



Universidad de Guayaquil

UNIVERSIDAD DE GUAYAQUIL
FACULTAD DE INGENIERÍA QUÍMICA
CARRERA DE INGENIERÍA QUÍMICA

**OBTENCIÓN DE ÉSTER METÁLICO (BIODIESEL), MEDIANTE REACCIÓN
DE TRANSESTERIFICACIÓN DEL ACEITE DE MORINGA OLEÍFERA**

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TUTOR:

ING. CARMEN LLERENA R, MSc.

GUAYAQUIL, SEPTIEMBRE 2019



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OBTENCIÓN DE ÉSTER METÁLICO (BIODIESEL), MEDIANTE REACCIÓN
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Este trabajo de titulación va dedicado a todas aquellas personas, amigos, profesores y familiares que nos aportaron de diferentes formas, con conocimiento, consejos y animo a lo largo de nuestra vida universitaria para lograr esta meta propuesta.

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Bryan Nivaldo Guerrero De la A

Karla Nicole Kocher Solano

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Bryan Nivaldo Guerrero De la A

Karla Nicole Kocher Solano



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**OBTENCIÓN DE ÉSTER METÍLICO (BIODIESEL), MEDIANTE REACCIÓN
DE TRANSESTERIFICACIÓN DEL ACEITE DE MORINGA OLEÍFERA**

Autores: Bryan Nivaldo Guerrero De La A, Karla Nicole Kocher Solano.

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RESUMEN

En la actualidad la humanidad enfrenta varios problemas los cuales uno de ellos es la sobreexplotación de los recursos naturales no renovables que provocan la combustión de combustibles fósiles además de procesos industriales no controlados. Por ende, se planteó en obtener éster metílico a partir del aceite de la semilla de moringa. Se empleó el método de soxhlet para la respectiva extracción de aceite siendo este la materia prima, obteniendo un 26% de rendimiento, dentro de todos los estudios realizados se desarrolló un diseño experimental para la producción de dicho combustible, mediante la reacción de transesterificación se obtiene como productos, biodiésel y glicerina donde reacciona el aceite con metanol e hidróxido de sodio como catalizador. Nuestro producto de interés se caracterizó, evaluando todas sus características físicas para luego ser comparado bajo la norma ASTM para biodiésel D6751-09 y un diésel comercial, concluyendo que se pudo obtener un biocombustible que cumple con la gran parte de las especificaciones estipuladas en dichas normas, además que aporta en disminuir el impacto ambiental producido por los combustibles fósiles.

Palabras claves: Moringa, Rendimiento, Transesterificación, Biodiesel



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**OBTENCIÓN DE ÉSTER METÁLICO (BIODIESEL), MEDIANTE REACCIÓN
DE TRANSESTERIFICACIÓN DEL ACEITE DE MORINGA OLEÍFERA**

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ABSTRACT

Humanity now faces several problems, which one of which is the overexploitation of non-renewable natural resources that cause the burning of fossil fuels in addition to uncontrolled industrial processes. Therefore, it was considered to obtain methyl ester from the oil of the moringa seed. The soxhlet method was used for the respective oil extraction being the raw material, obtaining 26% yield, within all the studies carried out an experimental design was developed for the production of said fuel, through the Transesterification reaction is obtained as biodiesel and glycerin products where the oil reacts with methanol and sodium hydroxide as a catalyst. Our product of interest was characterized, evaluating all its physical characteristics and then being compared under the norm ASTM para biodiésel D6751-09 and a commercial diesel, concluding that a biofuel could be obtained that meets the large part of the specifications stipulated in these standards, which also contributes to reducing the environmental impact produced by fossil fuels.

Keywords: Moringa, Yield, Transesterificación, Biodiesel

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INTRODUCCIÓN

En este trabajo de titulación se presenta una investigación experimental sobre la obtención de un biodiésel a partir del aceite de moringa oleífera como alternativa de producción de un combustible renovable que aporta de forma positiva en el impacto ambiental. La moringa oleífera es un árbol que fue descubierto al sur del Himalaya pero en la actualidad esta diseminado en todo el planeta, ya que se adapta con facilidad en diferentes condiciones que se encuentre el suelo o el clima; además de conocer que posee muchos beneficios en todas sus partes, ya sea su valor nutricional y los elevados rendimientos de biomasa los cuales permiten que sea recurso de importante en los sistemas de producción. [1]

Ante todos los estudios que sean realizado en base a los problemas que provocan la emisión de combustibles fósiles, se han comenzado a desarrollar distintos proyectos de combustibles y fuentes de energías renovables el cual argumenta la necesidad de producirlos. Este trabajo tiene enfoque cuantitativo y cualitativo ya que recolecta y analiza datos, además de un arduo estudio experimental para llevar a cabo la obtención del producto de interés. Se desarrolló un diseño experimental utilizando la semilla moringa, la cual es sometida a un método de extracción para obtención del aceite, siendo este la base fundamental y primordial para la producción del biocombustible. Para luego someterse a un proceso de transesterificación con las condiciones óptimas experimentadas, en donde reacciona el aceite obtenido con un alcohol y además actúa un catalizador, que ayuda en la producción y obtención del éster metílico y glicerina como subproducto. En el país no existen Normas vigentes que especifiquen los valores de cada característica que debe poseer un biodiesel,

pero se tomará como referencias las Normas Técnicas Colombianas NTC DE 100/04, incluso se realizarán comparaciones con un diésel comercial.

El presente trabajo tiene como objetivo Obtener éster metílico (biodiesel) mediante la reacción de transesterificación del aceite extraído de la moringa oleífera, para contribuir con una fuente alterna de combustibles. En el primer capítulo, se desarrolla la formulación, problemática y la sistematización de la investigación, además de los límites y objetivos que se cumplirán en el proceso de este trabajo. En el segundo capítulo, se sustentan todas las teorías fundamentales que son el estudio previo que se realiza para abarcar todos los conocimientos y llevar a cabo el desarrollo del estudio. En el capítulo tres, se lleva a cabo la experimentación del proyecto de forma explícita y detallada, la cual se desarrolla en base a la metodología y diseño experimental que se plantea en el proyecto. En el cuarto capítulo, se plantean los resultados obtenidos, los cuales se interpretarán según los análisis al que fueron sometidos con la finalidad de darle soporte de validez al biodiésel que se obtiene. Finalmente, en el capítulo cinco se concluye en base a todos los objetivos que fueron planteados, así como también se formulan algunas recomendaciones, que son necesarias tenerla en cuenta en el proceso de obtención del biodiésel.

En el presente trabajo de investigación se desarrollará una serie de condiciones normales de reacción hasta poder diseñar la de mejor rendimiento y viable, la cual servirá como guía de estudio para contribuir en planes de desarrollo energético y generar un beneficio social y ambiental, además de promover ideas y alternativas para la disminución del impacto que ha provocado a lo largo de este tiempo los combustibles fósiles.

CAPÍTULO I

1.1. Marco lógico

1.1.1. Planteamiento del problema

La problemática existente radica en que un biodiesel es un combustible alternativo que se obtiene principalmente a partir de aceites comestibles con el fin de superar estos inconvenientes, el uso del aceite de moringa no es frecuentemente el de un aceite comestible, por lo cual se establece como una materia prima del que se puede sintetizar un éster metílico (biodiesel); un sustituyente al diésel comercial que, actualmente constituye; entre otros combustibles fósiles uno de los mayores contaminantes ambientales.

En nuestro país existen cerca de 400 ha de cultivo de moringa oleífera, una cantidad importante que hasta ahora es desaprovechada, y que podría brindar grandes beneficios a la industria del combustible, sintetizando un biocombustible líquido, que ayudaría a disminuir significativamente las emisiones de gases tóxicos y de otras sustancias volátiles cuando se queme como combustible. [2]

1.2. Formulación y sistematización de la investigación

1.2.1. Formulación del problema de investigación

¿Cuáles son las condiciones óptimas de operación y reacción en el desarrollo experimental del aceite de moringa oleífera, obteniendo como producto biodiesel mediante la reacción de transesterificación empleando hidróxido de sodio como catalizador?

1.2.2. Sistematización del problema

¿Cuál es el rendimiento que se obtuvo en la extracción del aceite de la semilla de moringa a partir del método de Soxhlet?

¿Qué diferencia existe entre el método de Soxhlet y el prensado en frío para una eficiente extracción?

¿Qué normas vigentes cumple al ser caracterizado el biodiesel obtenido, a partir del aceite de moringa respecto a sus propiedades?

1.3. Justificación

1.3.1. Justificación teórica

El uso del aceite de moringa para obtener biodiesel como combustible alternativo producido por la reacción de un triglicérido (presente en el aceite) con alcohol metanol; empleando un catalizador como hidróxido de sodio que permite obtener una mezcla de ésteres metílicos.

La investigación se realiza con el fin de cuantificar y cualificar las mejores condiciones de reacción de transesterificación entre el aceite extraído de moringa oleífera y un alcohol como el metanol, para así promover la producción de biocombustibles con materia prima renovable en nuestro país.

1.3.2. Justificación metodológica

El proyecto se enfoca en la producción del éster metílico (biodiesel) que se obtiene mediante la reacción de transesterificación del aceite de moringa y metanol, por ser más barato y más reactivo que el etanol, trabaja bajo diferentes condiciones de reacción tomando en cuentas las variables principales, tales

como: temperatura, tiempo y rpm (agitación), y realizando variaciones de acuerdo al diseño experimental planteado, donde las variables son: relación molar, reactivo limitante y catalizador, estos esteres etílicos de ácidos grasos presentan diversas viscosidades, punto de nube y diversas fluidez y además de determinar si el producto obtenido cumple con las normativas vigentes; NTE INEN 2482 y ASTM D6751 - 18, y que sea una alternativa al uso de los combustibles fósiles contaminantes.

1.3.3. Justificación práctica

La obtención y caracterización de un biodiesel a partir del aceite extraído de la moringa, con metanol empleando como catalizador al hidróxido de sodio; para formar ésteres metílicos de los ácidos grasos a partir de aceites no comestibles con el fin de sustituir materias primas tradicionales.

Estos aceites no comestibles tienen altas cantidades de ácidos grasos, lo que ocasiona pasos químicos adicionales, que aumentan los costos de producción, pero los beneficios al medio ambiente son favorables y provoca el uso de este tipo de combustible.

1.4. Objetivos

1.4.1. Objetivo general

Obtener éster metílico (biodiesel) mediante la reacción de transesterificación del aceite extraído de la moringa oleífera, para contribuir con una fuente alterna de combustibles.

1.4.2. Objetivos específicos

- I. Extraer aceite de Moringa Oleífera mediante el método Soxhlet, y establecer condiciones de operación óptimas para la reacción de transesterificación.
- II. Caracterizar el biodiesel obtenido.
- III. Evaluar y comparar los resultados de la caracterización frente a las normativas internacionales vigentes.

1.5. Delimitación de la investigación

El presente trabajo de investigación se enfoca en la obtención de un biodiesel y está limitado a diversos campos como lo son las ciencias básicas, bioconocimiento y del desarrollo industrial además con las sublíneas de energía renovable.

1.6. Hipótesis

La reacción de transesterificación a condiciones normales permite obtener un biodiesel a partir del aceite de moringa oleífera con sus características correspondientes.

1.7. Variables

1.7.1. Variable dependiente

- Aceite de Moringa.

1.7.2. Variable independiente

- Condiciones de la extracción con solvente
- Condiciones para la reacción de transesterificación: tiempo, temperatura, agitación.

1.8. Operacionalización de variables

Tabla 1. Manejo de variables

Tipo de Variable	Variable	Definición Operacional	Indicador de Medición	Norma o Método	Unidad de medida
Dependiente	Biodiesel	Combustible orgánico que se obtiene a partir de la reacción de transesterificación	Poder calorífico	ASTM-240	Btu/lb
			Densidad	ASTM-287	-
			Viscosidad	ASTM-445	mm^2/s^2
			Punto de inflamación	ASTM-93	°C
			Destilación	ASTM-86	°C
			Contenido de agua	ASTM-95	%V
Independiente	Aceite de Moringa	Moringa Oleífera materia prima para la obtención de biodiesel.	Temperatura	Método de Soxhlet	°C
			Presión		psi

Fuente: (Elaboración propia)

CAPÍTULO II

2.1. Marco Referencial

2.1.1. Antecedentes

Una de las alternativas de solución para el medio ambiente es fomentar el uso de combustibles orgánicos como una fuente de energía renovable ya que contribuye a la reducción del consumo de combustibles fósiles los cuales generan emisiones de gases que con el pasar del tiempo provocaran muchos inconvenientes. [3]

En el año de 1900 Rudolf Diésel dio a conocer este combustible orgánico, ya que utilizo aceite de maní para emplearlo en un motor diésel, el cual dio a conocer según varios estudios, que podía ser utilizado en forma pura o mezclado con diésel. [4]

En Europa se desarrolló la primera planta de biodiésel en el año 1901, Austria fue el primero en la producción del mismo y el que desarrollo las especificaciones de calidad, se produjo el biodiésel con aceite de colza utilizándolo como materia prima y luego de varias estudios se pudo publicar el primer estándar de calidad para los metilésteres; desde ese momento la industria de producción del biodiesel ha aumentado significativamente con el pasar del tiempo la cual les permite evolucionar y dar nuevas alternativas de materias primas que sean rentables para la económica y medio ambiente del mundo como lo son los aceites vegetales, aceite reutilizado, grasas animal, etc.

De los países de sur américa, Colombia es el que empezó a tener interés en este combustible en ciertos centros de investigaciones y además universidades, se han desarrollo proyectos en base al biocombustible, tanto así

que el instituto colombiano de normas técnicas y certificación en el 2006 ratificó la NTC 5444 como la norma para biodiésel para uno de motores diésel, Colombia es reconocida como país líder en el desarrollo y producción del biodiésel en el ámbito latinoamericano. [5]

En nuestro país la producción del biocombustible todavía se presenta en fase de experimentación, aunque si se han desarrollados proyectos exitosos los cuales se han basado obtener biodiésel a partir de semillas de piñón, CELEC EP termo Pichincha implementó un laboratorio de control que se encarga de los biocombustibles e hidrocarburos con el fin de aportar mucho más en los procesos operativos y de mantenimiento, que contribuirá con las reducciones de emisiones y aprovechar tierras en los que se puedan cosechar diferentes materias primas para la elaboración de dicho producto y así mejorar la calidad de vida. [6]

2.2. Marco Teórico

2.2.1. Moringa

Pertenece a la familia de Moringaceae de Himalaya y en la actualidad es cultivado en todas las regiones tropicales, subtropicales y semiáridas del mundo. Es un árbol que puede ser utilizado como alimento que posee propiedades medicinales y antimicrobianas además su aceite se considera una fuente en la obtención de biocombustible. Las hojas, flores y las raíces son aprovechadas al máximo por el hombre, las hojas posee ricas vitaminas y diferentes aminoácidos hasta para la producción de biogás, la corteza del tronco sirve para la absorción de metales pesados así como la elaboración de alfombras, el aceite se lo utiliza en las perfumerías, cosméticos y en la producción de biocombustibles. [7]



Figura 1. Países usuarios de Moringa. [8]

2.2.1.1. Características agrícolas

Se trata de un árbol no longevo, con un periodo de crecimiento rápido que mejora el suelo por los nutrientes que posee, protegiéndolo de la erosión, la desecación. Los agricultores dan a conocer que la Moringa oleífera es resistente a la sequía y prefiere suelos neutros o ligeramente ácidos. [8]



Figura 2. Cultivos de moringa en Naranjal-Guayas-Ecuador [8]

Un árbol siempre verde de tamaño pequeño, pero con un crecimiento acelerado alcanza una altura de 12m. Tiene una copa abierta y esparcida de

ramas un follaje plumoso de hojas pinnadas en tres, una corteza gruesa, blanquecina. [9]

2.2.1.2. Taxonomía

- Familia: Moringceas
- Origen: Capparidales
- Clase: Magnoleopesida
- Género: Moringa
- Especies: Arbórea, concanensis, drocanensi, drouhardtii. pygmeae, peregrina, ovalaifolia, rospoliana, stenetala, oleífera, borziana

2.2.2. Aceites Esenciales

2.2.2.1. Generalidades

Existen numerosas definiciones que abarca diferentes puntos de vista y tienden a tener algunas perspectivas ya sea químico, botánico o de industrial pero no abarca una definición en su totalidad. Se define como aceites esenciales como parte del metabolismo de una planta que se activa como defensa a factores ecológicos y ambientales dichos aceites en las plantas constituyen al 0,1 al 1% del peso seco. Estos son solubles en alcoholes y disolventes orgánicos además que son líquidos con poca solubilidad en agua. Son densos que el agua la mayoría de estos aceites excepto los de canela, además poseen alto índice de refracción. [10]

2.2.2.2. Definición

Los aceites esenciales son compuestos orgánicos, que almacenan y producen secretores de las plantas como alcoholes, acetonas, cetonas, éteres, aldehídos. Existen varios métodos de extracción del aceite, ya sea como

corriente de vapor de agua, por soxhlet, etc. Estos son líquidos volátiles a temperatura ambiente [10]

2.2.2.3. Clasificación de los aceites esenciales

Los Aceites Esenciales se clasifican en base a diferentes puntos de vista según:

Por su Consistencia:

- Fluidas: Líquidos y volátiles a temperatura ambiente.
- Bálsamos: Aceites muy viscosos, poco volátiles que tienden a polimerizarse.
- Oleorresinas: son semisólidas muy viscosas.

Por su Origen

- Naturales: Poseen un rendimiento bajo y son muy costosas, aunque se obtienen directamente de la planta y no sufren modificaciones ya sea físicas o químicas
- Artificiales: Estos aceites se obtienen a través de procesos que enriquecen la misma esencia y combinaciones de sus componentes.
- Sintéticas: Se trata de aceites económicos combinados de diferentes componentes como su nombre lo indican los cuales se producen con procesos de síntesis química. Son los más utilizados como los aromatizantes y saborizantes como lo es la esencia de vainilla, fresa, etc. [10]

2.2.2.4. Métodos de extracción de aceites

Para extraer aceites esenciales existen diferentes métodos y tecnologías. Cada método cumple diferentes funciones los cuales con varios estudios y análisis se determina cuál es el indicado, cada método de extracción influye

mucho en la calidad del aceite, además de que existe posibilidad de extraerse nuevos elementos que alteren el equilibrio del aceite.

2.2.2.4.1. Extracción por prensado

Método conocido como expresión, vegetal que se somete a presión ya sea en prensa continua o batch. Este método se trata de prensar el material vegetal de la cual se extrae el aceite en un rango de temperatura de 10°C a 20°C, para evitar la evaporación y pérdida del aceite. Si se prensa a temperaturas ambiente de 24°C a 26°C la fricción de la prensa se tiende a elevar, siendo así la temperatura de los líquidos 60°C hasta 65°C, lo que provoca que algunos componentes del aceite esencial se volatilicen.

2.2.2.4.2. Extracción por arrastre con vapor

Se trata de una extracción de vaporización del componente volátil, la cual se inyecta vapor de agua directamente a la mezcla que se lo conoce como vapor de arrastre. Se observa la presencia de dos fases insolubles orgánica y acuosa. Este método se trata de una destilación en donde se coloca una planta en el extractor que se somete a calentamiento en la caldera que se evapora el agua, el cual se evapora el aceite volátil en el extractor, que se condensa y posterior a eso se lleva a cabo la separación por diferentes densidades. [10]

2.2.2.4.3. Extracción por Hidrodestilación

El principio de la destilación en agua es llevar a estado de ebullición una suspensión acuosa de un material vegetal aromático, de tal manera que los vapores generados puedan ser condensados y colectados, el aceite que es inmisible en agua, se separa posteriormente. En la hidrodestilación el material vegetal siempre deben encontrarse en contacto con el agua, si el calentamiento

del equipo es con fuego directo, el agua presente en la cámara extractora debe ser suficiente y permanente para llevar a cabo toda la destilación a fin de evitar el sobrecalentamiento y carbonización del material vegetal, dado que este hecho provoca la formación de olores desagradables en el producto final; el material vegetal debe ser mantenido en constante agitación a fin de evitar aglomeraciones o sedimentación del mismo en el fondo del recipiente, lo cual puede provocar su degradación térmica. El tiempo total de destilación es función de los componentes presentes en el aceite esencial. Si el aceite contiene compuestos de alto punto de ebullición, el tiempo de destilación deberá ser mayor, los aceites esenciales obtenidos mediante destilación en agua normalmente presentan aromas más fuertes y un color más oscuro con respecto a los producidos por otros métodos. [10]

2.2.2.4.4. Extracción con ácidos

Este tipo de extracción se la realiza con sustancias básicas mezcladas con sustancias neutras o ácidas. Se utiliza el ácido clorhídrico para transformar los básicos y orgánicos en hidrocloruros solubles en agua.

