 5 mL

The default syringe volume for the AT1000 is set to 10 mL. This application needs a 5 ml syringe. When loading an application, if the message **syringe to replace** is displayed, change the syringe volume in the **Syringe management** option of the **Maintenance** menu.

4.2. Working range

For a 2 g (± 0.2 g) sample and maximum KOH volume of 10 mL (two syringes of 5 mL), samples of up to 20 mg/g can be analyzed.

For more concentrated oils, it is recommended to weigh a smaller amount of sample. For low concentrations it is also possible to analyze more than 2 g of oil. The ASTM D664 standard gives the following indications:

Total Acid Number (mg/g)	Sample mass (g)	Weighing accuracy (g)
0.05 – < 1.0	20.0 ± 2.0	0.10
1.0 – < 5.0	5.0 ± 0.5	0.02
5.0 – < 20	1.0 ± 0.1	0.005
20 – < 100	0.25 ± 0.02	0.001
100 – < 260	0.1 ± 0.01	0.0005

4.3. Settings

Name	Default parameter	Unit
Sample		
Name	Sample	
Amount	2	[g]
Amount min	0.1	[g]
Amount max	22	[g]
Titrant		
Name	KOH	
Titrant concentration	0.1000	[eq/L]
Syringe	Syringe 1	
Probes		
Recommended pH probe	PHG201	
Recommended reference probe	REF361	
Rinsing step 1 (solvent)		
Active	Yes	
Time	30	[s]
Stirring speed	25	[%]
Rinsing step 2 (water)		
Active	Yes	
Time	30	[s]
Stirring speed	25	[%]
Rinsing step 3 (solvent)		
Active	Yes	
Time	30	[s]
Stirring speed	25	[%]
IP titration		
Stirring speed	20	[%]
Measured parameter		[mV]
Predose	0	[mL]
Max volume stop point	10	[mL]
Stop on last EQP	Yes	
Delay	15	[s]

Min increment size	0.03	[mL]
Max increment size	0.15	[mL]
Result 1 name	TAN (mgKOH/g)	
R1 resolution	3 decimals	
R1 min	0.05	[mg/g]
R1 max	260	[mg/g]
R1 QC min	0.05	[mg/g]
R1 QC max	260	[mg/g]
R1 EQP index	1	
R1 molar weight	56.11	[g/mol]

4.4. Modification of the settings

The parameters are defined in order to have the best compromise between accuracy and titration time.

For higher concentration with a high titrant volume, titration time can be reduced with an addition of titrant (predose) at the beginning of the titration. Enter the predose volume (in mL) and the stirring time after the addition in the application edit window.

5. Titration procedure

Launch the application **TAN**.

On the first screen, in **Sample type** choose **Define blank** and press **Start**. Follow the rinsing instructions on the screen. Then, place an empty beaker with a stir bar under the probe holder. Add 75 mL and press **OK**. Make sure that both electrodes are immersed. At the end of the titration, the equivalent volume corresponding to the blank is displayed and automatically recorded. Press **Next** and chose **New sample**.

Weigh approximately 2 g of oil in a 100 mL beaker. In **Sample type** choose **Sample with blank** and press **Start**. Follow the rinsing instructions on the screen. Then, place the beaker containing the sample and a stir bar under the probe holder. Add 75 mL of solvent and press **OK**. Make sure that both electrodes are immersed. At the end of the titration, TAN is displayed in mg of KOH per g of sample. This result is calculated taking into account the blank determined previously.

By pressing **Next** it is possible to:

- Replicate the sample. This is used to study the repeatability by analyzing several samples successively. At the end of each titration, a window displays the average value, the standard deviation (SD in g/L) and the relative standard deviation (RSD in %).
- Analyze a new sample. Another titration can be started but no Standard Deviation and RSD value will be made.

If no inflection is detected then it is possible to use endpoint detection.

- First, the endpoint potential must be determined. Immerse the electrodes in the basic buffer solution. Press **Maintenance > Live measure**, wait for a stable mV signal and read the mV value. Then, in **Settings > Applications > Edit** chose **TAN Endpoint**. In section **Method: Titration** type this mV value as the ordinate for the equivalence point. Launch the application **TAN Endpoint** and proceed as for the **TAN** application.

6. Results

6.1. Result calculation

The calculation used is:

$$
\begin{aligned}
\mathrm{TAN\ (mgKOH/g)} &= \frac{\mathrm{C_{titrant}\ (eq/L) \times V_{titrant}\ (mL)}}{\mathrm{n_{e\text{-}\ titrant} \times m_{sample}\ (g)}} \times \mathrm{M_{KOH}\ (g/mol)} \\
&= \frac{\mathrm{0.1\ (eq/L) \times V_{titrant}\ (mL)}}{\mathrm{1 \times m_{sample}\ (g)}} \times \mathrm{56.11\ (g/mol)}
\end{aligned}
$$

6.2. Experimental results

These results are indicative and have been obtained for 3 TAN standards (0.52, 2.02 and 4.57 mg/g) with 5 determinations for each standard.

- Inflection

Standard 0.52 mg/g	
Mean TAN (mg/g)	0.518
Standard deviation (mg/g)	0.019
Relative standard deviation (%)	3.6

Standard 2.02 mg/g	
Mean TAN (mg/g)	2.114
Standard deviation (mg/g)	0.085
Relative standard deviation (%)	4.0

Standard 4.57 mg/g	
Mean TAN (mg/g)	4.661
Standard deviation (mg/g)	0.175
Relative standard deviation (%)	3.8

- Endpoint

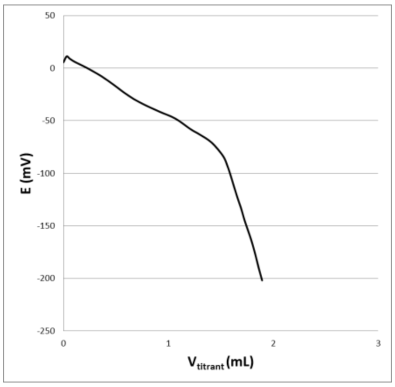
Standard 0.52 mg/g	
Mean TAN (mg/g)	0.507
Standard deviation (mg/g)	0.028
Relative standard deviation (%)	5.5

Standard 2.02 mg/g	
Mean TAN (mg/g)	1.923
Standard deviation (mg/g)	0.024
Relative standard deviation (%)	1.2

Standard 4.57 mg/g	
Mean TAN (mg/g)	4.793
Standard deviation (mg/g)	0.194
Relative standard deviation (%)	4.1

6.3. Example of a titration curve

This curve has been obtained during the analysis of one of the standards.



7. Recommendations

Always rinse the pH probe and the delivery tip between measurements.

Refill the electrode regularly with LiCl 1M in isopropyl alcohol to maintain the level of internal solution around 1 cm (0.4 inches) below the refill hole.

8. Bibliography

- *Standard ASTM D664*

9. Appendices

9.1. Electrode testing and storage

It is recommended to check the electrodes behavior when first put into use, or when new electrodes are installed, and retest at intervals thereafter. Rinse the electrodes with solvent and then with deionized water. Dip them into the pH 4 aqueous buffer solution. Press **Maintenance > Live measure**.

Read the mV value after stirring for 1 minute. Remove the electrodes, rinse with water and dip them into the pH 7 aqueous buffer solution. Read the mV value after stirring for 1 minute. Calculate the mV difference. A good electrode system will have a difference of at least 162 mV (20 to 25 °C). If the difference is less than 162 mV, lift the sleeve of the reference electrode and make sure of the electrolyte flow. Repeat the measurements. If the difference is still less than 162 mV, clean or replace the electrode(s).

When not in use, store the reference electrode in LiCl electrolyte and the glass electrode in an acidic aqueous media (pH 4 to 5.5).

9.2. Titrant calibration

The sodium hydroxide solution can be calibrated. Its exact concentration can be determined from an acid-base titration using potassium hydrogen phthalate.

Weigh 60 mg of potassium hydrogen phthalate powder in a 100 mL beaker and use a graduated cylinder to add 70 mL of boiled deionized water (CO_2-free). Put in a stir bar, dip the probes and the delivery tip into the solution and launch the titrant calibration sequence. When prompted, type in the exact weighed amount of powder.

At the end of the titrant calibration, titer (eq/L) is displayed and the user can reject, replicate, or save the result. The saved value will be used for calculations.

Default settings for titrant calibration

Name	Default parameter	Unit
Titrant		
Name	KOH	
Titrant concentration	0.1000	[eq/L]
Syringe	Syringe 1	
Standard		
Name	Potassium hydrogen phthalate	
Amount	60	[mg]
Amount min	40	[mg]
Amount max	80	[mg]
Molar weight	204.22	[g/mol]
EP titration		
Stirring speed	20	[%]
Measured parameter		[mV]
Predose	0	[mL]
Max volume stop point	10	[mL]
Stop on last EQP	True	
Delay	0	[s]
Min increment size	0.05	[mL]
Max increment size	0.5	[mL]
Result name	Titer	
Result resolution	4 decimals	
Result min	0.09	[eq/L]
Result max	0.11	[eq/L]

NSAI
Standards

Irish Standard
I.S. EN 15751:2014

Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by accelerated oxidation method

I.S. EN 15751:2014

Incorporating amendments/corrigenda/National Annexes issued since publication:

The National Standards Authority of Ireland (NSAI) produces the following categories of formal documents:

I.S. xxx: Irish Standard — national specification based on the consensus of an expert panel and subject to public consultation.