2.2.2.4.5. Extracción con Solventes

Extracción sólida – líquido: esta operación es una de las más utilizadas en diversas industrias, la cual se extrae componentes que son solubles sólidos, teniendo la facilidad de emplear disolvente que permita obtener un aceite más rápido y que ocurra una extracción completa. Para este método influyen distintos factores como el tamaño de las partículas sólidas, la temperatura y el tipo de disolvente que se emplee.

Extracción líquida – líquido: este método consiste en la recuperación del soluto que se encuentra en una solución que está mezclado con un solvente, el cual se encuentra en una fase acuosa con un disolvente orgánico.

2.2.3. Método de extracción Soxhlet

El método de extracción soxhlet es uno de los más utilizados en lo que se refiere a una extracción sólido - líquido, debido a su poca complejidad y la gran eficiencia que este provee.

2.2.3.1. Origen del método soxhlet

En Mesopotamia desde el año 3500 A.C se comenzó a extraer materia orgánica con agua caliente, la cual se lo conoce como una extracción solido-líquido que ha sido de ayuda desde la antigüedad a obtener ciertas esencias de perfumes e infusiones. Luego gracias a los estudios del químico alemán Anselmo Payen pudo implementar un proceso automático, posteriormente el francés Franz Ritter Von Soxhlet, en el año 1879 propuso este método para determinar las grasas en los alimentos, el cual ha estado vigente 14 décadas. [11]

2.2.3.2. Funcionalidad del método soxhlet

Este método consiste en la solubilidad de las grasas con ayuda de un solvente orgánico, dicho disolvente se volatiliza por el calor y luego se recupera por condensación cumpliendo un ciclo de extraer y purificar de forma continua. Este método soxhlet es uno de los más utilizados para muestras sólidas a comparación de los otros, además que solo se utiliza en escala de laboratorio ya que industrial es muy elevado su costo. [12]

El equipo posee el mismo nombre del método y sirve para determinar las grasas de un alimento sólido, inicia con poseer una muestra seca y algo triturada para evitar que el agua actúe en el proceso y lo pueda alterar. Dicha muestra se coloca dentro del sifón con un pedazo de algodón, para luego verter el disolvente y este se somete a calentamiento a una temperatura adecuada y controlada hasta que alcance su punto de ebullición y esta suba en forma de vapor y hasta que llega al refrigerante y lo hace condensar, formando una recirculación, ya que se densa y regresa al sifón en estado líquido. Cuando el disolvente se condensa es donde tiene contacto con la muestra gota a gota, acumulando en el matraz las grasas de la muestra, se observa en base a la recirculación que el solvente torna a cambiar de tonalidad dependiendo del alimento que se está procesando. Como último paso se evapora lo que hemos obtenido para evitar restos de disolvente en el aceite. [11]

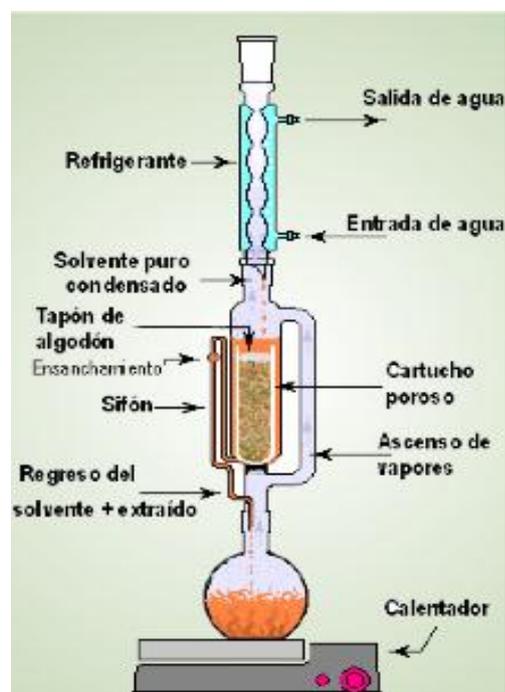


Figura 3. Extracción con Soxhlet: sifoneamiento del solvente.

2.2.3.3. Ventajas del método de Soxhlet

- La recirculación permite que la muestra este en contacto directamente con el disolvente.
- Se somete a calentamiento al disolvente hasta que este llegue a su punto de ebullición lo cual favorece la solubilidad.
- El proceso empleado es simple. [12]

2.2.4. Solventes

Los disolventes son sustancias orgánicas de varios orígenes y naturaleza, se los encuentra en los diversos estados de la materia ya sea líquido, sólidos o gases, se recalca que si el solvente se considera que se encuentra en condiciones normales de temperatura y presión se presenta en estado líquido. [13] El más utilizado en los procesos industriales es el etanol o también llamado alcohol común o de farmacia compuesto de 95,6% de etanol y 4,4% de agua.

2.2.4.1. Características de los solventes

Se respeta la regla de polaridad de las moléculas, donde el disolvente polar disuelve la molécula polar, y el disolvente apolar disuelve la molécula apolar. Cuando una cierta sustancia (orgánica, por ejemplo) posee dos grupos distintos que difieren en las características de polaridad, se observa cuál prevalece, y el solvente será semejante a este. Pero la interacción entre el solvente y el soluto está relacionada con la diferencia (o ausencia) de la disputa entre las partes polares y apolares.

Muchos disolventes utilizados en la industria son sustancias altamente volátiles, es decir, que se evaporan fácilmente, por lo que puede ser inhalada

(introducida en el organismo a través de la aspiración, por la nariz o la boca).

Otra característica es que son inflamables, es decir, se queman fácilmente.

2.2.4.2. Tipos de solventes

Existe una variedad de solventes que se emplea de acuerdo a su efectividad, y/o máxima capacidad de recuperación.

2.2.4.2.1. Aromáticos

Tolueno: conocido también como metilbenceno ya que tiene una similitud en el olor al benceno además de ser incoloro e inflamable. Su uso principal es en la gasolina ya que permite elevar su octanaje y como disolvente para el proceso de elaboración de pinturas, resinas perfumes, etc. [14]

Xileno: contiene 3 isómeros que son orto, meta y para; es un líquido incoloro e inflamable. Se utiliza como diluyente o solvente para esmaltes, tintas, también la elaboración de fragancias, etc. [14]

2.2.4.2.2. Acetatos

Acetato de etilo: este solvente se obtiene por destilación; líquido incoloro e inflamable, uso principalmente es para extracciones de antibióticos y además en la elaboración de pinturas, resinas, perfumes, etc. [14]

2.2.4.2.3. Cetanos

Acetonas: se conoce como una cetona sencilla, aromático. Solvente incoloro e inflamable, sus usos principales son lubricantes, colorantes, productos farmacéuticos, aceites, etc. [14]

Metil Isobutil cetona: es un solvente líquido tóxico, soluble al agua y con un olor muy similar al de la cetona y al alcanfor.

2.2.4.2.4. Alcoholes

Los alcoholes empleados con mayor frecuencia son el metanol y etanol, el primero y más barato, más reactivos.

Metanol: líquido incoloro; soluble en las acetonas y esterés que se utilizan como solvente industrial en la fabricación de formol, colesterol vitaminas y hormonas además en la elaboración de plásticos, jabones, etc. [14]

Etanol: es meno tóxico y se considera más renovable porque se obtiene de fermentación de una fuente amilácea o sacarificable.

Isopropanol: Solvente con olor y propiedades similar al alcohol etílico, mismo que lo sustituye en la elaboración de cosméticos, permanentes, etc.

2.2.4.2.5. Gasolvente

Hexano: solvente mayor utilizado en las extracciones de aceites vegetales empleado en el método soxhlet; líquido volátil e incoloro.

Heptano: solvente que sustituye al hexano en las extracciones de aceite además de ser usado en las industrias de pintura, papelera, tintas, etc. Líquido incoloro, posee un olor característico de las gasolinas. [14]

2.2.4.3. Purificación de los solventes

Es necesario realizar una purificación a los solventes y así recuperarlos para poderlos emplear en otras extracciones, porque estos tienden a tener un

costo elevado, además, que algunos procesos son indispensables un tratamiento previo ya que nos permite obtener grandes resultados de extracción. [13]

2.2.4.3.1. Métodos convencionales de recuperación de solventes

Existen diversos métodos para la recuperación y purificación de un solvente, entre los métodos más empleados tanto a escala de laboratorio e industrial se encuentran la filtración y destilación, dos operaciones unitarias básicas con altos porcentajes de rendimiento en cuanto a recuperación de sustancias.

2.2.4.3.1.1. Destilación

Una de las técnicas más utilizadas es la destilación para una buena purificación del disolvente, que se basa en los puntos de ebullición de las sustancias. Depende de las propiedades que posee el solvente y lo que se desea purificar en la elección de la destilación ya que existen diferentes tipos. [13]

2.2.4.3.1.2. Filtración

Este proceso permite la eliminación de los componentes que podría poseer dichos solventes, dependiendo de las partículas sólidas que contenga el solvente se determina el tipo de filtración a utilizar ya sea de membrana o cartucho. [13]

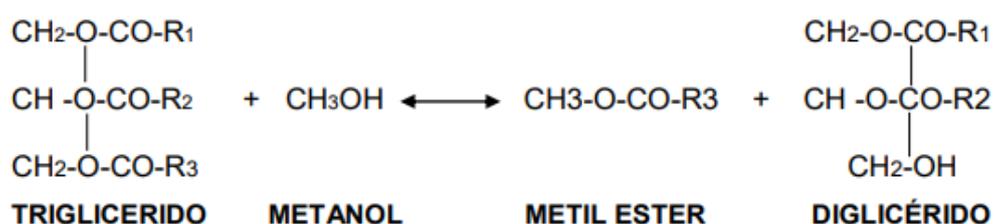
2.2.5. Reacción de Transesterificación

Alcoholisis de un aceite vegetal, se conoce como una molécula de triglicéridos que componen en mayor cantidad en un aceite que reacciona con

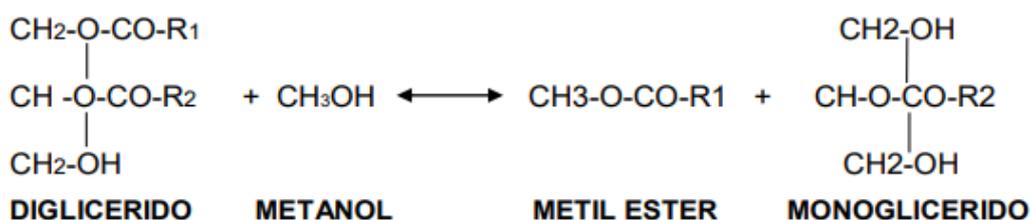
un alcohol con ayuda de un catalizador alcalino para producir una reacción y una mezcla obteniendo así ésteres metílicos de ácidos grasos y glicerol. [15]

La reacción consta de 3 etapas consecutivas y reversibles siendo así que el triglicérido se transforma en diglicérido, monoglicérido y glicerina

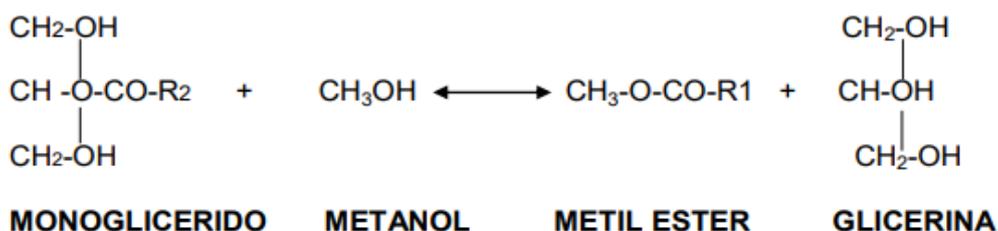
ETAPA INICIAL



ETAPA SECUNDARIA



ETAPA TERCEARIA

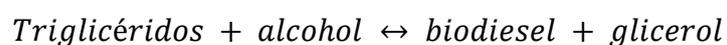


En la primer etapa es un ataque sobre el átomo de carbono carbonilo de la molécula de triglicérido sobre el anión del alcohol (ion metóxido), luego en la segundo etapa, reacciona con un alcohol (metanol) para regenerar el anión del alcohol (ion metóxido), en la etapa terciaria resulta en la formación de un éster de ácido graso y en diglicérido.

Las reacciones que se producen en una transesterificación suceden de forma consecutiva, por lo cual no se puede distinguir entre ellas. Sin embargo, lo que se realiza es una variación de sus condiciones de reacción para facilitar el rendimiento de una de los productos de la misma.

2.2.5.1. Productos de una reacción de transesterificación

Después de la transesterificación de los triglicéridos, los productos son una mezcla de esteres, glicerol, alcohol, catalizador y tri-, di- y monoglicéridos. Uno en mayor proporción que el otro, y por ende de acuerdo a las condiciones de reacción la pureza de uno de los dos productos será beneficiado. Las características de esos productos serán influenciadas en su totalidad por el origen del triglicérido (grasa) y el alcohol, razón por lo cual existen un sinnúmero de investigaciones basadas en esta reacción y sus productos.



2.2.5.1.1. Biodiesel

Se describe químicamente como una mezcla de esteres de alquilo con cadenas largas de ácidos grasos. Estas cadenas al estar oxigenadas, le dan al motor una combustión más limpia.[5] El biodiesel es un líquido de color amarillo que contiene una viscosidad similar al diésel de petróleo, no es inflamable superior comparado al diésel tradicional, además de disminuir las emisiones de gases tóxicos y otras sustancias volátiles. Presenta menor índice de cetano y mejor eficiencia con la lubricación. Estos cumplen con las normativas internacionales ASTM.

Se obtiene por la reacción de transesterificación de aceites vegetales utilizando como catalizador hidróxido de sodio o de potasio el único problema que existe en esta reacción es la formación de jabones los cuales con las condiciones de experimentación. [16]

2.2.5.1.2. Glicerina

También conocido como glicerol, es un compuesto orgánico y subproducto de la reacción, este es un alcohol de azúcar. Su fórmula química es $C_3H_5(OH)_3$. La glicerina es un componente principal de los ésteres llamados triglicéridos, a veces también conocidos simplemente como grasas o aceites. El glicerol se encuentra en todos los tipos de aceites, así como en las grasas animales o vegetales, siempre que éstas vayan asociadas a otros ácidos grasos como puede ser, por ejemplo, el oleico, o esteárico. Ecuador importa glicerina desde Perú y Colombia, pero debido a ciertas reformas gubernamentales del gobierno en turno, estas han disminuidos y los comerciantes e industrias prefieren importar desde Asia, que según ellos les resulta más económico y rentable.

2.2.5.2. Catalizadores

Existen varios tipos de catálisis para la transesterificación, pero comercialmente para la producción de biodiesel la más comúnmente utilizada es la catálisis homogénea, que se puede dividir principalmente en alcalina y ácida.

Los más usados a nivel comercial son los catalizadores homogéneos básicos, por ser los más eficientes y los que permiten operar en las condiciones

mejores, ya que si se emplearan catalizadores ácidos serían necesarias temperaturas elevadas y largos tiempos de reacción.

La reacción de transesterificación precisa de catalizadores, y estos pueden ser de varios tipos:

- Desde ácidos homogéneos (caso del ácido sulfúrico, el ácido clorhídrico o el ácido fosfórico)
- Hasta ácidos heterogéneos (zeolitas, resinas sulfónicas, etc.)
- Básicos homogéneos (hidróxido sódico o potásico)
- Básicos heterogéneos (óxidos de calcio o magnesio, entre otros)
- Incluso enzimáticos del tipo lipasas.

2.3. Marco Conceptual

2.3.1. Aceite de Moringa

Es un aceite vegetal comestible y cosmético que se extrae de las semillas del árbol de moringa (*moringa oleífera*) de origen tropical. Este aceite es de color amarillo claro, tiene propiedades utilizadas en cosmética y medicina natural. Sirve además para la producción de biodiesel como aceite lubricante para maquinaria y como componente en la industria de los cosméticos. Contiene un 80% de ácido oleico (omega 9) siendo este superior al aceite de oliva [17].

2.3.2. Método de Soxhlet

Método más antiguo de extracción sólido-líquido, el cual hasta en la actualidad es la técnica más utilizada por la agencia de protección medioambiental, el cual consiste en colocar el material de interés previamente pesado en el equipo el cual está conectado en un balón y un refrigerante con alguna de un disolvente, la diferencia de las otras técnicas es que saben ser

muchas horas de extracción pero se concentra los extractos orgánicos de interés. [18]

2.3.3. Equipo soxhlet

Franz Ritter Von propuso en el año de 1879 un método para determinar las grasas en alimentos, dicho método ha permanecido dentro de 14 décadas después. Tiene como objetivo recircular el disolvente condensado, arrastrando consigo el aceite de la materia prima.

2.3.4. Transesterificación

Consiste en la reacción de un triéster de glicérido (triglicérido) En el transcurso de la reacción se producen tres reacciones intermedias obteniendo como producto el biodiesel y como subproducto glicerina aclarando que actúa con catalizador que podría ser hidróxido de sodio. [19]

2.3.5. Ésteres metílicos de ácidos grasos

Una mezcla de diferentes ésteres metílicos se lo denomina biodiesel, estos poseen características diferentes dependiendo del tipo de aceite utilizado, dicho éster es una alternativa renovable del combustible, ya que posee ciertas similitudes físicas a las del diésel convencional, sin embargo, los ME (metil ésteres) son biodegradables y no son tóxicos. Los ésteres metílicos grasos se obtienen a partir de ácidos grasos donde el grupo sustituyente es un radical metilo [19]

2.3.6. Biodiesel

Es un combustible orgánico que está compuesta por una cadena de monoalquilésteres de ácidos grasos los cuales son grasas y aceites vegetal o

animal, el objetivo es sustituir al diésel convencional aportando así al medio ambiente. [20]

2.3.7. Poder Calorífico

Es una de las principales características del biodiesel la cual es la cantidad de energía que libera una combustión completa en unidad de masa o de volumen que lleva a formación de CO₂ Y H₂O además de depender de las características químicas del combustible. [21]; [22]

2.3.8. Destilación

Proceso simple que tiene como función calentar un líquido hasta su temperatura de ebullición además de condensar los vapores que se formaron y recolectar todo el líquido destilado. [23]

2.4. Marco Contextual

El uso de combustible fósiles abarca muchos inconvenientes como lo es agotamiento del mismo además de la protección ambiental ya que en el mundo para saciar necesidades industriales y domésticas se utiliza dichos combustibles que producen contaminantes físicos y químicos. En el 2004 en el Ecuador, la fuente de energía fue de 83% del petróleo, dicho inconvenientes permitió el desarrolló programas ambientales con el fin de ya no depender de productos derivados del petróleo y desarrollar alternativas de combustibles con productos agrícolas. Según estudios del programa en el año del 2008 el biodiésel se proyecta 3.220 barriles, aunque no es rentable producir en el país y los proyectos se afectan al no existir una legislación sobre los biocombustibles.

El presente proyecto de titulación se direcciona a la obtención de biodiesel a partir de moringa en base a la extracción del aceite a través del método de soxhlet sólido-líquido y posteriormente someterlo a la transesterificación con las condiciones normales de la reacción, donde intervendrá una serie de procesos, análisis teóricos y prácticos, para así obtener los resultados deseados; se desarrollará realizando varias pruebas experimentales dentro las instalaciones del laboratorio de la Facultad de Ingeniería Química.

2.5. Marco Legal

En el país la producción del biodiésel es muy restringida ya que solo existe una planta productora, la cual comenzó a operar en el 2005, además de no tener la capacidad de competir con el diésel ya que posee un fuerte subsidio. Ecuador posee dos programas ERGAL con el fin de ser independiente de la energía fósil en las islas Galápagos y el otro es de ecopaís que desee contemplar la utilización de aceites vegetales para la producción de biodiesel. (Pedro Rosero Álvarez Zamorano, 2010)

Las asociaciones y fundaciones nacionales deberían exigir al estado que existan legislaciones sobre el biodiésel y así ejercer mayor presión para que se lleve a cabo los planes del biocombustible.

CAPÍTULO III

3.1. Metodología de diseño

3.1.1. Enfoque de la Investigación

Enfoque Cuantitativo ya que recolecta y analiza datos para probar Hipótesis que han sido formuladas previamente, enfatiza el análisis de partes o componentes del fenómeno en observación, confía en mediciones, uso de la estadística para hacer inferencias, requiere de precisión y exactitud. Enfoque Cualitativo, ya que obtiene información que permitirá conocer el fenómeno en su totalidad antes de adelantarse a formular Hipótesis.