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NOTE: The date of any NSAI previous adoption may not match the date of its original CEN/CENELEC document.

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EUROPÄISCHE NORM

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English Version

Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by accelerated oxidation method

Carburants pour automobiles - Esters méthyliques d'acides gras (EMAG) et mélanges avec du gazole - Détermination de la stabilité à l'oxydation par méthode d'oxydation accélérée

Kraftstoffe für Kraftfahrzeuge - Kraftstoff Fettsäuremethylester (FAME) und Mischungen mit Dieselkraftstoff - Bestimmung der Oxidationsstabilität (beschleunigtes Oxidationsverfahren)

This European Standard was approved by CEN on 20 December 2013.

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Contents

Foreword

This document (EN 15751:2014) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2014 and conflicting national standards shall be withdrawn at the latest by September 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15751:2009.

Significant changes between this document and EN 15751:2009 are:

a) the limitation of the scope of the method to a maximum induction period of 48 h, reflecting the precision range of the method,

b) indication of a potential alteration of the induction period in the presence of cetane enhancers,

c) inclusion of the results of a short applicability check on non-petroleum based (such as Fischer-Tropsch synthesis or hydrotreatment process originated) diesel type of fuels (see Introduction),

d) editorial changes in order to clarify the test procedure.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This document is based on EN 14112 [1], which was specifically adapted for the determination of oxidation stability of fatty acid methyl esters (FAME). This method had been developed under CEN/TC 307 (Fats and oils). At the time of development the method was applicable for FAME fuel according to EN 14214 [2], but questions remained on the accuracy towards blends of FAME and diesel fuel.

The modifications to EN 14112 as given in this document, allow application of this test method for oxidation stability for pure FAME and diesel/FAME blends at various levels.

The goal was to have one single test method for FAME fuel, diesel/FAME blends and pure diesel fuels. Although the modifications cover FAME fuel and diesel/FAME blends, CEN/TC 307 decided that it was better to retain EN 14112 for methyl esters and publish a separate standard for all automotive fuel and heating oil applications, as the use of 'diesel and diesel blends' falls out the scope of CEN/TC 307.

While developing the fuels specification for paraffinic diesel fuel, three labs executed a small test on neat fuel and on 7 % (*V/V*) FAME blend based on product originating from both Fischer-Tropsch synthesis and hydrotreatment process. No indications towards a different interaction with the methodology of this document were found, so it was concluded that the stability of these paraffinic diesel fuels can be determined with the test method described in this document. The stability of these products usually is that high that the results do not match the scope of this European Standard.

The modifications required a new validation covering pure FAME, diesel/FAME blends and pure diesel fuels which resulted in the fact that the method is not suitable for pure petroleum-based diesel fuels.

1 Scope

This European Standard specifies a test method for the determination of the oxidation stability of fuels for diesel engines, by means of measuring the induction period of the fuel up to 48 h. The method is applicable to fatty acid methyl esters (FAME) intended for the use as pure biofuel or as a blending component for diesel fuels, and to blends of FAME with diesel fuel containing 2 % (*V/V*) of FAME at minimum.

NOTE 1 EN 14112 [1] describes a similar test method for oxidation stability determination of pure fatty acid methyl esters (see the Introduction to this European Standard).

NOTE 2 For induction periods higher than 48 h the precision is not covered by the precision statement of this method. The limit values of the relevant fuel standards are well within the scope of this test method.

NOTE 3 The presence of cetane improver can reduce the oxidation stability determined by this test method. Limited studies with EHN (2-ethyl hexyl nitrate) indicated, however, that the stability is reduced to an extent which is within the reproducibility of the test method.

NOTE 4 For the purposes of this European Standard, the term “% (*V*/*V*)” is used to represent the volume fraction (φ) of a material.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170)*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

induction period

time which passes between the moment when the measurement is started and the moment when the formation of oxidation products begins to increase rapidly

3.2

oxidation stability

induction period determined according to the procedure specified in this European Standard, expressed in hours

4 Principle

A stream of purified air is passed through the sample which has been heated to the target temperature which is 110 °C in the usual application of the method. Volatile compounds are formed during the oxidation process. They are, passed together with the air into a flask containing demineralised or distilled water, equipped with a conductivity electrode. The electrode is connected to a measuring and recording device. It indicates the end of the induction period by rapid increase of the conductivity due to the dissociation of volatile carboxylic acids produced during the oxidation process and absorbed in the water. For more details on the background of the method, see Annex A.

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