3.1.2. Modalidad de la Investigación

3.1.2.1. Investigación Documental.

Como una variante de la investigación científica, cuyo objetivo fundamental es el análisis de diferentes, utiliza técnicas muy precisas, de la documentación existente, que directa o indirectamente, aporte la información documental como parte esencial de un proceso de investigación científica, constituyéndose en una estrategia donde se observa y reflexiona sistemáticamente sobre realidades (teóricas o no) usando para ello diferentes tipos de documentos. Indaga, interpreta, presenta datos e informaciones sobre un tema determinado de cualquier ciencia; teniendo como finalidad obtener resultados que pudiesen ser base para el desarrollo de la creación científica.

3.1.2.2. Investigación Experimental.

Es la investigación en la que se obtiene la información por medio de la observación de los hechos, y que se encuentra dirigida a modificar la realidad

con el propósito de estudiarla en circunstancias en las que normalmente no se encuentran, con el fin de describir y analizar lo que ocurriría en determinadas condiciones.

3.1.2.3. Investigación Descriptiva

Mediante este tipo de investigación, que utiliza el método de análisis, se logra caracterizar un objeto de estudio o una situación concreta, señalar sus características y propiedades. Combinada con ciertos criterios de clasificación sirve para ordenar, agrupar o sistematizar los objetos involucrados en el trabajo indagatorio.

3.1.3. Tipo de Investigación de investigación

3.1.3.1. Investigación Explicativa

Se encarga de buscar el porqué de los hechos mediante el establecimiento de relaciones causa-efecto. En este sentido, los estudios explicativos pueden ocuparse tanto de la determinación de las causas, como de los efectos (investigación experimental), mediante la prueba de hipótesis.

3.2. Desarrollo Experimental

El trabajo de investigación se realizó en los laboratorios de la Facultad de Ingeniería Química de la Universidad de Guayaquil, se basa en la extracción del aceite de las semillas de Moringa, mediante el método de soxhlet, para luego, proceder a la reacción de transesterificación con metanol, realizando múltiples pruebas físico-químicas, que tiene como finalidad determinar el diseño experimental en base a las condiciones óptimas de reacción, lo cual lleva a cabo

la obtención del éster etílico (biodiesel), para posteriormente caracterizarlo y compararlo en base a la normativa ASTM para biodiesel 6751.

3.3. Diseño del Experimento

Se lleva a cabo un diseño, en el cual se toman en cuenta varios parámetros debido las características necesarias para una reacción de transesterificación; estas son pH, temperatura, cantidad de NaOH, mismo que funciona como catalizador y variación de la relación molar de aceite y metanol, como reactivos primarios de la reacción dada.

Tabla 1. Variables de entrada de reactivos – Transesterificación

<i>Factor</i>	<i>Mínimo</i>	<i>Máximo</i>
<i>Relación Molar</i>	6:1	9:1
<i>% Metanol / Aceite</i>	40%	-
<i>% NaOH / Metanol</i>	-	1%
<i>% NaOH / Aceite</i>	-	0.4%

Tabla 2. Variables de entrada en el proceso de neutralización ácida.

<i>Factores</i>	<i>Mínimo</i>	<i>Máximo</i>
<i>Agua (% volumen)</i>	25%	40%
<i>Ácido Fosfórico</i>	2 mL	5 mL
<i>pH</i>	8	12

Tabla 3. Condiciones en el proceso de purificación de Biodiesel.

<i>Factores</i>	<i>Mínimo</i>	<i>Máximo</i>
<i>Temperatura</i>	50 °C	100 °C
<i>pH</i>	5	7

3.4. Materiales y Equipos

Todos los materiales se describen conforme las etapas del desarrollo experimental:

- Extracción de aceite, y recuperación del solvente.

<i>Materiales</i>	<i>Sustancias y Reactivos</i>
Balanza Analítica Sarthoryus Genesis 24T01	Semilla de Moringa Oleífera
Equipo de Soxhlet	Agua destilada
Estufa	Hexano GR.
Balón 1000 mL	
Pipeta Graduada 10 mL, 25 mL	
Papel filtro Whatman N1	
Algodón	
Mortero	
Espátula	

- Reacción de Transesterificación

<i>Equipos</i>	<i>Sustancias y Reactivos</i>
Manta térmica 500 °C	Aceite de semilla de Moringa Oleífera
Agitador magnético (700 rpm)	Metanol GR
Termómetro 200° C ± 10 °C	Hidróxido de Sodio NaOH

- Purificación del Biodiesel

<i>Etapas de Lavado</i>	<i>Etapas de Secado</i>
Embudo de decantación	Calentador
Potenciómetro	Termómetro 200° C ± 10 °C
Termómetro 200° C ± 10 °C	Balón de fondo plano

3.5. Procedimiento Experimental

3.5.1. Preparación de la Muestra

Se extrae la semilla de moringa para luego, someterse a secar la dicha muestra en la estufa a una temperatura de 60 °C. Se muele en mortero la semilla con cuidado que no agarre humedad y se la procede a pesar con ayuda de una balanza además de medir en una probeta el solvente que en esta ocasión es el hexano.

3.5.2. Montaje del equipo de Soxhlet

El montaje del equipo de Soxhlet se realiza al momento que se lo va utilizar para la extracción de aceite, con los siguientes parámetros de operación para el Hexano.

Tabla. 4. Parámetros de Equipos Soxhlet

Parámetros	Unidad
Temperatura	70° C.
Tiempo	2 a 3 h.
Volumen	250 ml
Muestra	100g semilla

3.5.3. Procedimiento del equipo Soxhlet

Amar el equipo correspondiente, colocar un pedazo de algodón dentro del equipo y verter lentamente primero las semillas y luego el solvente (hexano), en la parte inferior colocar el balón y en la parte superior el condensador.

Encender el equipo y graduar la temperatura, tener cuidado con la recirculación.

Dicha extracción se la realiza en el transcurso de 2 o 3 horas, por lo cual cada cierto periodo de tiempo (15 min) se debe observar la temperatura y la potencia del equipo; además de la tonalidad del extracto, ya que este servirá como indicador de la cantidad de aceite obtenido, mismo que debe ser un amarillo intenso.

Tabla 5. Composición del Aceite de Moringa Oleífera

Ácidos Grasos	Plain	Supergenious	Nicaragua
<i>C 14:0 (mirístico)</i>	0,11	0,07	0,08
<i>C 16:0 (palmítico)</i>	7,66	5,00	5,31
<i>C16:1 (palmitoleico)</i>	1,56	1,12	1,14
<i>C18:0 (esteárico)</i>	7,06	4,60	4,68
<i>C18:1 (oleico)</i>	65,38	64,30	65,14
<i>C18:2 (linoleico)</i>	2,12	4,91	4,73
<i>C18:3 (linolénico)</i>	0,47	0,58	0,49
<i>C20:0 (araquídico)</i>	4,53	3,21	3,09
<i>C20:1 (gondoico)</i>	3,25	2,24	2,19
<i>C22:0 (behénico)</i>	-	6,31	6,06
<i>C24:0 (lignocérico)</i>	0,89	0,99	0,91

Fuente: [24]

3.5.4. Recuperación de Solvente

En la recuperación de solvente se usa el equipo Rotavapor *Heldolph Type Heizbad Hei Vap No.517-61000-01-0* con las siguientes condiciones de operación.

Tabla. 6. Condiciones de Operación

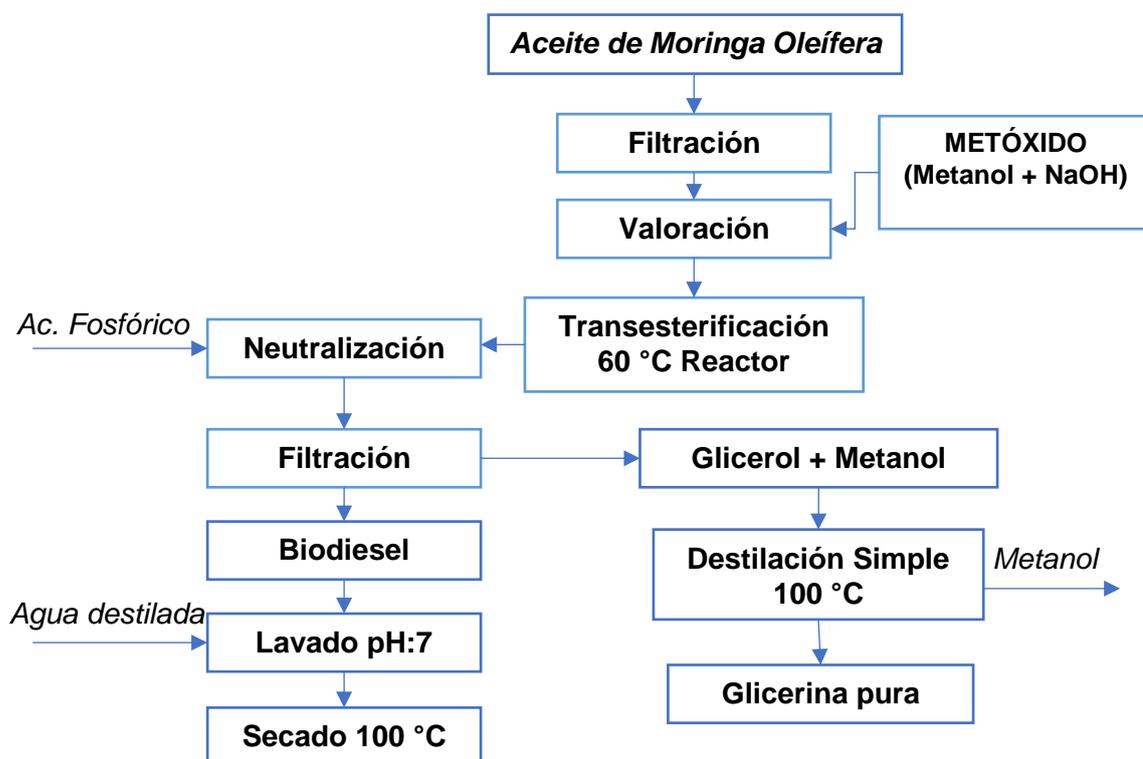
Parámetros	Unidad
Temperatura	70° C.
Tiempo	30 – 45 min
Tiempo de Enfriamiento	24 hrs

3.5.5. Procedimiento de Rotavapor

Para la recuperación del solvente (Hexano) se utiliza un rotavapor, en el cual se evapora el solvente quedando en el balón el aceite, sometidos a una temperatura aproximada de 70°C por 30 a 45 minutos a baño de glicerina. Se deja enfriar el aceite obtenido y pesar en la balanza luego procedemos a dejarlo en la estufa por 24h para eliminar cualquier exceso de hexano que no pudo ser removido y se lo vuelve a pesar para proceder a realizar los cálculos correspondientes.

3.6. Obtención del Biodiesel

Gráfico 1. Diagrama de proceso para la obtención de biodiesel



3.6.1. Acondicionamiento del aceite

3.6.1.1. Secado Del Aceite

Calentar el aceite en una hornilla en un vaso de precipitación de 500 ml a una temperatura de 110 °C y mantener durante 20 minutos, dejar de calentar y esperar a que se enfríe. Para el secado se tiene de referencia la temperatura de ebullición del solvente que se empleó para la extracción.

3.6.1.2. Preparación Del Metóxido

- Se tritura el NaOH puro
- Se mezcla el NaOH con el Metanol (Metóxido)
- Se agita hasta que se encuentre completamente disuelto

3.6.2. Reacción de Transesterificación.

Para que la reacción química se produzca sin problemas, debe calentarse el aceite hasta los 40 - 50° C. Durante la transesterificación los ácidos grasos se separan de la glicerina, y el metanol se une a ellos formando metilésteres. El hidróxido de sodio estabiliza la glicerina.

Reacción química: *Aceite Obtenido* + $6CH_3O$ → *ME* + *GLICERINA*

3.6.2.1. Experimentación de la reacción de transesterificación

La experimentación se basa en el diseño de experimento planteado junto con la investigación en la cual se toman referencias datos experimentales de investigaciones relacionadas.

Tabla 7. Variables de entrada en la reacción de Transesterificación.

<i>Ensayo</i>	<i>Reactivos</i>		
	Aceite MO (ml)	Metanol (ml)	Hidróxido de Sodio (g)
1	100	40	0.4
2	150	40	0.4
3	200	80	0.4
4	250	100	0.8
5	300	140	1
6	350	140	1.4

Una vez determinadas las cantidades para la realización de la transesterificación el único parámetro que varió fue el de la temperatura para obtener las muestras, estas temperaturas fueron 50 °C y 70 °C respectivamente para así determinar a qué temperatura es donde hubo una mayor conversión de metil ésteres.

Tabla 8. Condiciones variables en la reacción de Transesterificación.

<i>Ensayo</i>	<i>Parámetros</i>		
	Temperatura (°C)	Agitación (rpm)	Tiempo (min)
1A	50 – 60	250	60
1B	60 - 70	500	60
2A	50 – 60	250	60
2B	60 - 70	500	60
3A	50 – 60	250	60
3B	60 - 70	500	60
4A	50 – 60	250	60
4B	60 - 70	500	60
5A	50 – 60	250	60

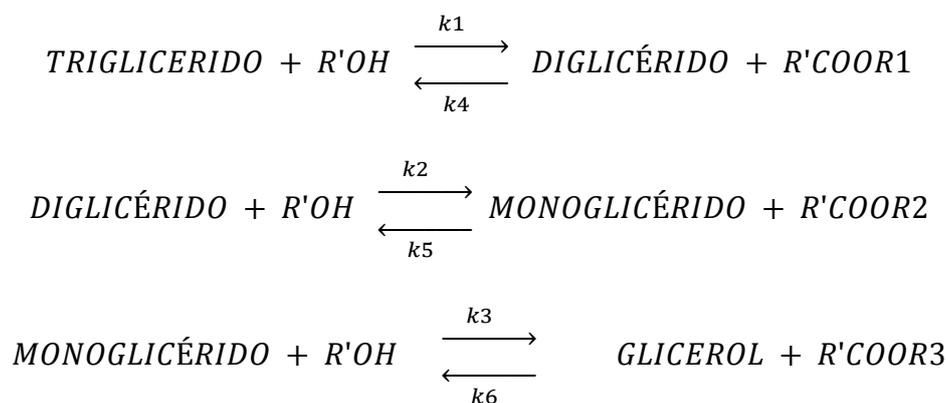
5B	60 - 70	500	60
6A	50 – 60	250	60
6B	60 - 70	500	60

La velocidad de agitación en los experimentos realizados si tuvo que ser modificada, puesto que esta influye directamente en la obtención del producto. Para una temperatura de 50 – 60 °C, se aplicó una agitación media de 250 rpm. Para una temperatura de 60 - 70 °C, se aplicó una agitación rápida de 500 - 600 rpm. El tiempo de la reacción fue de aproximadamente 60 (min)

3.6.2.2. Cinética de la reacción

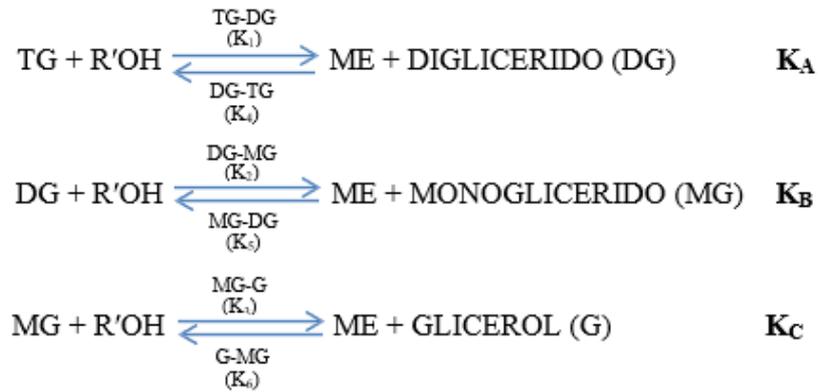
La transesterificación consiste de un número de reacciones reversibles consecutivas. El triglicérido es convertido paso a paso a diglicérido, monoglicérido y finalmente a glicerol.

Esquema de reacción.



Un mol de éster es liberado a cada paso. Las reacciones son reversibles, aunque el equilibrio se desplaza hacia la producción de ésteres de ácidos grasos y glicerol.

Reacciones principales.



K_A , K_B Y K_C son las constantes de equilibrio de las reacciones principales.

Dónde:

$$K_A = \frac{k_4}{k_1}; K_B = \frac{k_5}{k_2}; K_C = \frac{k_6}{k_3}$$

De acuerdo con el esquema anterior, la ecuación de velocidad de reacción para la aparición de metil - esteres (biodiesel) es la siguiente:

$$\begin{aligned}
 r_{\text{Biodiesel}} = & k_1[(\text{TG})(\text{R}'\text{OH}) - K_A(\text{ME})(\text{DG})] + k_2[(\text{DG})(\text{R}'\text{OH}) \\
 & - K_B(\text{ME})(\text{MG})] + k_3[(\text{MG})(\text{R}'\text{OH}) - K_C(\text{ME})(\text{G})]
 \end{aligned}$$

O también, podemos deducir las ecuaciones para cada uno de los componentes presentes durante el proceso de la transesterificación que son las siguientes:

$$\frac{dC_{\text{TG}}}{dt} = -k_1 C_{\text{TG}} C_A + k_2 C_{\text{DG}} C_{\text{ME}}$$

Triglicérido

$$\frac{dC_{\text{DG}}}{dt} = -k_3 C_{\text{DG}} C_A + k_4 C_{\text{MG}} C_{\text{ME}} + k_1 C_{\text{TG}} C_A - k_2 C_{\text{DG}} C_{\text{ME}}$$

Diglicérido

$$\frac{dC_{\text{MG}}}{dt} = -k_5 C_{\text{MG}} C_A + k_6 C_{\text{G}} C_{\text{ME}} + k_3 C_{\text{DG}} C_A - k_4 C_{\text{MG}} C_{\text{ME}}$$

Monoglicérido

$$\frac{dC_G}{dt} = k_5 C_{MG} C_A - k_6 C_G C_{ME}$$

Glicerol

$$\frac{dC_{BD}}{dt} = k_1 C_{TG} C_A - k_2 C_{DG} C_{ME} + k_3 C_{DG} C_A - k_4 C_{MG} C_{ME} + k_5 C_{MG} C_A - k_6 C_G C_{ME}$$

Biodiesel

3.7. Neutralización

Se realizó una neutralización a la muestra en el proceso de lavado para así evitar el avance de trazas de glicerol, jabón, metanol, entre otros. Al término del calentamiento se procedió a trasvasar la muestra directamente al decantador y se deja reposar hasta obtener dos fases totalmente definidas, biodiesel y la glicerina respectivamente.

- En la parte superior se puede apreciar un compuesto de color amarillo claro y de menor densidad, que es el éter metílico (biodiesel), nuestro producto de interés.
- En la parte inferior de color café claro y más denso contiene compuestos como glicerina formados con trazas de ácidos grasos, gliceroles, terpenos y carotenos estos serán eliminados mediante procesos de purificación.

Consiguientemente, se midió el pH del biodiesel y se obtuvo un pH de 8 (Básico); al obtener estos resultados, se realiza la neutralización con ácido fosfórico al 85% para así evitar el progreso de las trazas y disminuir el pH hasta llegar neutro. Para cada ensayo se utiliza una cantidad diferente de ácido, y se mide el pH de la muestra hasta que se la misma se neutralice.

Tabla 9. Cantidades de la neutralización del biodiesel

<i>Ensayo</i>	<i>Neutralizador</i>	<i>Cantidad de gotas</i>	<i>Cantidad en ml</i>	<i>pH</i>
1A	H ₂ PO ₄	5	0.25	6
1B	H ₂ PO ₄	5	0.25	6
2A	H ₂ PO ₄	2	0.10	6
2B	H ₂ PO ₄	2	0.05	7
3A	H ₂ PO ₄	1	0.05	7
3B	H ₂ PO ₄	1	0.05	7
4A	H ₂ PO ₄	3	0.15	7
4B	H ₂ PO ₄	2	0.10	7
5A	H ₂ PO ₄	3	0.15	5
5B	H ₂ PO ₄	3	0.15	6
6A	H ₂ PO ₄	1	0.05	7
6B	H ₂ PO ₄	1	0.05	7

3.8. Purificación del biodiesel

Se debe realizar la purificación del biodiesel para eliminar posibles residuos de glicerina, metanol y catalizador que no pudieron ser removidos durante la decantación. Para que los metil ésteres alcancen los parámetros aceptables de

Luego se realizan varios lavados con agua destilada a temperatura ambiente, la cantidad de agua utilizada es la mitad en proporción de lo que se obtiene del biodiesel. Esto se lo realiza hasta que, de un pH de 7, la muestra se decanta y se desecha la capa inferior que es el agua con los restos catalizador.

Inmediatamente al momento de terminar el lavado se procede a calentar la muestra a 110 °C para eliminar los residuos de agua y metanol. Al final de este procedimiento se puede apreciar que el biodiesel está más claro y limpio.

3.8.1. Lavado

El lavado se lo realiza con agua destilada a temperatura ambiente para eliminar las trazas de impurezas en el biodiesel, además, para asegurar la neutralización, se usó ácido fosfórico (H_2PO_3) al 85% el cual nos va a permitir o garantizar la neutralización del biocombustible. La separación del exceso de agua en el biodiesel se la realiza mediante decantación.

Se realizan varios lavados con agua destilada a temperatura ambiente, la cantidad de agua utilizada es la mitad en proporción de lo que se obtiene del biodiesel; la muestra se decanta y se desecha la capa inferior que es el agua con los restos de catalizador.

Tabla 10. Resultados de la etapa de purificación ensayo 1

<i>Ensayo</i>	<i>(Biodiesel + Otros Componentes)</i>	<i>Numero de Lavados</i>	<i>Agua Destilada</i>	<i>pH resultante</i>	<i>Fases presentadas</i>
1A	120 ml	1	50 ml	7	1
1B	120 ml	1	50 ml	7	1

Tabla 11. Resultados de la etapa de purificación ensayo 2

<i>Ensayo</i>	<i>(Biodiesel + Otros Componentes)</i>	<i>Numero de Lavados</i>	<i>Agua Destilada</i>	<i>pH resultante</i>	<i>Fases presentadas</i>
2A	170 ml	4	50 ml	7	1
2B	170 ml	4	50 ml	7	1

Tabla 12. Resultados de la etapa de purificación ensayo 3

<i>Ensayo</i>	<i>(Biodiesel + Otros Componentes)</i>	<i>Numero de Lavados</i>	<i>Agua Destilada</i>	<i>pH resultante</i>	<i>Fases presentadas</i>
3A	215 ml	4	80 ml	7	2
3B	215 ml	5	80 ml	6	2

Tabla 13. Resultados de la etapa de purificación ensayo 4

<i>Ensayo</i>	<i>(Biodiesel + Otros Componentes)</i>	<i>Numero de Lavados</i>	<i>Agua Destilada</i>	<i>pH resultante</i>	<i>Fases presentadas</i>
4A	270 ml	5	100 ml	6	2
4B	270 ml	4	100 ml	6	2

Tabla 14. Resultados de la etapa de purificación ensayo 5

<i>Ensayo</i>	<i>(Biodiesel + Otros Componentes)</i>	<i>Numero de Lavados</i>	<i>Agua Destilada</i>	<i>pH resultante</i>	<i>Fases presentadas</i>
5A	325 ml	4	150 ml	6	2
5B	325 ml	3	150 ml	6	2

Tabla 15. Resultados de la etapa de purificación ensayo 6

<i>Ensayo</i>	<i>(Biodiesel + Otros Componentes)</i>	<i>Numero de Lavados</i>	<i>Agua Destilada</i>	<i>pH resultante</i>	<i>Fases presentadas</i>
6A	380 ml	3	150 ml	7	2
6B	380 ml	3	150 ml	7	2

Para la siguiente etapa de secado solo se tomarán en cuenta los ensayos que presentaron dos fases en el momento del lavado, debido a que se toma como indicador en base a la reacción. Es decir, los que presentaron una sola fase indican que existían una cantidad excesiva de trazas de gliceroles, por lo cual al agregar el agua de lavado se formaba una reacción de saponificación de forma instantánea, dando así como resultado un líquido espumoso que no era el producto deseado, mientras que los que presentaban dos fases, la cantidad de trazas de gliceroles era menor, por lo cual al agregar el agua de lavado se producían pequeñas partículas sólidas que se separaban mediante decantación, a estos ensayos se los denomina exitosos.

3.8.2. Secado

El proceso de secado se lo realiza por calentamiento entre un rango de 100 a 110 °C con la finalidad de eliminar los restos de agua que quedaron en el biodiesel después del lavado, esto se efectúa en un tiempo de 15 min, posterior se lo deja enfriar, para así obtener un producto más limpio.

Tabla 16. Resultados de la etapa de secado ensayo 3

<i>Ensayo</i>	<i>Biodiesel*</i> <i>Volumen Inicial</i>	<i>Biodiesel</i> <i>Volumen Final</i>	<i>Tiempo</i>
3A	185 ml	158 ml	15 min
3B	185 ml	160 ml	15 min

*Cantidad obtenido de la etapa de lavado

Tabla 17. Resultados de la etapa de secado ensayo 4

<i>Ensayo</i>	<i>Biodiesel*</i> <i>Volumen Inicial</i>	<i>Biodiesel</i> <i>Volumen Final</i>	<i>Tiempo</i>
4A	220 ml	196 ml	15 min
4B	220 ml	198 ml	15 min

*Cantidad obtenido de la etapa de lavado

Tabla 18. Resultados de la etapa de secado ensayo 5

<i>Ensayo</i>	<i>Biodiesel*</i> <i>Volumen Inicial</i>	<i>Biodiesel</i> <i>Volumen Final</i>	<i>Tiempo</i>
5A	270 ml	254 ml	15 min
5B	270 ml	250 ml	15 min

*Cantidad obtenido de la etapa de lavado

Tabla 19. Resultados de la etapa de secado ensayo 6

<i>Ensayo</i>	<i>Biodiesel*</i> <i>Volumen Inicial</i>	<i>Biodiesel</i> <i>Volumen Final</i>	<i>Tiempo</i>
6A	320 ml	302 ml	15 min
6B	320 ml	310 ml	15 min

*Cantidad obtenido de la etapa de lavado

3.9. Caracterización del Biodiesel.

La caracterización del producto obtenido se realizará de acuerdo a las especificaciones internacionales,

Tabla 20. Pruebas físico químicas para caracterizar Biodiesel

Características	Unidad	Método ASTM	Aplicabilidad
Punto Inflamación	°C	D-93	Si
Grados API		D-287	Si
Densidad 15 °C		D-287	Si
Destilación 90%	°C	D-86	Si
Viscosidad Cinemática 40 °C	mm ² /s	D-287	Si
Cenizas Sulfatadas	%p	D-874	No
Poder Calorífico	Btu/Lb	D-240	No
Contenido de Agua	%v	D-95	No
Índice de Cetano		D-976	No
Residuo Carbonoso	%p	D-189	No

Parte de las pruebas de caracterización se realizarán con los equipos disponibles dentro del laboratorio de petróleo de la Facultad de Ingeniería Química de la Universidad de Guayaquil, los demás se realizarán con la ayuda de un ente externo, mismo que garantiza los resultados obtenidos, y posterior se realiza la comparación de los mismos.

3.9.1. Prueba de crepitación

Esta prueba se realiza con el fin de verificar el contenido de agua en una muestra de combustible, o tal es el caso de un biocombustible, se realiza de forma visual y auditiva.

3.9.1.1. Técnica y resultados de crepitación

En una plancha caliente colocar una gota de agua, para comprobar el calentamiento de la plancha, posterior se coloca una gota de la muestra a analizar (Biodiesel) en la plancha caliente. Si el biodiesel contiene agua en una proporción mayor de 0.1% crepitará (sonido burbujeante).

Tabla 21. Resultados de la prueba de crepitación

<i>Ensayo</i>	<i>Muestra (ml)</i>	<i>Prueba visual</i>	<i>Prueba auditiva</i>
3A	0.05 ml	-	-
3B	0.05 ml	-	-
4A	0.05 ml	-	-
4B	0.05 ml	-	+
5A	0.05 ml	-	-
5B	0.05 ml	-	+
6A	0.05 ml	-	-
6B	0.05 ml	-	-

Los resultados datan como positivos (presencia de agua) y negativos (ausencia) por lo cual se considera que pese a la etapa de secado se acuerdo a los establecido en el diseño experimental, tanto el ensayo 4B y 5B aún tenían un contenido de agua mayor al 0.1%.

3.9.2. Prueba de Densidad Absoluta

Mayores densidades indican mayor energía térmica y una economía de combustible mejor, pero de igual manera la mayor densidad del biodiesel puede

causar problemas en los actuales sistemas de inyección de los motores y, a bajas temperaturas, plantea problemas de solidificación y congelación.

3.9.2.1. Técnica y resultados de densidad

Para realizar las pruebas de densidad se requieren un mínimo de 500 ml, para hacer uso del hidrómetro, por lo cual se eligen cuatros ensayos, estos se eligen bajo el criterio de efectividad ante las pruebas ejecutadas con anterioridad.

Una vez hecha las repeticiones de los ensayos requeridas, para obtener el volumen necesario para la prueba, se procede con la ejecución de la misma, de acuerdo al siguiente procedimiento.

- Verter la muestra (Biodiesel) en un vaso de precipitación para evitar derrames.
- Trasvasar lentamente la muestra dentro de la probeta limpia de manera inclinada. Sin agitar ni salpicar, de modo que se evite la formación de burbujas de aire.
- Remover con papel secante cualquier burbuja de aire que se forme en la superficie de la muestra antes de introducir el hidrómetro.
- Colocar la probeta que contiene la muestra en un lugar libre de corriente de aire.
- Durante el periodo de prueba la temperatura de la muestra no debe variar más de 5°F.
- Sumergir el hidrómetro suavemente dentro de la muestra, y cuando se haya asentado, sumerja, unas dos divisiones de la escala dentro del líquido y después suéltese.

- Mantener seco el resto del vástago, ya que el líquido innecesario sobre el vástago cambia el peso efectivo del instrumento y afecta la lectura.
- Cuando, el hidrómetro ha quedado inmóvil, flotando libremente y la temperatura de la muestra, es constante dentro de 0,2° C (0.5°F), lea el hidrómetro, hasta la más próxima media división de la escala.
- Determinar este punto colocando el ojo ligeramente por debajo del nivel del líquido e incrementar lentamente hasta la superficie, primeramente, observará una elipse distorsionada. La lectura correcta es el punto de la escala del hidrómetro en la cual la superficie del líquido corta el escalda.
- Para efectuar lecturas en líquidos no transparentes, observar el punto en la escala del hidrómetro en la cual la muestra tiene su punto más alto sobre su superficie principal, colocando el ojo ligeramente sobre la superficie plana del líquido.
- Inmediatamente después de observar la escala del hidrómetro, agite cuidadosamente con el termómetro la muestra y observe la temperatura.
- Mantenga el bulbo del termómetro completamente sumergido.
- Corregir las lecturas del hidrómetro a 15.56 (60°F) utilizando las Tablas de la Guía D1250.

Tabla 22. Resultados de las pruebas de densidad.

<i>Ensayo</i>	<i>Muestra (ml)</i>	<i>API leído</i>	<i>API corregido</i>	<i>SG</i>
4A	500 ml	32.1	30.9	0.8713
5A	500 ml	32.7	31.5	0.8680
6A	500 ml	32.4	31.2	0.8696
6B	500 ml	32.5	31.4	0.8686

3.9.3. Prueba de viscosidad cinemática

Para la prueba de viscosidad se utilizan los resultados de los ensayos mencionados en el apartado anterior, por razones de rendimiento.

Preparación del equipo.

1. Limpiar completamente el viscosímetro usando un disolvente apropiado miscible con la muestra y luego un disolvente completamente volátil.
2. Secar el viscosímetro pasando una corriente de aire seco y filtrado durante dos minutos, hasta que cualquier traza de disolvente haya desaparecido.
3. Limpiar periódicamente el viscosímetro con ácido crómico para remover los depósitos orgánicos, enjuagando completamente con agua destilada y acetona y secarlo con aire limpio y seco.
4. Los depósitos inorgánicos pueden removerse por tratamiento con ácido clorhídrico antes del ácido de limpieza, especialmente si las sales de bario se encuentran presentes.

Ejecución del ensayo:

1. Mantener el baño a la temperatura de ensayo.
2. Colocar el termómetro en el baño.
3. Seleccionar un viscosímetro limpio, seco y calibrado, que tenga un rango que cubra la viscosidad estimada.
4. Un capilar amplio para un líquido muy viscoso y un capilar más angosto para un líquido fluido.

5. Filtrese una porción de la muestra de aceite a través de un tamiz de 200 mallas o de cualquier otro filtro adecuado, para eliminar partículas sólidas.
6. Cargar el viscosímetro en la forma apropiada de acuerdo con el diseño.
7. Dejar que el viscosímetro cargado permanezca en el baño un tiempo o suficientemente largo para alcanzar la temperatura.
8. Mantener en el baño a temperatura de la prueba.
9. Con la muestra fluyendo libremente, medir el tiempo requerido con aproximación de 0.2 segundos, que el menisco pase desde la primera a la segunda 68 marca registradora del tiempo.
10. Si el tiempo de flujo es menor de 200 segundos, seleccionar un viscosímetro con un capilar de diámetro más pequeño y repetir el ensayo.
11. En los viscosímetros de “flujo en reversa” realizar determinaciones duplicadas, los tiempos de flujo deben concordar dentro del 0.35% si no se obtiene esta concordancia, rechazar los resultados del ensayo.

Tabla 23. Resultado de la Viscosidad cinemática obtenida del biodiesel

<i>Ensayo</i>	<i>Muestra</i>	<i>Tiempo (s)</i>	<i>Viscosidad (mm²/s²)</i>
4A	1	350	5.383
	2	352	5.4138
5A	1	355	5.4598
	2	360	5.5367
6A	1	380	5.8444
	2	382	5.8752
6B	1	385	5.9213
	2	388	5.9674

3.9.4. Prueba de punto de inflamación (copa cerrada)

El punto de inflamación da una idea de la cantidad de compuestos volátiles o muy volátiles que puede tener un combustible. Teniendo en cuenta el punto de inflamación se puede estimar cuales van a ser las condiciones de almacenamiento de ese combustible. Según como vayan a ser las condiciones de almacenamiento, el punto de inflamación se determina en vaso abierto Cleveland o en vaso cerrado Perski-Maters. En este caso se aplicará la técnica de vaso o copa cerrada, con el producto de los ensayos usados en las pruebas anteriores, de acuerdo a la técnica el procedimiento es el siguiente.

1. Llenar la copa de ensayo con 75 mL de muestra de biodiesel y colocar en posición de ensayo sobre el bloque de calentamiento. La temperatura de la muestra a ensayar debe ser al menos 18°C ó 32°F menor que el punto de inflamación esperado.
2. Ajustar la tapa de la copa de ensayo a esta última, asegurar de que quede en la posición correcta.
3. Comprobar que el termómetro a emplear en la determinación es el adecuado para el ensayo que se va a llevar a cabo (en este caso se empleará un termómetro ASTM 9F).
4. Conectar la tapa de la copa de ensayo al rotor adecuado de acuerdo con la velocidad de agitación necesaria (Procedimiento A, 90-120 rpm).
5. Comprobar el paso de gas y ajustar el tamaño de las llamas piloto y test a un diámetro aproximado de 4 mm, comprobando mediante la aplicación manual de llama que esta no se apaga con el movimiento derivado de dicha aplicación.

6. Aplicar la calefacción necesaria para conseguir una velocidad de calentamiento de entre 5-6°C/min (9-11°F/min).
7. Comenzar a realizar aplicaciones de llama (detener la agitación para llevar a cabo la aplicación de llama) sobre la muestra a una temperatura $23 \pm 5^\circ\text{C}$ ó $41 \pm 9^\circ\text{F}$ por debajo del punto de inflamación esperado.
8. Realizar aplicaciones de llama a intervalos de temperatura de 1°C ó 2°F , el tiempo de exposición de la llama no debe ser superior a un segundo.
9. Anotar la temperatura a la que se observa la inflamación, con una precisión de $0,5^\circ\text{C}$ o bien 1°F .

Tabla 24. Resultado del punto de inflamación del biodiesel

<i>Ensayo</i>	<i>Punto de Inflamación</i>
4A	-
5A	-
6A	97 °C
6B	106 °C

Los resultados del ensayo 4A y 5A no presentaron indicios de inflamación hasta la temperatura de 200 °C por lo cual se detuvo la experimentación y se continuo con los otros dos ensayos.

CAPITULO IV

4.1. Análisis e interpretación

4.1.1. Evaluación de Extracción de Aceite

Continuando con el cumplimiento de los objetivos planteados, se evalúa el rendimiento que posee la semilla de Moringa Oleífera, con el fin de establecer si este es óptimo para su uso a gran escala.

El porcentaje se establece realizando una media de los porcentajes de aceite obtenido de los tres experimentos que tuvieron éxito en el momento de la transesterificación, ya que estos conllevan al valor real de la muestra, siendo un 26% el rendimiento total en cuanto a la extracción.

La experimentación y obtención del aceite se realizan en mayor proporción y varias veces consecutivas hasta obtener aproximadamente 4000 ml de aceite, mismos que se usaron en cada ensayo experimental planteado, y según los resultados favorables se procedía con la repetividad de un ensayo en la reacción de transesterificación.

Tabla 25. Porcentaje de Rendimiento

Ensayo	<i>Materia Prima</i>	<i>Aceite Obtenido</i>	<i>Aceite Final</i>	<i>Porcentaje de Rendimiento Final</i>
1	100 g	27g	25g	25%
2	100 g	24g	21g	21%
3	100 g	35g	32g	32%

4.1.2. Establecer las condiciones óptimas de reacción

Para establecer las condiciones óptimas de la reacción se realizan pruebas de ensayo y error, método aplicado en el desarrollo de nuevos productos. En este caso consiste en variar cada uno de los parámetros establecidos durante la experimentación, los factores más importantes son temperatura, tiempo y velocidad de agitación, todos determinados a partir del ensayo 6, según lo establecido en la tabla 8 siendo los más viables los que datan en la siguiente tabla.

Tabla 26. Condiciones óptimas de reacción

Condiciones	Valor	Unidades
<i>Temperatura</i>	60 - 70	°C
<i>Tiempo</i>	60	Minutos
<i>Agitación</i>	500 - 700	rpm
<i>Relación Molar</i>	6:1	-

4.1.2.1. Efecto de la relación molar y elección del alcohol

Una de las variables más importantes que afectan el rendimiento del éster es la relación molar de alcohol a triglicérido. La relación estequiométrica para la transesterificación requiere tres moles de alcohol y un mol de triglicérido para producir tres moles de ésteres de ácidos grasos de alquilo y un mol de glicerol. Sin embargo, la transesterificación es una reacción de equilibrio en la que se requiere un gran exceso de alcohol para conducir la reacción hacia la derecha. Para la máxima conversión del éster, se requiere como mínimo una relación de 6:1. La relación molar no tiene efecto en el valor ácido, índices de peróxido, saponificación y de yodo de los ésteres de metilo. Sin embargo, la alta relación

molar de alcohol a aceite vegetal interfiere con la separación de la glicerina porque hay un aumento de la solubilidad.

Cuando la glicerina permanece en solución, ayuda a conducir el equilibrio hacia la izquierda, bajando el rendimiento de los ésteres. La formación catalizada por una base de etilésteres es difícil en comparación con la formación de ésteres de metilo. Específicamente, la formación de una emulsión estable durante la etanólisis es un problema. El metanol y el etanol no son miscibles con los triglicéridos a temperatura ambiente, y las mezclas de reacción son usualmente agitadas mecánicamente para mejorar la transferencia de masa. Durante el curso de la reacción, usualmente se forman emulsiones. En el caso de la metanólisis, estas emulsiones rápida y fácilmente se descomponen para formar una capa inferior rica en glicerol y una capa superior rica en éster metílico. En la etanólisis, estas emulsiones son más estables y complican severamente la separación y purificación de los ésteres. Las emulsiones son causadas en parte por la formación de los intermediarios monoglicéridos y diglicéridos, los cuales tienen grupos polares hidroxilo y cadenas de hidrocarburos no polares. Estos intermediarios son agentes fuertemente activos. En el proceso de alcoholólisis, el catalizador, hidróxido de sodio se disuelve en la fase polar de alcohol, en la que los triglicéridos deben transferirse con el fin de reaccionar. Cuando la concentración de estos compuestos intermediarios alcanza un nivel crítico, se forman las emulsiones. [25]

4.1.3. Caracterización de Biodiesel obtenido.

Los resultados de caracterización son determinados por un laboratorio acreditado de la ciudad de Guayaquil, cuyos resultados se encuentran establecidos en la sección de anexos, apéndice 3, mismo que certifica la veracidad de las características evaluadas.

Tabla 27. Resultados de caracterización.

Características	Unidad	Resultado	Método ASTM
Punto Inflamación	°C	103	D-93
Grados API		27.9	D-287
Densidad 15 °C	g/mL	0.8872	D-287
Destilación 90%	°C	340	D-86
Viscosidad Cinemática 40 °C	mm ² /s	5.087	D-287
Cenizas Sulfatadas	%p	0.004	D-874
<i>Poder Calorífico</i>	Btu/Lb	18019	D-240
<i>Contenido de Agua</i>	%v	0.00	D-95
<i>Índice de Cetano</i>		45.00	D-976
<i>Residuo Carbonoso</i>	%p	0.3	D-189

Fuente: EP Ingeniería Química – Laboratorio de Petróleo

4.1.3.1. Punto de Inflamación

El resultado del punto de inflamación fue de 103 °C, valor que según las Norma ASTM para biodiésel D6751-09 se encuentra dentro del rango, el valor es aceptado en su totalidad ya que se encuentra muy cercano al mínimo registrado por parte de la norma ya mencionada, por ende esta características lo vuelve competitivo frente a otros biocombustibles, debido a que si el punto de

inflamación es más próximo al de un petrodiesel o Diesel comercial, a nivel automovilístico las modificaciones para su uso son relativamente menores.

Tabla 28. Resultados de caracterización del punto de inflamación

<i>Característica</i>	<i>Unidad</i>	<i>Resultado</i>	<i>Método</i>		<i>Especificación</i>	
			ASTM	Interno	Min	Max
Punto Inflamación	°C	103	D-93	PEE/UCC/LP/03	93	-

La determinación del punto de inflamación está relacionada con el contenido de metanol residual, pues cantidades muy pequeñas de este alcohol reducen notablemente este parámetro. El metanol puede afectar los equipos de bombeo del combustible, sellos y empaques, además de generar problemas de lubricidad. El poseer un punto de ignición por encima del establecido (93 °C) asegura que el metanol presente está por debajo del 0.2% en peso. [26]

4.1.4. Grados API y Densidad

La densidad es un parámetro esencial en lo que respecta a los combustibles; el biodiesel obtenido posee un resultado óptimo ya que encuentra dentro de los valores establecidos por la norma ASTM para biodiesel D6751-09, por lo consiguiente se realiza el cálculo de grados API, que posteriormente es analizado para su determinación y aceptación.

La densidad refleja las propiedades de los compuestos químicos (metilésteres) componentes del biodiesel, además, de proporcionar información acerca del contenido de energía del combustible, es decir, mayores densidades indican mayor energía térmica y una mejor economía de combustible. [27]

Tabla 29. Resultados de caracterización densidad

<i>Característica</i>	<i>Unidad</i>	<i>Resultado</i>	<i>Método</i>		<i>Especificación</i>	
			ASTM	Interno	Min	Max
Grados API		27.9	D-287	PEE/UCC/LP/05	-	-
Densidad 15 °C	g/ml	0.8872	D-287		0.86	0.90

4.1.5. Destilación

El resultado de punto de destilación, no posee un valor establecido de especificaciones máximos y mínimos en norma ASTM para biodiésel D6751-09 para este producto, sin embargo, el dato obtenido es aceptado y se espera pueda aplicarse como referencial en investigaciones basadas en la obtención de biodiesel a partir de materia prima vegetal.

Tabla 30. Resultados de destilación

<i>Característica</i>	<i>Unidad</i>	<i>Resultado</i>	<i>Método</i>		<i>Especificación</i>	
			ASTM	Interno	Min	Max
Destilación	°C	340	D-86	PEE/UCC/LP/01	-	-

La norma ASTM también contiene una especificación (D1160) para el comportamiento de destilación de un combustible a presión atmosférica.

Aunque está contenida en la norma de biodiésel, en la realidad no es aplicable al biodiésel ya que los ésteres grasos tienen tendencia a descomponerse antes de llegar al punto de ebullición bajo las condiciones aplicadas y por tanto se indica una presión reducida en D6751. Este método proviene de la norma astm para petrodiesel D975 (American Society for Testing and Materials, astm

Standard D6751). El límite de 360°C puede restringir materias primas, especialmente con longitudes de cadena más largas. [28]

Por lo antes expuesto y pese al límite que establece la derivada de la norma, el resultado obtenido de esta caracterización se encuentra dentro de parámetros.

4.1.6. Viscosidad Cinemática

La viscosidad es un valor que nos permite saber hasta qué punto es posible deformarse un fluido, por ende, un indicador de su vida útil en el proceso de auto movilización. El resultado es favorable pues se muestra un valor muy cercano al límite, lo que representa que el fluido es viscoso, factor esencial en los combustibles comerciales, una razón por la cual se muestra competitivo comercialmente.

Tabla 31. Resultados de viscosidad

<i>Característica</i>	<i>Unidad</i>	<i>Resultado</i>	<i>Método</i>		<i>Especificación</i>	
			ASTM	Interno	Min	Max
Viscosidad Cinemática 40 °C	mm ² /s	5.087	D-287	PEE/UCC/LP/04	1.4	6.0

4.1.7. Cenizas Sulfatadas, Contenido de Agua y Residuo Carbonoso

Los resultados de cenizas, residuos y el contenido de agua son favorables, esto se muestran dentro de los parámetros establecidos por la norma ASTM para biodiésel D6751-09, por lo consiguiente estos valores sirven como indicadores de que el proceso de obtención se dio de forma óptima.

Los residuos de carbono tienen que ver con la tendencia de una muestra a formar depósitos carbonosos esencialmente por pirolisis de la muestra.

Tabla 32. Resultados de caracterización

<i>Característica</i>	<i>Unidad</i>	<i>Resultado</i>	<i>Método</i>	<i>Especificación</i>	
			ASTM	Min	Max
Cenizas Sulfatadas	%p	0.004	D-874	-	0.02
Contenido de Agua	%v	0.00	D-95	-	0.05
Residuo Carbonoso	%p	0.3	D-189	-	0.3

4.1.8. Índice de Cetano

En el combustible diésel, el cetano es un indicativo de la capacidad o facilidad de ignición. Se puede medir en laboratorio en un motor especialmente acondicionado para ello se habla del "Número de Cetano". Como en este caso es calculado a partir de los datos de la destilación se denomina del "Índice de Cetano". Entre mayor es el cetano, mayor será la facilidad de ignición del combustible e indica mejor calidad.

Tabla 33. Resultados de caracterización

<i>Característica</i>	<i>Unidad</i>	<i>Resultado</i>	<i>Método</i>	<i>Especificación</i>	
			ASTM	Min	Max
Numero de Cetano		45	D-976	47	-

El resultado obtenido se encuentra fuera de especificaciones si nos regimos en la norma, pero como los datos obtenidos se realizaron mediante los datos de destilación, la variación del resultado entre el número e índice de cetano es aceptable en variación de ± 2 puntos, por lo cual estaría en el límite de la caracterización. [29]

4.1.9. Poder Calorífico

El valor promedio encontrado en este estudio para el poder calorífico del biodiesel a partir de aceite de semillas de moringa modificadas genéticamente fue de 18 019 Btu/Lb equivalente a 41 912.19 KJ/Kg (ver tabla 27). El límite establecido por la norma de ensayo ASTM D240 es mínimo de 39500 KJ/kg por lo cual el biocombustible producido cumple a cabalidad con esta normatividad. Este resultado es una ventaja para el biodiesel obtenido ya que altos valores de este parámetro pueden evitar tanto pérdidas de potencia en el motor como mayor consumo del mismo, además disminuir el tiempo de encendido del motor. [30]

Tabla 34. Resultados de poder calorífico

<i>Característica</i>	<i>Unidad</i>	<i>Resultado</i>	<i>Método</i>	<i>Especificación</i>	
			ASTM	Min	Max
Poder Calorífico	Btu/Lb	18 019	D-240	-	-

4.2. Comparación de características físico químicas obtenidas

En la siguiente tabla se muestra un consolidado de todas las características obtenidas, el requerimiento se basa en la norma ASTM 6751, mientras que los datos adicionales de comparación fueron obtenidos de investigaciones similares, para poder establecer el nivel de competitividad y proyección al mercado que tiene el biodiesel a partir de aceite de moringa.

Tabla. 35. Características fisicoquímicas del Biodiesel de moringa, girasol y diésel comercial

Características	Unidad	Resultado	Requerimiento	Biodiesel Girasol	Diesel Comercial
Punto Inflamación	°C	103	93 mín.	178.67	53
Grados API		27.9	-	-	-
Densidad 15 °C	g/mL	0.8872	0.86 – 0.90	0.884	0.85
Destilación 90%	°C	340	- - -	-	-
Viscosidad Cinemática 40 °C	mm ² /s	5.087	1.9 - 6.0	5.03	2.36
Cenizas Sulfatadas	%p	0.004	0.02 máx.	0.0013	0.1014
Poder Calorífico	Btu/Lb	18 019	-----	16 767	19 386
Contenido de Agua	%v	0.00	0.05 máx.	0.1992	0.005

Índice de Cetano		45.00	47 mín.	45.44	43
Residuo Carbonoso	%p	0.3	0.3 máx.	0.070	0.20

Fuente: Elaboración propia

Fuente de Apoyo: [31]

Como se ya se mencionó en los puntos anteriores cada una de las características y todas se encuentran dentro de la norma establecida, el resultado de la investigación de concluye como favorable.

Existen dos características que crean una diferencia notable entre el biodiesel obtenido y un diésel comercial, en este caso son el punto de inflamación y el poder calorífico, que basados en las investigación y datos recopilados son las más importantes, debido a que de ellas depende la efectividad y rendimiento del combustible cuando se emplea en un motor.

CAPITULO V

CONCLUSIONES

En el proceso de transesterificación se detalla el diseño experimental con sus respectivas condiciones óptimas reacción, con un rendimiento del 63.37%, que nos permitió obtener un éster metílico (biodiesel) a partir del aceite extraído de las semillas de la moringa oleífera.

En la extracción del aceite mediante el método soxhlet, a partir de la semilla de moringa oleífera, aplicando como solvente el hexano, se establece un rendimiento del 26%, que muestra alta efectividad, factor favorable para su aplicabilidad industrial. Además, se determinó, en la reacción de transesterificación mediante las variantes durante la operación, que el ensayo 6B fue el óptimo y destacado en la obtención de biodiesel, para las variables de condición de reacción, temperatura en rango de 60 - 70°C (65 °C), velocidad de agitación 600 rpm (media), relación molar 6:1, y tiempo de 60 min.

Se realizó la caracterización fisicoquímica del biodiesel obtenido, siendo estos puntos inflamación 103 °C, densidad (15 °C) 0.8872 g/ml, destilación 90%, 340 °C, viscosidad cinemática (40°C) 5.087 mm²/s, cenizas sulfatadas 0.004 %p, poder calorífico 18019 Btu/lb, contenido de agua 0.00 %v, índice de cetano 45.00, residuo carbonoso 0.3 %p.

El biodiesel obtenido de la reacción de transesterificación fue evaluado en base a los resultados de su caracterización, con la norma ASTM para biodiésel D6751-09, los cuales se encuentran dentro de las especificaciones establecidas por la norma, además de ser comparado con un biodiesel de girasol

y un diésel comercial que nos permite establecer un nivel de competitividad y proyección al mercado.

Con los resultados obtenidos, se puede concluir que el desarrollo experimental para la obtención de biodiesel a partir del aceite extraído de la semilla de la moringa oleífera, se pueda replicar a gran escala, de tal manera que se le aplique su uso industrial, y se presente como una nueva propuesta de mercado en el Ecuador.

RECOMENDACIONES

Las condiciones de reacción establecidas no son fijas, pueden presentar variación por lo cual se recomienda una repetibilidad de los ensayos, realizando una variación de la relación molar y los porcentajes establecidos en el diseño experimental con respecto al catalizador, para complementar la investigación y determinar otras condiciones óptima de reacción.

En relación a catalizador se recomienda el uso de hidróxido de sodio, tal cual se realizó en la investigación, ya que basados en la literatura su efecto es superior al hidróxido de potasio. Existen tres tipos de catalizadores heterogéneos, los óxidos metales alcalinotérreos, las zeolitas y heteropoliácidos que han demostrado ser eficientes, selectivos, fáciles de separar, reutilizables y algunos más que otros, tolerables a la presencia de agua y ácidos grasos libres.

La reacción debe darse bajo condiciones ambientales normales, para el buen desarrollo de la misma, por otro lado, el uso del alcohol podría variar, sin embargo, al utilizar el etanol, que es un alcohol con dos átomos de carbono, puede inducir a la reacción al desarrollo y estabilidad del subproducto de la misma, como lo es el glicerol, por lo tanto si de la reacción de transesterificación se desea obtener un éter metílico estable, usar solamente el metanol.

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Apéndices

Apéndice 1 Abreviaturas

NaOH	Hidróxido de Sodio
TG	Triglicérido
DG	Diglicérido
MG	Monoglicérido
ME	Metil-esteres
BD	Biodiesel
H₂PO₃	Ácido Fosfórico
CH₃OH	Metanol
C₃H₅(OH)₃	Glicerina

Apéndice 2 Unidades

°C	Grados Celsius
°F	Fahrenheit
h	Hora
ml	Mililitro
g	Gramo
min	Minuto
s	Segundos
rpm	Revoluciones por minutos
mm²/s	Milímetro al cuadrado sobre segundos
%p	Porcentaje en peso
%v	Porcentaje en volumen
Btu/lb	British Thermal Unit (unidad de energía) sobre libras

Apéndice 3 Ilustraciones de la extracción del aceite de moringa



Ilustración 1 Semilla de moringa seca y molida



Ilustración 2 Pesaje de la muestra y medición del solvente



Ilustración 3. Montaje del equipo de soxhlet



Ilustración 4 Colocación de la muestra y del solvente dentro del equipo

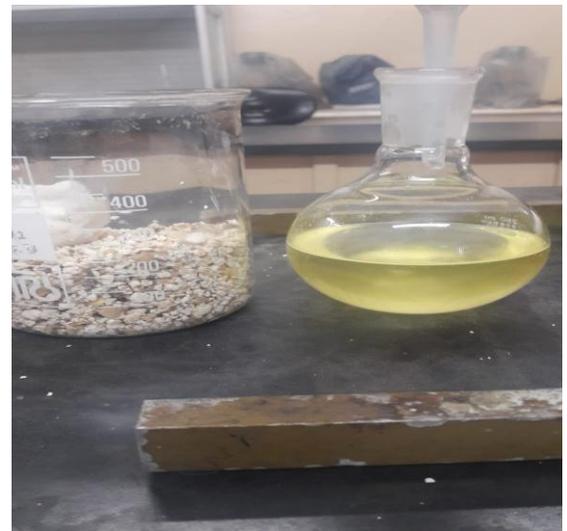


Ilustración 5 Aceite extraído más solvente



Ilustración 6 Recuperación del solvente con el rotavapor



Ilustración 7 Aceite extraído y hexano recuperado



Ilustración 8 Secado para la eliminación del exceso del hexano



Ilustración 9 Aceite final libre de solvente

Apéndice 4 Ilustraciones de la obtención de biodiesel



A



Ilustración 10 Preparación del metóxido



Ilustración 11 Calentamiento de aceite



Ilustración 12 Reacción de transesterificación

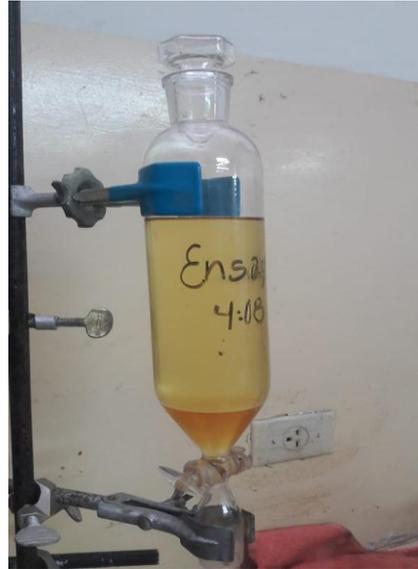


Ilustración 13 Decantación de la muestra



Ilustración 14 Extracción del biodiesel obtenido

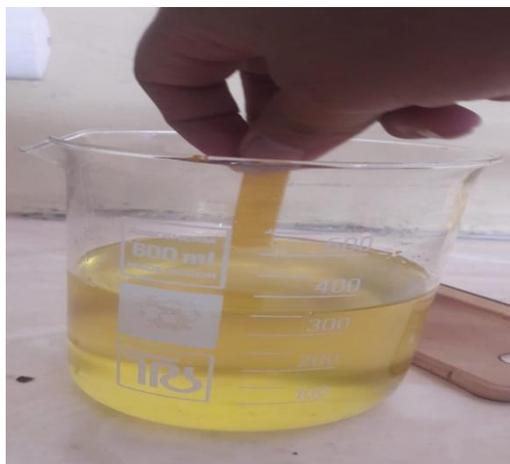


Ilustración 15 Medición del pH



Ilustración 16 Lavado del biodiesel



Ilustración 17 Secado del biodiesel



Ilustración 18 Biodiesel obtenido

Apéndice 5 Ilustraciones de la caracterización del biodiesel



Ilustración 19 Prueba de crepitación

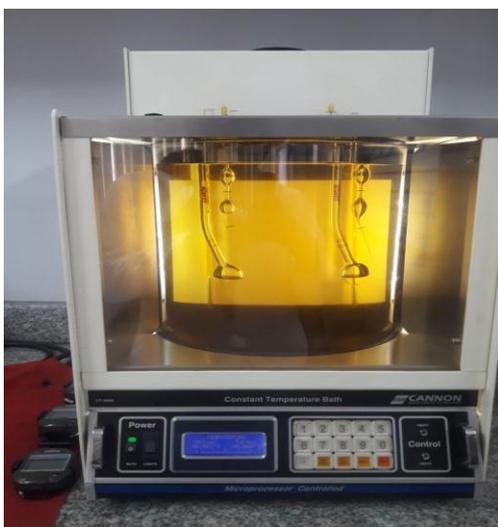


Ilustración 20 Viscosidad cinemática 40°C



Ilustración 21 Prueba de densidad



Ilustración 22 Destilación al 90°C



Ilustración 23 Punto de inflamación

Apéndice 6 Resultados de la Caracterización del biodiesel

	<p>UNIDAD DE CONTROL DE CALIDAD LABORATORIOS AGUAS-PETRÓLEO Y MEDIO AMBIENTE FACULTAD DE INGENIERÍA QUÍMICA UNIVERSIDAD DE GUAYAQUIL Cda. Universitaria Salvador Allende Teléfono: 22390947 - Fax: 2289883 Guayaquil - Ecuador</p>	
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**INFORME DE ANÁLISIS
LABORATORIO DE PETRÓLEO**

		INFORME N° : LP/018/19
SOLICITADO POR:	Karla Kocher Solano ¹	FECHA DE EMISIÓN: 24/06/2019
EMPRESA:		
DIRECCIÓN:	Samanes 1 Mz. 139 V:1 ¹	
FECHA Y HORA DE RECEPCIÓN:	20/06/2019	12h40
FECHA DE INICIO DE ANÁLISIS:	20/06/2019	
FECHA DE CULMINACIÓN DE ANÁLISIS:	21/06/2019	

BIODIESEL ¹

DETERMINACIÓN	UNIDAD	MÉTODO		U	RESULTADO	ESPECIFICACIÓN	
		ASTM	INTERNO			MIN.	MAX.
Punto de Inflamación c.c	°C	D-93	PEE/UCC/LP/03		103	93	-
Grados API		D-287	PEE/UCC/LP/05		27,9	-	-
Densidad 15°C		D-287			0,8872	0,86	0,9
Destilación		D-86	PEE/UCC/LP/01				
90%	°C				340	-	360
Viscosidad Cinemática 40°C	mm ² /s	D-287	PEE/UCC/LP/04		5,087	1,9	6,0
Cenizas sulfatadas	%p	D-874			0,004	-	0,02
Poder calorífico	Btu/Lb	D-240			18 019		
Contenido de Agua	%v	D-95			0,00	-	0,05
Índice de cetano		D-976			45,0	47,0	-
Residuo carbonoso	%p	D-189			0,3	-	0,3

Temperatura amb. : 22,5 °C Presión: 1006 hPa

Observaciones: Especificaciones proporcionadas por el cliente, establecidas en la NORMA ASTM para Biodiésel D6751-09

Los análisis fueron realizados de acuerdo al ASTM STANDARD



Autorizado: Ing. Elizabeth García Piloso
DIRECTOR LABORATORIO DE PETRÓLEO

Nota:

- * Los resultados obtenidos en este informe son exclusivos de la muestra a ensayo.
- * Queda prohibido la reproducción parcial o total de este informe sin previa autorización de esta Unidad.

Apéndice 7 Método Estándar para Gravedad API de petróleo crudo y productos derivados del petróleo



Designation: D287 – 12b

Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)¹

This standard is issued under the fixed designation D287; 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 by means of a glass hydrometer in conjunction with a series of calculations of the API gravity of crude petroleum and petroleum products normally handled as liquids and having a Reid vapor pressure (Test Method **D323**) of 101.325 kPa (14.696 psi) or less. Gravities are determined at 60°F (15.56°C), or converted to values at 60°F, by means of Adjunct to **D1250** Guide for Petroleum Measurement Tables (API *MPMS* Chapter 11.1). These tables are not applicable to nonhydrocarbons or essentially pure hydrocarbons such as the aromatics.

1.2 The initial values obtained are uncorrected hydrometer readings and not density measurements. Values are measured on a hydrometer at either the reference temperature or at another convenient temperature, and readings corrected for the meniscus effect, the thermal glass expansion effect, alternate calibration temperature effects and to the reference temperature by means of volume correction tables.

1.3 The hydrometer readings determined shall be recorded before performing any calculations. Then the calculations required in Section 9 shall be performed and documented before using the final result in a subsequent calculation procedure (measurement ticket calculation, meter factor calculation, or base prover volume determination).

1.4 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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 warning statement, see **8.3**.

¹ This test method is under the jurisdiction of ASTM Committee **D02** on Petroleum Products and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee **D02.02** /COMQ, the joint ASTM-API Committee on Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API).

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2. Referenced Documents

2.1 *ASTM Standards*:²

D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)

D1250 Guide for Use of the Petroleum Measurement Tables

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method (API *MPMS* Chapter 9.1)

D6822 Test Method for Density, Relative Density, and API Gravity of Crude Petroleum and Liquid Petroleum Products by Thermohydrometer Method (API *MPMS* Chapter 9.3)

E1 Specification for ASTM Liquid-in-Glass Thermometers

E100 Specification for ASTM Hydrometers

2.2 *EI Standards*:³

Specifications for **IP Standard Thermometers**

IP Specifications for Petroleum Hydrometers

2.3 *API Standards*:⁴

MPMS Chapter 9.1 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method (ASTM Test Method **D1298**)

MPMS Chapter 9.3 Test Method for Density, Relative Density, and API Gravity of Crude Petroleum and Liquid Petroleum Products by Thermohydrometer Method (ASTM Test Method **D6822**)

MPMS Chapter 11.1 Temperature and Pressure Volume Correction Factors for Generalized Crude Oils, Refined Products, and Lubricating Oils (Adjunct to ASTM **D1250**)

2.4 *ASTM Adjuncts*:

Adjunct to **D1250** Guide for Petroleum Measurement Tables (API *MPMS* Chapter 11.1)⁵

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>.

⁴ Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, <http://www.api.org>.

⁵ Available from ASTM International Headquarters. Order Adjunct No. **ADJD1250**. Original adjunct produced in 1983.

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

3. Terminology

3.1 Definitions:

3.1.1 *API gravity, n*—a special function of relative density 60/60°F (15.56/15.56°C), represented by:

$$^{\circ}\text{API} = [141.5 / (\text{relative density } 60/60^{\circ}\text{F})] - 131.5 \quad (1)$$

No statement of reference temperature is required, since 60°F is included in the definition.

3.1.2 *hydrometer reading, n*—the point on the hydrometer scale at which the surface of the liquid cuts the scale.

3.1.2.1 *Discussion*—In practice for transparent fluids this can be readily determined by aligning the surface of the liquid on both sides of the hydrometer and reading the Hydrometer scale where these surface readings cut the scale (Hydrometer Reading – Observed). For nontransparent fluids the point at which the liquid surface cuts the Hydrometer scale cannot be determined directly and requires a correction (Meniscus Correction). The value represented by the point (Meniscus Reading) at which the liquid sample rises above the main surface of the liquid subtracted from the value represented by where the main surface of the liquid cuts the Hydrometer scale is the amount of the correction or Meniscus correction. This meniscus correction is documented and then subtracted from the value represented by the Meniscus Reading to yield the Hydrometer Reading corrected for the Meniscus (Hydrometer Reading – Observed, Meniscus Corrected).

3.1.3 *specific gravity, n*—historical term, no longer used, which has been replaced by *relative density*.

4. Summary of Test Method

4.1 This test method is based on the principle that the gravity of a liquid varies directly with the depth of immersion of a body floating in it. The floating body, which is graduated by API gravity units in this test method, is called an API hydrometer.

4.2 The API gravity is read by observing the freely floating API hydrometer and noting the graduation nearest to the apparent intersection of the horizontal plane surface of the liquid with the vertical scale of the hydrometer, after temperature equilibrium has been reached. The temperature of the sample is read from a separate accurate ASTM thermometer in the sample.

4.3 The observed hydrometer reading is corrected for the meniscus effect, the thermal glass expansion effect, alternate calibration temperature effects and reduced to the reference temperature by means of the volume correction tables. If necessary, the hydrometer cylinder and its contents are placed in a constant temperature bath to avoid excessive temperature variation during the test.

5. Significance and Use

5.1 Accurate determination of the gravity of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperature of 60°F (15.56°C).

5.2 This procedure is most suitable for determining the API gravity of low viscosity transparent liquids. This test method can also be used for viscous liquids by allowing sufficient time for the hydrometer to reach temperature equilibrium, and for

opaque liquids by employing a suitable meniscus correction. Additionally for both transparent and opaque fluids the readings shall be corrected for the thermal glass expansion effect before correcting to the reference temperature.

5.3 When used in connection with bulk oil measurements, volume correction errors are minimized by observing the hydrometer reading at a temperature as close to reference temperature as feasible.

5.4 Gravity is a factor governing the quality of crude oils. However, the gravity of a petroleum product is an uncertain indication of its quality. Correlated with other properties, gravity can be used to give approximate hydrocarbon composition and heat of combustion.

5.5 Gravity is an important quality indicator for automotive, aviation and marine fuels, where it affects storage, handling and combustion.

6. Apparatus

6.1 *Hydrometers*, of glass, graduated in degrees API as listed in Table 1 and conforming to Specification E100.

6.1.1 The user should ascertain that the instruments used for this method conform to the requirements set out above with respect to materials, dimensions, and scale errors. In cases where the instrument is provided with a calibration certificate issued by a recognized standardizing body, the instrument is classed as certified and the appropriate corrections for the meniscus effect, the thermal glass expansion effect, and alternative calibration temperature effects shall be applied to the observed readings prior to corrections. Instruments that satisfy the requirements of this test method, but are not provided with a recognized calibration certificate, are classed as uncertified.

6.2 *Thermometers*, having a range from –5 to +215°F and conforming to the requirements for Thermometer 12F as prescribed in Specification E1 or Thermometer 64F of the Specification for IP Standard Thermometers.

6.2.1 Alternate measuring devices or systems may be used, provided that the total uncertainty of the calibrated system is no greater than when using liquid-in-glass thermometers. The stated repeatability and reproducibility values are not applicable if alternate fluids are used in the liquid-in-glass thermometers.

NOTE 1—The ASTM Gravity Thermometer 12F has 0.5°F subdivisions and allowable ±0.25°F scale error.

6.3 *Hydrometer Cylinder*, clear glass, plastic, or metal (see 6.3.1). The inside diameter of the cylinder shall be at least 25 mm greater than the outside diameter of the hydrometer and the height shall be such that the appropriate hydrometer floats in the test portion with at least 25 mm clearance between the bottom of the hydrometer and the bottom of the cylinder.

TABLE 1 Available Hydrometers Scaled, Degrees API

Designation	Type	API Range, deg		Scale	
		Series Total	Each Unit	Division	Error
1H to 10H	long plain	–1 to 101	12	0.1	0.1
21H to 40H	short plain	0 to 101	6	0.1	0.2
51H to 60H	thermo	–1 to 101	12	0.1	0.1
71H to 74H	thermo	–1 to 41	12	0.1	0.1
^A	thermo	15 to 51	8		

^A Eight-degree range thermohydrometers are available.

6.3.1 Hydrometer cylinders constructed of plastic materials shall be resistant to discoloration or attack by oil samples and shall not affect the material being tested. They shall not become opaque under prolonged exposure to sunlight.

7. Temperature of Test

7.1 The gravity determined by the hydrometer method is most accurate at or near the standard temperature of 60°F (15.56°C). Use this or any other temperature between 0 and 195°F (-18 and + 90°C) for the test, so far as it is consistent with the type of sample and necessary limiting conditions shown in **Table 2**.

8. Procedure

8.1 For referee testing, use the long plain form of hydrometer (1H to 10H). For field testing, use the thermohydrometer method in Test Method **D6822** (API *MPMS* Chapter 9.3).

8.2 Adjust the temperature of the sample in accordance with **Table 2**. For field testing, test temperatures other than those listed in **Table 2** may be used. The hydrometer cylinder shall be approximately the same temperature as the sample to be tested.

8.3 Transfer the sample into the clean hydrometer cylinder without splashing, so as to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower boiling constituents of the more volatile samples. (**Warning**—Extremely flammable. Vapors may cause flash fire.) For the more volatile samples, transfer to the hydrometer cylinder by siphoning. (Do not start the siphon by mouth.) Use a rubber aspirator bulb. Remove any air bubbles formed, after they have collected on the surface of the sample, by touching them with a piece of clean filter paper or other suitable means before inserting the hydrometer. For field testing, make the gravity measurement directly in the sampling thief. Place the cylinder containing the sample in a vertical position in a location free from air currents. Take precautions to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. During this period, the temperature of the surrounding medium should not change more than 5°F (2°C).

8.4 Lower the hydrometer gently into the sample and, when it has settled, depress it about two scale divisions into the liquid and then release it; keep the rest of the stem dry, as unnecessary liquid on the stem changes the effective weight of the instrument, and so affects the reading obtained. With samples of low viscosity, a slight spin imparted to the instrument on releasing assists in bringing it to rest, floating freely away from the walls

of the hydrometer cylinder. Allow sufficient time for the hydrometer to become completely stationary and for all air bubbles to come to the surface. This is particularly necessary in the case of the more viscous samples.

8.5 When the hydrometer has come to rest, floating freely, and the temperature of the sample is constant to 0.2°F (0.1°C), read the hydrometer to the nearest scale division. The correct reading is that point on the hydrometer scale at which the surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

8.6 To make a reading with nontransparent liquids, observe the point on the hydrometer scale to which the sample rises above its main surface, placing the eye slightly above the plane surface of the liquid. This reading requires a correction. Determine this correction for the particular hydrometer in use by observing the height above the main surface of the liquid to which the sample rises on the hydrometer scale when the hydrometer in question is immersed in a transparent liquid having a surface tension similar to that of a sample under test.

8.7 Observe the temperature of the sample to the nearest 0.25°F (0.1°C) immediately before and after the observation of the gravity, the liquid in the cylinder being thoroughly but cautiously stirred with the thermometer (**Note 2**), and the whole of the mercury thread being immersed. Should these temperature readings differ by more than 1°F (0.5°C), repeat the temperature and gravity observations when the temperature of the sample has become more stable. Record the mean of the thermometer reading before and after the final hydrometer reading, to the nearest 1°F, as the temperature of the test.

NOTE 2—When thermohydrometers are used, stir the sample by carefully raising and lowering the hydrometer. It is satisfactory in this case to read the thermometer scale after the hydrometer reading has been observed. Read the thermometer to the nearest 1°F (0.5°C).

9. Calculation

9.1 Apply any relevant thermometer corrections to the temperature reading observed in **8.3** and **8.7** and record the average of those two temperatures to the nearest 1°F.

9.2 Record the observed hydrometer scale readings to the nearest 0.1° API for transparent liquids.

9.3 When gravities have been observed on opaque liquids using the procedure given in **8.6**, subtract the meniscus correction from the hydrometer reading observed.

TABLE 2 Limiting Conditions and Testing Temperatures

Sample Type	Gravity Limits	Initial Boiling Point Limits	Other Limits	Test Temperature
Highly volatile	lighter than 70° API			Cool to 35°F (2°C) or lower in original closed container.
Moderately volatile	heavier than 70° API	below 250°F (120°C)		Cool to 65°F (18°C) or lower in original closed container.
Moderately volatile and viscous	heavier than 70° API	below 250°F (120°C)	Viscosity too high at 65°F (18°C)	Heat to minimum temperature for sufficient fluidity.
Nonvolatile	heavier than 70° API	above 250°F (120°C)		Any temperature between 0 and 195°F (-18 and 90°C) as convenient.
Mixtures of nonpetroleum products or essentially pure hydrocarbons				60 ± 0.25°F (15.56 ± 0.1°C)

NOTE 3—The meniscus correction for a particular hydrometer in use is determined by observing the maximum height above the principal surface of the liquid to which liquid rises on the hydrometer scale when the hydrometer in question is immersed in a transparent liquid having a surface tension similar to that of the sample under test.

9.4 Apply any meniscus hydrometer correction to the observed hydrometer reading and record the meniscus corrected hydrometer scale reading to the nearest 0.1° API.

9.5 Application of the glass thermal expansion correction depends upon what edition of Adjunct to D1250 Guide for Petroleum Measurement Tables (API MPMS Chapter 11.1) will be used to calculate the base density.

9.5.1 The 1980 version of the Adjunct to D1250 Guide for Petroleum Measurement Tables (API MPMS Chapter 11.1) has the hydrometer glass thermal expansion correction included. Input into the VCF software requires the Hydrometer Reading – Observed or Hydrometer Reading – Observed, Meniscus Corrected in API units from 9.2 or 9.4, observed temperature of the sample, and the built-in hydrometer glass thermal correction switch set to on (0) or off (1). It will return API @ 60°F.

9.5.2 The 2004 version of the Adjunct to D1250 Guide for Petroleum Measurement Tables (API MPMS Chapter 11.1) does not include the hydrometer glass thermal expansion correction, so that correction must be made before entering the software. Depending on the specific end use of the calculation results, the final value may be left rounded or unrounded. See 9.6.

9.6 The following steps are required to implement 9.5.2:

Step 1. Convert the meniscus corrected hydrometer scale reading to density in kg/m³ using Eq 2.

Hydrometer Scale Reading Units	Conversion to Density
For API gravity:	
	$density (kg/m^3) = (141.5 * 999.016) / (131.5 + API)$ (2)

Leave the result unrounded.

Step 2. Calculate the hydrometer thermal glass expansion correction factor (HYC) using the appropriate equation below (*t* is observed temperature).

Correction for a Base Temperature (*T_b*) of 60°F:

$$HYC = 1.0 - [0.00001278 (t - 60)] - [0.000000062 (t - 60)^2]$$
 (3)

Leave the result unrounded.

Step 3. Multiply the hydrometer reading in kg/m³ from Step 1 by HYC from Step 2 to obtain the glass expansion corrected hydrometer reading.

$$kg/m^3_{HYC} = kg/m^3 * HYC$$
 (4)

Step 4a. Convert the hydrometer reading in density (kg/m³_{HYC}) from Step 3 to a R.D. (relative density) hydrometer reading.

NOTE 4—The current C source code, compiled dll and Excel Add-in has an omission and cannot use a kg/m³ call with degree F.

$$R.D. = kg/m^3_{HYC} / 999.016$$
 (5)

Step 4b. Input R.D. and degree F into section 11.1.6.2 of the Adjunct to D1250-04 Guide for Petroleum Measurement Tables (API MPMS Chapter 11.1-2004) which returns R.D. @ 60°F.

NOTE 5—Pressure will have to be atmospheric gauge, or 0 psig as the Adjunct to D1250 Guide for Petroleum Measurement Tables (API MPMS Chapter 11.1) values are only valid at atmospheric pressure.

Step 4c. Convert the calculated R.D. value @ 60°F to a calculated API Gravity @ 60°F using Eq 6.

$$API\ Gravity = (141.5 / R.D.) - 131.5$$
 (6)

9.7 Future versions of the Adjunct to D1250 Guide for Petroleum Measurement Tables (API MPMS Chapter 11.1) code will be corrected so that it can accept any combination of input units and return any combination of output units. When available, the Adjunct to D1250 Guide for Petroleum Measurement Tables (API MPMS Chapter 11.1) code can be accessed directly from Step 3 and return API Gravity @ 60 °F, R.D. @ 60 °F, and kg/m³ at any selected base temperature.

Example 1

Sample:	Crude Oil	
Observed Temperature:	77°F	
Observed Hydrometer Reading:	33.2 API Gravity	
Observed Pressure:	0 psig	
Base Temperature:	60°F	
Step 1:	858.292434730...	(Eq 2)
Step 2:	0.999780948...	(Eq 3)
Step 3:	858.104424227...	(Eq 4)
Step 4a:	0.858949631...	(Eq 5)
Step 4b:	0.865678279...	(Eq 6)
Step 4c.1:	31.955643312...	unrounded (Eq 6)
Step 4c.2:	32.0 °API rounded	(Eq 6)

10. Report

10.1 Report the corrected hydrometer reading as degrees API (°API) or as API Gravity.

10.2 Report the final value as API gravity, at the reference temperature, to the nearest 0.1° API.

10.3 The reporting values have no precision or bias determination. It is up to the user to determine whether this test method provides results of sufficient accuracy for the intended purpose.

10.4 If the hydrometer readings are being used as an input to a calculation process intended to return a volume correction factor for use in ticket or meter proving calculations, stop the calculation process identified above at Step 3 (if the density value is desired at flowing conditions) or Step 4 (if the density value is desired at base density conditions) and input the results into the calculation process.

10.5 Certified hydrometers from a recognized standardizing body, such as NIST, report the output density as 'Density in Vacuo'.

11. Precision and Bias

11.1 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

11.1.1 *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 0.2° API only in one case in twenty.

11.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators, working in different laboratories on identical test material, would in

 **D287 – 12b**

the long run, in the normal and correct operation of the test method, exceed 0.5° API only in one case in twenty.

NOTE 6—The precision for this test method was not obtained in accordance with RR:D02-1007.

NOTE 7—This precision statement applies only to measurements made at temperatures differing from 60°F (15.56°C) by less than 18°F (10°C).

11.2 *Bias*—Bias for this test method has not been determined.

12. Keywords

12.1 API gravity; crude petroleum; hydrometer; thermohydrometer; thermometer

SUMMARY OF CHANGES

Subcommittee D02.02 has identified the location of selected changes to this standard since the last issue (D287–12a) that may impact the use of this standard. (Approved June 1, 2012)

(1) Revised Section 1.

(2) Revised Section 3.

Subcommittee D02.02 has identified the location of selected changes to this standard since the last issue (D287–12) that may impact the use of this standard. (Approved May 15, 2012)

(1) Revised Section 9.

Subcommittee D02.02 has identified the location of selected changes to this standard since the last issue (D287–92(2006)) that may impact the use of this standard. (Approved April 1, 2012)

(1) Expanded Section 8.

(3) Revised Sections 10 and 11.

(2) Added 9.6.

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Apéndice 8 Método estándar para el índice calculado de cetano de los combustibles del destilado



Designation: D 976 – 06

An American National Standard

Standard Test Method for Calculated Cetane Index of Distillate Fuels¹

This standard is issued under the fixed designation D 976; 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 the Calculated Cetane Index formula, which represents a means for directly *estimating* the ASTM cetane number of distillate fuels from API gravity and mid-boiling point. The index value, as computed from the formula, is termed the Calculated Cetane Index.²

1.2 The Calculated Cetane Index is not an optional method for expressing ASTM cetane number. It is a supplementary tool to estimate cetane number when used with due regard for its limitations.

1.3 The Calculated Cetane Index formula is particularly applicable to straight-run fuels, catalytically cracked stocks, and blends of the two.

NOTE 1—This test method is temporarily retained because the proposal to the U.S. EPA to control diesel fuel aromatics concentrations via a 40 Calculated Cetane Index minimum is based on the correlation between Test Method D 976 and aromatics concentration. Test Method D 4737 is the preferred method as estimator of cetane number.

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.*

2. Referenced Documents

- 2.1 *ASTM Standards:*³

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0.02 on Diesel Fuel Oils.

Current edition approved Dec. 1, 2006. Published January 2007. Originally approved in 1966. Last previous edition approved in 2004 as D 976-04b¹.

² A method of calculating cetane index was developed by the Diesel Fuels Division, Coordinating Fuel and Equipment Research Committee of the Coordinating Research Council. See Young, H. D., "Methods for Estimating Cetane Number," *Proceedings*, PPIRA, American Petroleum Institute, Vol. 30 M (III), 1950. This method was revised in 1960 by Research Division I of Committee D02 to conform to the revised Test Method D 613.

³ 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.

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D 613 Test Method for Cetane Number of Diesel Fuel Oil

D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter

D 4737 Test Method for Calculated Cetane Index by Four Variable Equation

2.2 *ASTM Adjuncts:*⁴

Nomograph for Calculated Cetane Index

3. Significance and Use

3.1 The Calculated Cetane Index is one tool available for estimating ASTM cetane number where a test engine is not available for determining this property. It may be employed for approximating cetane number where the quantity of sample is too small for an engine rating. In cases where the cetane number of a fuel has been initially established, the index is useful as a cetane number check on subsequent samples of that fuel, provided its source and mode of manufacture remain unchanged.

3.2 Test Method D 4737 may also be used to approximate the ASTM cetane number of diesel fuels.

3.2.1 Procedure A of Test Method D 4737 was developed as a result of a larger degree of offset between Test Method D 976 Cetane Index and the results of Test Method D 613 over the entire range of the correlation. Generally, it has been found that use of Test Method D 4737 results in less offset than use of Test Method D 976, but there can be specific cases where this is not true.

⁴ Available from ASTM International Headquarters. Order Adjunct No. ADJD0976. Original adjunct produced in 1989.

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

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3.2.2 Procedure A of Test Method **D 4737** is recommended to estimate the cetane number of diesel fuels with sulfur contents above 500 ppm or No. 1–D diesel fuels.

3.2.3 Procedure B of Test Method **D 4737** is recommended to estimate the cetane number of No. 2–D diesel fuels with sulfur contents at or below 500 ppm.

3.3 Calculated Cetane Index, as described in Test Method **D 976–80**, is recognized by the United States EPA as an alternative method to meet the U.S. Federal Diesel aromatics limit for diesel fuels containing less than 500 ppm sulfur. The equation for Calculated Cetane Index in Test Method **D 976–80** and in this version (**D 976–06**) of the test method are the same.

4. Equation for Calculated Cetane Index

4.1 The Calculated Cetane Index is determined from the following equation:

$$\text{Calculated cetane index} = -420.34 + 0.016 G^2 + 0.192 G \log M + 65.01 (\log M)^2 - 0.0001809 M^2 \quad (1)$$

or

$$\text{Calculated cetane index} = 454.74 - 1641.416 D + 774.74 D^2 - 0.554 B + 97.803 (\log B)^2 \quad (2)$$

where:

G = API gravity, determined by Test Method **D 287**, **D 1298**, or **D 4052**,

M = mid-boiling temperature, °F, determined by Test Method **D 86** and corrected to standard barometric pressure,

D = density at 15°C, g/mL, determined by Test Method **D 1298** or **D 4052**, and

B = mid-boiling temperature, °C, determined by Test Method **D 86** and corrected to standard barometric pressure.

4.2 Calculated Cetane Index values for distillate fuels may be conveniently determined by means of the alignment chart in **Fig. 1** rather than by direct application of the equation. Use of the chart is illustrated by the example shown on the chart.

4.3 Test Method **D 2887** may be used as an alternative to Test Method **D 86** to determine the mid-boiling temperature of the fuel.

4.3.1 If Test Method **D 2887** is used, convert the data to estimated Test Method **D 86** data following Appendix X5,

Correlation of Jet and Diesel Fuel of test Method **D 2887** and use the data from Test Method **D 86** in place of the actual Test Method **D 86** data in the calculations.

NOTE 2—Current U.S. EPA regulations for low sulfur (500 ppm maximum) No. 1–D and No. 2–D diesel fuel require the use of Test Method **D 976–80** to calculate Cetane Index.

5. Limitations of Equation

5.1 The Calculated Cetane Index equation possesses certain inherent limitations which must be recognized in its application. These are:

5.1.1 It is not applicable to fuels containing additives for raising cetane number.

5.1.2 It is not applicable to pure hydrocarbons, synthetic fuels, such as certain products derived from shale oils and tar sands, alkylates, or coal-tar products.

5.1.3 Substantial inaccuracies in correlation may occur if used for crude oils, residuals, or products having a volatility of below 500°F end point.

6. Report

6.1 Round and report the calculated cetane index to the nearest tenth. Any calculated cetane index value ending in exactly “5” in the second decimal place shall be rounded to the nearest even tenth number. For example, round “35.55” and “35.65” to “35.6 cetane index.”

7. Precision and Bias

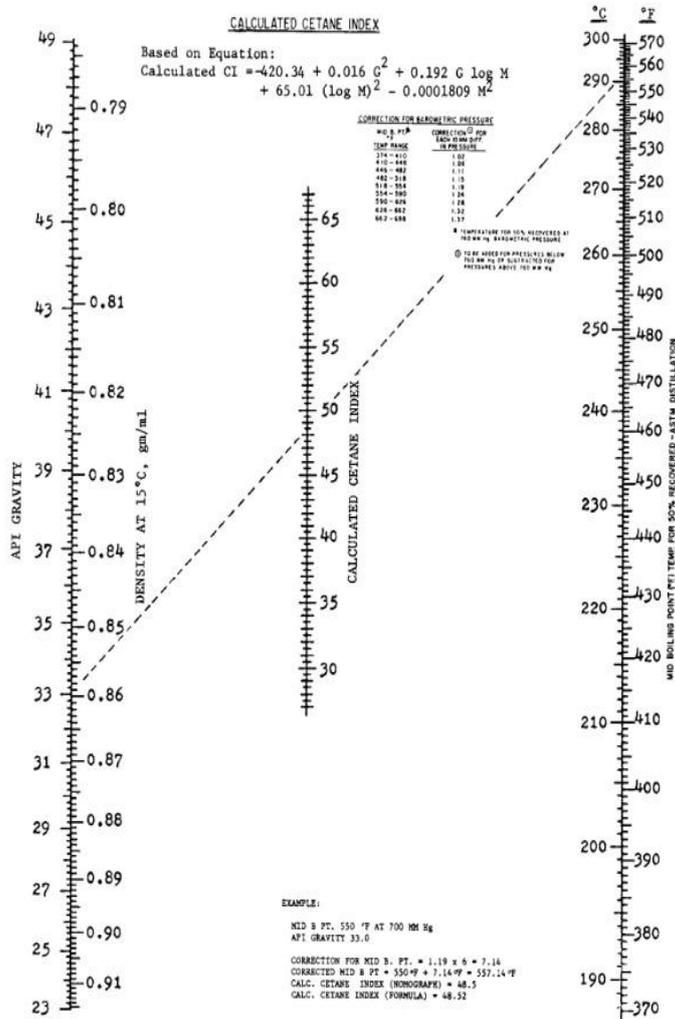
7.1 Correlation of index values with ASTM cetane number is dependent to a great extent upon the accuracy of determination of both API gravity and midboiling point.

7.2 Within the range from 30 to 60 cetane number, the expected correlation of the Calculated Cetane Index with the ASTM cetane number will be somewhat less than ± 2 cetane numbers for 75 % of the distillate fuels evaluated. Errors in correlation may be greater for fuels whose cetane numbers are outside this range. Correlation is best for straight-run and catalytically cracked distillates and blends of the two, and least satisfactory for blends containing substantial proportions of thermally cracked stocks.

8. Keywords

8.1 cetane; cetane index; diesel fuel

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NOTE—The Calculated Cetane Index equation represents a useful tool for *estimating* cetane number. Due to inherent limitations in the equation's application, Index values may not be a valid substitute for ASTM cetane numbers as determined in a test engine.

FIG. 1 Nomograph for Calculated Cetane Index (ECS-1 Meter Basis—Test Method D 613)



D 976 – 06

SUMMARY OF CHANGES

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D 976-04b^{e1}) that may impact the use of this standard.

- (1) Revised 1.2.
- (2) Revised 3.1.

- (3) Added 3.2-3.3.

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Standard Test Method for Calculated Cetane Index of Distillate Fuels¹

This standard is issued under the fixed designation D 976; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

1. Scope*

1.1 This test method covers the Calculated Cetane Index formula, which represents a means for directly *estimating* the ASTM cetane number of distillate fuels from API gravity and mid-boiling point. The index value, as computed from the formula, is termed the Calculated Cetane Index.²

1.2 The Calculated Cetane Index is not an optional method for expressing ASTM cetane number. It is a supplementary tool to estimate cetane number when used with due regard for its limitations.

1.3 The Calculated Cetane Index formula is particularly applicable to straight-run fuels, catalytically cracked stocks, and blends of the two.

NOTE 1—This test method is temporarily retained because the proposal to the U.S. EPA to control diesel fuel aromatics concentrations via a 40 Calculated Cetane Index minimum is based on the correlation between Test Method D 976 and aromatics concentration. Test Method D 4737 is the preferred method as estimator of cetane number.

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.*

2. Referenced Documents

2.1 ASTM Standards:³

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0.02 on Diesel Fuel Oils.

Current edition approved Dec. 1, 2006. Published January 2007. Originally approved in 1966. Last previous edition approved in 2004 as D 976-04b⁴.

² A method of calculating cetane index was developed by the Diesel Fuels Division, Coordinating Fuel and Equipment Research Committee of the Coordinating Research Council. See Young, H. D., "Methods for Estimating Cetane Number," *Proceedings*, PPIRA, American Petroleum Institute, Vol. 30 M [III], 1950. This method was revised in 1960 by Research Division I of Committee D02 to conform to the revised Test Method D 613.

³ 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.

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D 613 Test Method for Cetane Number of Diesel Fuel Oil

D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter

D 4737 Test Method for Calculated Cetane Index by Four Variable Equation

2.2 ASTM Adjuncts:⁴

Nomograph for Calculated Cetane Index

3. Significance and Use

3.1 The Calculated Cetane Index is one tool available for estimating ASTM cetane number where a test engine is not available for determining this property. It may be employed for approximating cetane number where the quantity of sample is too small for an engine rating. In cases where the cetane number of a fuel has been initially established, the index is useful as a cetane number check on subsequent samples of that fuel, provided its source and mode of manufacture remain unchanged.

3.2 Test Method D 4737 may also be used to approximate the ASTM cetane number of diesel fuels.

3.2.1 Procedure A of Test Method D 4737 was developed as a result of a larger degree of offset between Test Method D 976 Cetane Index and the results of Test Method D 613 over the entire range of the correlation. Generally, it has been found that use of Test Method D 4737 results in less offset than use of Test Method D 976, but there can be specific cases where this is not true.

⁴ Available from ASTM International Headquarters. Order Adjunct No. ADJD0976. Original adjunct produced in 1989.

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

3.2.2 Procedure A of Test Method **D 4737** is recommended to estimate the cetane number of diesel fuels with sulfur contents above 500 ppm or No. 1–D diesel fuels.

3.2.3 Procedure B of Test Method **D 4737** is recommended to estimate the cetane number of No. 2–D diesel fuels with sulfur contents at or below 500 ppm.

3.3 Calculated Cetane Index, as described in Test Method **D 976–80**, is recognized by the United States EPA as an alternative method to meet the U.S. Federal Diesel aromatics limit for diesel fuels containing less than 500 ppm sulfur. The equation for Calculated Cetane Index in Test Method **D 976–80** and in this version (**D 976–06**) of the test method are the same.

4. Equation for Calculated Cetane Index

4.1 The Calculated Cetane Index is determined from the following equation:

$$\text{Calculated cetane index} = -420.34 + 0.016 G^2 + 0.192 G \log M + 65.01 (\log M)^2 - 0.0001809 M^2 \quad (1)$$

or

$$\text{Calculated cetane index} = 454.74 - 1641.416 D + 774.74 D^2 - 0.554 B + 97.803 (\log B)^2 \quad (2)$$

where:

G = API gravity, determined by Test Method **D 287**, **D 1298**, or **D 4052**,

M = mid-boiling temperature, °F, determined by Test Method **D 86** and corrected to standard barometric pressure,

D = density at 15°C, g/mL, determined by Test Method **D 1298** or **D 4052**, and

B = mid-boiling temperature, °C, determined by Test Method **D 86** and corrected to standard barometric pressure.

4.2 Calculated Cetane Index values for distillate fuels may be conveniently determined by means of the alignment chart in **Fig. 1** rather than by direct application of the equation. Use of the chart is illustrated by the example shown on the chart.

4.3 Test Method **D 2887** may be used as an alternative to Test Method **D 86** to determine the mid-boiling temperature of the fuel.

4.3.1 If Test Method **D 2887** is used, convert the data to estimated Test Method **D 86** data following Appendix X5,

Correlation of Jet and Diesel Fuel of test Method **D 2887** and use the data from Test Method **D 86** in place of the actual Test Method **D 86** data in the calculations.

NOTE 2—Current U.S. EPA regulations for low sulfur (500 ppm maximum) No. 1–D and No. 2–D diesel fuel require the use of Test Method **D 976–80** to calculate Cetane Index.

5. Limitations of Equation

5.1 The Calculated Cetane Index equation possesses certain inherent limitations which must be recognized in its application. These are:

5.1.1 It is not applicable to fuels containing additives for raising cetane number.

5.1.2 It is not applicable to pure hydrocarbons, synthetic fuels, such as certain products derived from shale oils and tar sands, alkylates, or coal-tar products.

5.1.3 Substantial inaccuracies in correlation may occur if used for crude oils, residuals, or products having a volatility of below 500°F end point.

6. Report

6.1 Round and report the calculated cetane index to the nearest tenth. Any calculated cetane index value ending in exactly “5” in the second decimal place shall be rounded to the nearest even tenth number. For example, round “35.55” and “35.65” to “35.6 cetane index.”

7. Precision and Bias

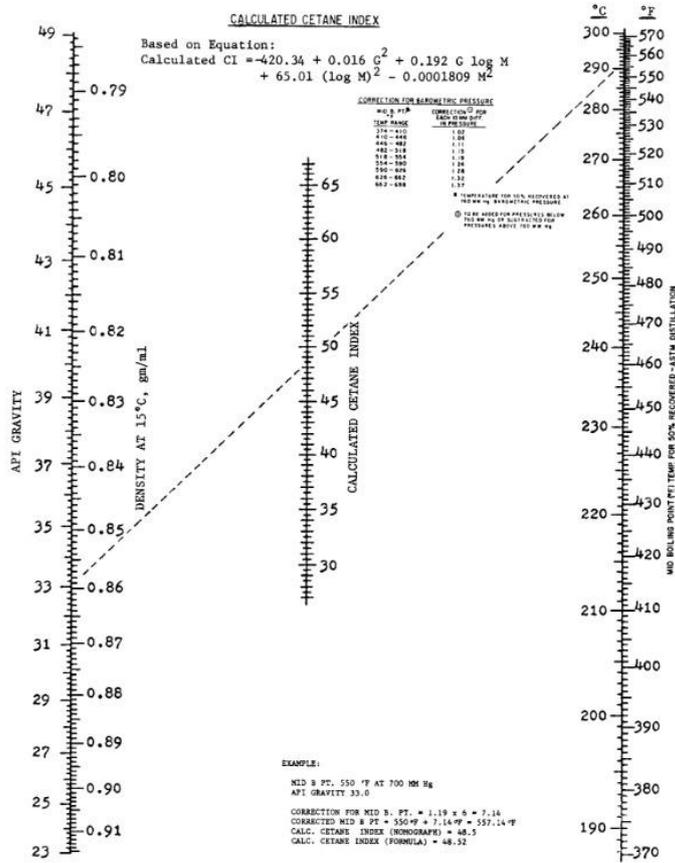
7.1 Correlation of index values with ASTM cetane number is dependent to a great extent upon the accuracy of determination of both API gravity and midboiling point.

7.2 Within the range from 30 to 60 cetane number, the expected correlation of the Calculated Cetane Index with the ASTM cetane number will be somewhat less than ± 2 cetane numbers for 75 % of the distillate fuels evaluated. Errors in correlation may be greater for fuels whose cetane numbers are outside this range. Correlation is best for straight-run and catalytically cracked distillates and blends of the two, and least satisfactory for blends containing substantial proportions of thermally cracked stocks.

8. Keywords

8.1 cetane; cetane index; diesel fuel

ASTM D 976 - 06



Note—The Calculated Cetane Index equation represents a useful tool for *estimating* cetane number. Due to inherent limitations in the equation's application, Index values may not be a valid substitute for ASTM cetane numbers as determined in a test engine.

FIG. 1 Nomograph for Calculated Cetane Index (ECS-1 Meter Basis—Test Method D 613)



D 976 – 06

SUMMARY OF CHANGES

Subcommittee D02.E0 has identified the location of selected changes to this standard since the last issue (D 976-04b^{e1}) that may impact the use of this standard.

- (1) Revised 1.2.
- (2) Revised 3.1.
- (3) Added 3.2-3.3.

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Apéndice 9 Método estándar para la destilación de productos de petróleo y aceites líquidos a presión atmosférica

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.



Designation: D86 – 18

Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure¹

This standard is issued under the fixed designation D86; 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 U.S. Department of Defense.

1. Scope*

1.1 This test method covers the atmospheric distillation of petroleum products and liquid fuels using a laboratory batch distillation unit to determine quantitatively the boiling range characteristics of such products as light and middle distillates, automotive spark-ignition engine fuels with or without oxygenates (see Note 1), aviation gasolines, aviation turbine fuels, diesel fuels, biodiesel blends up to 20 %, marine fuels, special petroleum spirits, naphthas, white spirits, kerosines, and Grades 1 and 2 burner fuels.

Note 1—An interlaboratory study was conducted in 2008 involving 11 different laboratories submitting 15 data sets and 15 different samples of ethanol-fuel blends containing 25 % volume, 50 % volume, and 75 % volume ethanol. The results indicate that the repeatability limits of these samples are comparable or within the published repeatability of the method (with the exception of FBP of 75 % ethanol-fuel blends). On this basis, it can be concluded that Test Method D86 is applicable to ethanol-fuel blends such as Ed75 and Ed85 (Specification D5798) or other ethanol-fuel blends with greater than 10 % volume ethanol. See ASTM RR:D02-1694 for supporting data.²

1.2 The test method is designed for the analysis of distillate fuels; it is not applicable to products containing appreciable quantities of residual material.

1.3 This test method covers both manual and automated instruments.

1.4 Unless otherwise noted, the values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

1.5 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause

serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 All standards are subject to revision, and parties to agreement on this test method are to apply the most recent edition of the standards indicated below, unless otherwise specified, such as in contractual agreements or regulatory rules where earlier versions of the method(s) identified may be required.

2.2 *ASTM Standards:*³

D97 Test Method for Pour Point of Petroleum Products

D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

In the IP, the equivalent test method is published under the designation IP 123. It is under the jurisdiction of the Standardization Committee.

Current edition approved Dec. 1, 2018. Published December 2018. Originally approved in 1921. Last previous edition approved in 2017 as D86 – 17. DOI: 10.1520/D0086-18.

² Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1694. Contact ASTM Customer Service at service@astm.org.

³ 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.

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

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D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
 D4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)
 D5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method) (Withdrawn 2012)⁴
 D5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)
 D5798 Specification for Ethanol Fuel Blends for Flexible-Fuel Automotive Spark-Ignition Engines
 D5842 Practice for Sampling and Handling of Fuels for Volatility Measurement
 D5949 Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)
 D5950 Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)
 D5985 Test Method for Pour Point of Petroleum Products (Rotational Method)
 D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants
 D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
 E1 Specification for ASTM Liquid-in-Glass Thermometers
 E77 Test Method for Inspection and Verification of Thermometers
 E1272 Specification for Laboratory Glass Graduated Cylinders
 E1405 Specification for Laboratory Glass Distillation Flasks
 2.3 *Energy Institute Standards*:⁵
 IP 69 Determination of Vapour Pressure—Reid Method
 IP 123 Petroleum Products—Determination of Distillation Characteristics
 IP 394 Determination of Air Saturated Vapour Pressure
 IP Standard Methods for Analysis and Testing of Petroleum and Related Products 1996—Appendix A

3. Terminology

3.1 Definitions:

3.1.1 *decomposition, n*—of a hydrocarbon, the pyrolysis or cracking of a molecule yielding smaller molecules with lower boiling points than the original molecule.

3.1.2 *decomposition point, n*—in distillation, the corrected temperature reading that coincides with the first indications of thermal decomposition of the specimen.

3.1.3 *dry point, n*—in distillation, the corrected temperature reading at the instant the last drop of liquid evaporates from the lowest point in the flask.

3.1.4 *dynamic holdup, n*—in D86 distillation, the amount of material present in the neck of the flask, in the sidearm of the flask, and in the condenser tube during the distillation.

⁴The last approved version of this historical standard is referenced on www.astm.org.

⁵Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

3.1.5 *emergent stem effect, n*—the offset in temperature reading caused by the use of total immersion mercury-in-glass thermometers in the partial immersion mode.

3.1.5.1 *Discussion*—In the partial immersion mode, a portion of the mercury thread, that is, the emergent portion, is at a lower temperature than the immersed portion, resulting in a shrinkage of the mercury thread and a lower temperature reading.

3.1.6 *end point (EP) or final boiling point (FBP), n*—the maximum corrected thermometer reading obtained during the test.

3.1.6.1 *Discussion*—This usually occurs after the evaporation of all liquid from the bottom of the flask. The term maximum temperature is a frequently used synonym.

3.1.7 *front end loss, n*—loss due to evaporation during transfer from receiving cylinder to distillation flask, vapor loss during the distillation, and uncondensed vapor in the flask at the end of the distillation.

3.1.8 *fuel ethanol (Ed75-Ed85), n*—blend of ethanol and hydrocarbon of which the ethanol portion is nominally 75 % to 85 % by volume denatured fuel ethanol. **D4175**

3.1.9 *initial boiling point (IBP), n*—in D86 distillation, the corrected temperature reading at the instant the first drop of condensate falls from the lower end of the condenser tube.

3.1.10 *percent evaporated, n*—in distillation, the sum of the percent recovered and the percent loss.

3.1.10.1 *percent loss, n*—in distillation, one hundred minus the percent total recovery.

3.1.10.2 *corrected loss, n*—percent loss corrected for barometric pressure.

3.1.11 *percent recovered, n*—in distillation, the volume of condensate collected relative to the sample charge.

3.1.11.1 *percent recovery, n*—in distillation, maximum percent recovered relative to the sample charge.

3.1.11.2 *corrected percent recovery, n*—in distillation, the percent recovery, adjusted for the corrected percent loss.

3.1.11.3 *percent total recovery, n*—in distillation, the combined percent recovery and percent residue.

3.1.12 *percent residue, n*—in distillation, the volume of residue relative to the sample charge.

3.1.13 *rate of change (or slope), n*—the change in temperature reading per percent evaporated or recovered, as described in 13.2.

3.1.14 *sample charge, n*—the amount of sample used in a test.

3.1.15 *temperature lag, n*—the offset between the temperature reading obtained by a temperature sensing device and the true temperature at that time.

3.1.16 *temperature measurement device, n*—a thermometer, as described in 6.3.1, or a temperature sensor, as described in 6.3.2.

3.1.16.1 *temperature reading, n*—the temperature obtained by a temperature measuring device or system that is equal to the thermometer reading described in 3.1.16.3.

3.1.16.2 *corrected temperature reading, n*—the temperature reading, as described in 3.1.16.1, corrected for barometric pressure.

3.1.16.3 *thermometer reading (or thermometer result), n*—the temperature of the saturated vapor measured in the neck of the flask below the vapor tube, as determined by the prescribed thermometer under the conditions of the test.

3.1.16.4 *corrected thermometer reading, n*—the thermometer reading, as described in 3.1.16.3, corrected for barometric pressure.

4. Summary of Test Method

4.1 Based on its composition, vapor pressure, expected IBP or expected EP, or combination thereof, the sample is placed in one of four groups. Apparatus arrangement, condenser temperature, and other operational variables are defined by the group in which the sample falls.

4.2 A 100 mL specimen of the sample is distilled under prescribed conditions for the group in which the sample falls. The distillation is performed in a laboratory batch distillation unit at ambient pressure under conditions that are designed to provide approximately one theoretical plate fractionation. Systematic observations of temperature readings and volumes of condensate are made, depending on the needs of the user of the data. The volume of the residue and the losses are also recorded.

4.3 At the conclusion of the distillation, the observed vapor temperatures can be corrected for barometric pressure and the data are examined for conformance to procedural requirements, such as distillation rates. The test is repeated if any specified condition has not been met.

4.4 Test results are commonly expressed as percent evaporated or percent recovered versus corresponding temperature, either in a table or graphically, as a plot of the distillation curve.

5. Significance and Use

5.1 The basic test method of determining the boiling range of a petroleum product by performing a simple batch distillation has been in use as long as the petroleum industry has existed. It is one of the oldest test methods under the jurisdiction of ASTM Committee D02, dating from the time when it was still referred to as the Engler distillation. Since the test method has been in use for such an extended period, a tremendous number of historical data bases exist for estimating end-use sensitivity on products and processes.

5.2 The distillation (volatility) characteristics of hydrocarbons have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behavior of the fuel during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors.

5.3 The distillation characteristics are critically important for both automotive and aviation gasolines, affecting starting, warm-up, and tendency to vapor lock at high operating

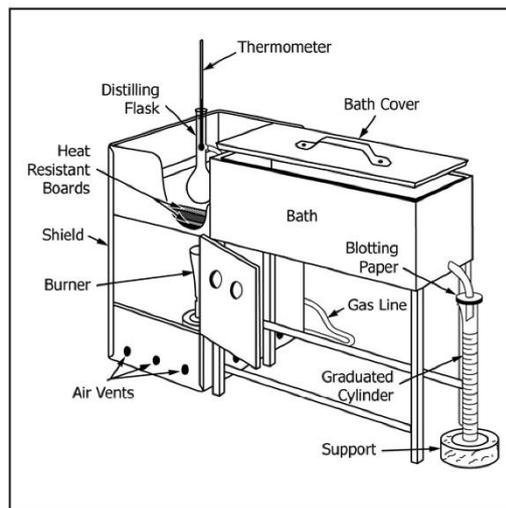


FIG. 1 Apparatus Assembly Using Gas Burner

temperature or at high altitude, or both. The presence of high boiling point components in these and other fuels can significantly affect the degree of formation of solid combustion deposits.

5.4 Volatility, as it affects rate of evaporation, is an important factor in the application of many solvents, particularly those used in paints.

5.5 Distillation limits are often included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for compliance to regulatory rules.

6. Apparatus

6.1 Basic Components of the Apparatus:

6.1.1 The basic components of the distillation unit are the distillation flask, the condenser and associated cooling bath, a metal shield or enclosure for the distillation flask, the heat source, the flask support, the temperature measuring device, and the receiving cylinder to collect the distillate.

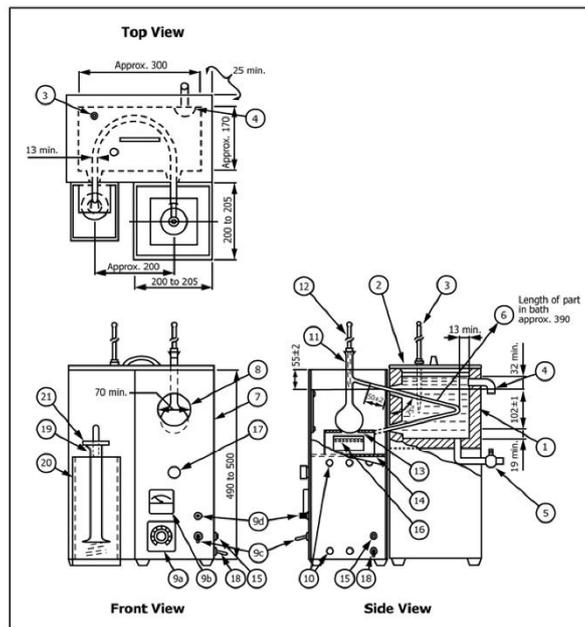
6.1.2 Figs. 1 and 2 are examples of manual distillation units.

6.1.3 In addition to the basic components described in 6.1.1, automated units also are equipped with a system to measure and automatically record the temperature and the associated recovered volume in the receiving cylinder.

6.2 A detailed description of the apparatus is given in Annex A2.

6.3 Temperature Measuring Device:

6.3.1 Mercury-in-glass thermometers, if used, shall be filled with an inert gas, graduated on the stem and enamel backed. They shall conform to Specification E1 or IP Standard Methods for Analysis and Testing of Petroleum and Related Products



- | | |
|---------------------------|---|
| 1-Condenser bath | 11-Distillation flask |
| 2-Bath cover | 12-Temperature sensor |
| 3-Bath temperature sensor | 13-Flask support board |
| 4-Bath overflow | 14-Flask support platform |
| 5-Bath drain | 15-Ground connection |
| 6-Condenser tube | 16-Electric heater |
| 7-Shield | 17-Knob for adjusting level of support platform |
| 8-Viewing window | 18-Power source cord |
| 9a-Voltage regulator | 19-Receiver cylinder |
| 9b-Voltmeter or ammeter | 20-Receiver cooling bath |
| 9c-Power switch | 21-Receiver cover |
| 9d-Power light indicator | |
| 10-Vent | |

FIG. 2 Apparatus Assembly Using Electric Heater

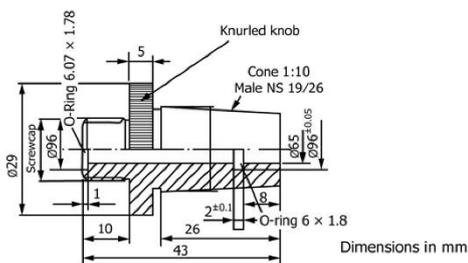


FIG. 3 PTFE Centering Device for Ground Glass Joint

1996—Appendix A, or both, for thermometers ASTM 7C/IP 5C and ASTM 7F for the low range thermometers, and ASTM 8C/IP 6C and ASTM 8F for the high range thermometers.

6.3.1.1 Thermometers that have been exposed for an extended period above an observed temperature of 370 °C shall not be reused without a verification of the ice point or checked as prescribed in Specification E1 and Test Method E77.

NOTE 2—At an observed thermometer reading of 370 °C, the temperature of the bulb is approaching a critical range in the glass and the thermometer may lose its calibration.

6.3.2 Temperature measurement systems other than those described in 6.3.1 are satisfactory for this test method, provided that they exhibit the same temperature lag, emergent stem effect, and accuracy as the equivalent mercury-in-glass thermometer.

6.3.2.1 The electronic circuitry or the algorithms, or both, used shall include the capability to simulate the temperature lag of a mercury-in-glass thermometer.

6.3.2.2 Alternatively, the sensor can also be placed in a casing with the tip of the sensor covered so that the assembly, because of its adjusted thermal mass and conductivity, has a temperature lag time similar to that of a mercury-in-glass thermometer.

NOTE 3—In a region where the temperature is changing rapidly during the distillation, the temperature lag of a thermometer can be as much as 3 s.

6.3.3 In case of dispute, the referee test method shall be carried out with the specified mercury-in-glass thermometer.

6.4 Temperature Sensor Centering Device:

6.4.1 The temperature sensor shall be mounted through a snug-fitting device designed for mechanically centering the sensor in the neck of the flask without vapor leakage. Examples of acceptable centering devices are shown in Figs. 3 and 4. (Warning—The use of a plain stopper with a hole drilled through the center is not acceptable for the purpose described in 6.4.1.)

NOTE 4—Other centering devices are also acceptable, as long as they position and hold the temperature sensing device in the proper position in the neck of the distillation column, as shown in Fig. 5 and described in 10.5.

NOTE 5—When running the test by the manual method, products with a low IBP may have one or more readings obscured by the centering device. See also 10.14.3.1.

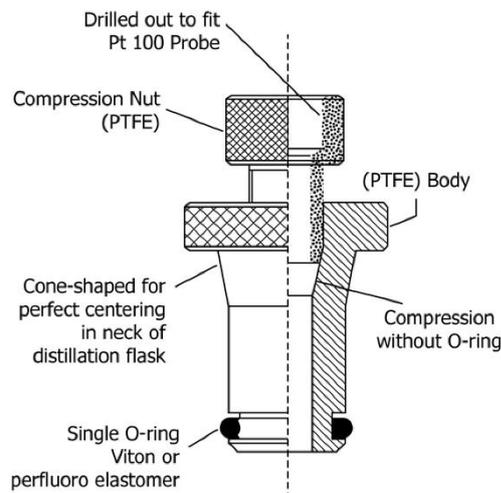


FIG. 4 Example of Centering Device Designs for Straight-Bore Neck Flasks

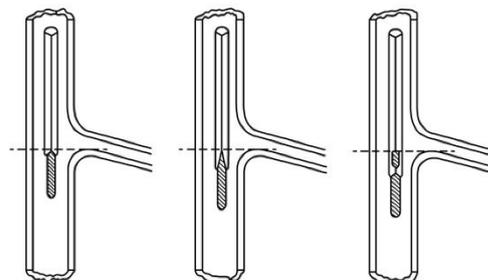


FIG. 5 Position of Thermometer in Distillation Flask

6.5 Automated equipment manufactured in 1999 and later shall be equipped with a device to automatically shut down power to the unit and to spray an inert gas or vapor in the chamber where the distillation flask is mounted in the event of fire.

NOTE 6—Some causes of fires are breakage of the distillation flask, electrical shorts, and foaming and spilling of liquid sample through the top opening of the flask.

6.6 Barometer—A pressure measuring device capable of measuring local station pressure with an accuracy of 0.1 kPa (1 mm Hg) or better, at the same elevation relative to sea level as the apparatus in the laboratory. (Warning—Do not take readings from ordinary aneroid barometers, such as those used at weather stations and airports, since these are precorrected to give sea level readings.)

TABLE 1 Group Characteristics

	Group 1	Group 2	Group 3	Group 4
Sample characteristics				
Distillate type				
Vapor pressure at				
37.8 °C, kPa	≥65.5	<65.5	<65.5	<65.5
100 °F, psi	≥9.5	<9.5	<9.5	<9.5
(Test Methods D323, D4953, D5190, D5191, D5842, IP 69 or IP 394)				
Distillation, IBP °C			≤100	>100
°F			≤212	>212
EP °C	≤250	≤250	>250	>250
°F	≤482	≤482	>482	>482

7. Sampling, Storage, and Sample Conditioning

7.1 Determine the Group characteristics that correspond to the sample to be tested (see Table 1). Where the procedure is dependent upon the group, the section headings will be so marked.

7.2 Sampling:

7.2.1 Sampling shall be done in accordance with Practice D4057 or D4177 and as described in Table 2.

7.2.1.1 Group 1—Condition the sample container to below 10 °C, preferably by filling the container with the cold liquid sample and discarding the first sample. If this is not possible because, for instance, the product to be sampled is at ambient temperature, the sample shall be drawn into a container and then discarded, to condition the container, and then refilled in such a manner that agitation is kept at a minimum. Close the container immediately with a tight-fitting closure. (Warning—Do not completely fill and tightly seal a cold container of sample because of the likelihood of expansion and breakage on warming.)

7.2.1.2 Groups 2, 3, and 4—Collect the sample at ambient temperature. After sampling, close the sample container immediately with a tight-fitting closure.

7.2.1.3 If the sample received by the testing laboratory has been sampled by others and it is not known whether sampling has been performed as described in 7.2, the sample shall be assumed to have been so sampled.

7.3 Sample Storage:

7.3.1 If testing is not to start immediately after collection, store the samples as indicated in 7.3.2, 7.3.3, and Table 2. All samples shall be stored away from direct sunlight or sources of direct heat.

7.3.2 Group 1—Store the sample at a temperature below 10 °C.

NOTE 7—If there are no, or inadequate, facilities for storage below 10 °C, the sample may also be stored at a temperature below 20 °C, provided the operator ensures that the sample container is tightly closed and leak-free.

7.3.3 Group 2—Store the sample at a temperature below 10 °C.

NOTE 8—If there are no, or inadequate, facilities for storage below 10 °C, the sample may also be stored at a temperature below 20 °C, provided the operator ensures that the sample container is tightly closed and leak-free.

7.3.4 Groups 3 and 4—Store the sample at ambient or lower temperature.

7.4 Sample Conditioning Prior to Analysis:

7.4.1 Samples shall be conditioned to the temperature shown in Table 2 before opening the sample container.

7.4.1.1 Groups 1 and 2—Samples shall be conditioned to a temperature of less than 10 °C (50 °F) before opening the sample container, except when the sample is to be immediately tested and is already at the prescribed sample temperature in Table 3.

7.4.1.2 Groups 3 and 4—If the sample is not fluid at ambient temperature, it is to be heated to a temperature of 9 °C to 21 °C above its pour point (Test Method D97, D5949, or D5985) prior to analysis. If the sample has partially or completely solidified during storage, it shall be vigorously shaken after melting prior to opening the sample container to ensure homogeneity.

7.4.1.3 If the sample is not fluid at room temperature, the temperature ranges shown in Table 2 for the flask and for the sample do not apply.

7.5 Wet Samples:

7.5.1 Samples of materials that visibly contain water are not suitable for testing. If the sample is not dry, obtain another sample that is free from suspended water.

7.5.2 Groups 1 and 2—If such a sample cannot be obtained, the suspended water can be removed by maintaining the sample at 0 °C to 10 °C, adding approximately 10 g of anhydrous sodium sulfate per 100 mL of sample, shaking the mixture for approximately 2 min, and then allowing the mixture to settle for approximately 15 min. Once the sample shows no visible signs of water, use a decanted portion of the sample, maintained between 1 °C and 10 °C, for the analysis. Note in the report that the sample has been dried by the addition of a desiccant.

NOTE 9—Suspended water in hazy samples in Groups 1 and 2 can be removed by the addition of anhydrous sodium sulfate and separating the liquid sample from the drying agent by decanting without statistically affecting the results of the test.⁶

7.5.3 Groups 3 and 4—In cases in which a water-free sample is not practical, the suspended water can be removed by shaking the sample with anhydrous sodium sulfate or other suitable drying agent and separating it from the drying agent by decanting. Note in the report that the sample has been dried by the addition of a desiccant.

8. Preparation of Apparatus

8.1 Refer to Table 3 and prepare the apparatus by choosing the appropriate distillation flask, temperature measuring device, and flask support board, as directed for the indicated group. Bring the temperature of the receiving cylinder, the flask, and the condenser bath to the indicated temperature.

8.2 Make any necessary provisions so that the temperature of the condenser bath and the receiving cylinder will be

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1455.

TABLE 2 Sampling, Storage, and Sample Conditioning

		Group 1	Group 2	Group 3	Group 4
Temperature of sample container	°C	<10 ^A			
	°F	<50 ^A			
Temperature of stored sample	°C	<10 ^B	<10	ambient	ambient
	°F	<50 ^B	<50	ambient	ambient
Temperature of sample after conditioning prior to analysis	°C	<10 ^C	<10 ^C	Ambient or 9 °C to 21 °C above pour point ^D	Ambient or 9 °C to 21 °C above pour point ^D
	°F	<50	<50	Ambient or 48 °F to 70 °F above pour point ^D	Ambient or 48 °F to 70 °F above pour point ^D
If sample is wet		resample	resample		
If resample is still wet ^E		dry in accordance with 7.5.2	dry in accordance with 7.5.2	dry in accordance with 7.5.3	dry in accordance with 7.5.3

^A If sample is warmer than 10 °C, see 7.2.1.1.

^B Under certain circumstances, samples can also be stored at temperatures below 20 °C (68 °F). See also 7.3.2 and 7.3.3.

^C If sample is to be immediately tested and is already at the temperature prescribed in Table 3, see 7.4.1.1.

^D If sample is (semi)-solid at ambient temperature, see also 10.3.1.1.

^E If sample is known to be wet, resampling may be omitted. Dry sample in accordance with 7.5.2 and 7.5.3.

TABLE 3 Preparation of Apparatus and Specimen

		Group 1	Group 2	Group 3	Group 4
Flask, mL		125	125	125	125
ASTM distillation thermometer		7C (7F)	7C (7F)	7C (7F)	8C (8F)
IP distillation thermometer range		low	low	low	high
Flask support board		B	B	C	C
diameter of hole, mm		38	38	50	50
Temperature at start of test					
Flask	°C	13–18	13–18	13–18	not above ambient
	°F	55–65	55–65	55–65	
Flask support and shield		not above ambient	not above ambient	not above ambient	
Receiving cylinder and sample					
	°C	13–18	13–18	13–18 ^A	13–ambient ^A
	°F	55–65	55–65	55–65 ^A	55–ambient ^A

^A See 10.3.1.1 for exceptions.

maintained at the required temperatures. The receiving cylinder shall be in a bath such that either the liquid level is at least as high as the 100 mL mark or the entire receiving cylinder is surrounded by an air circulation chamber.

8.2.1 *Groups 1, 2, and 3*—Suitable media for low temperature baths include, but are not limited to, chopped ice and water, refrigerated brine, and refrigerated ethylene glycol.

8.2.2 *Group 4*—Suitable media for ambient and higher bath temperatures include, but are not limited to, cold water, hot water, and heated ethylene glycol.

8.3 Remove any residual liquid in the condenser tube by swabbing with a piece of soft, lint-free cloth attached to a cord or wire.

9. Calibration and Standardization

9.1 *Temperature Measurement System*—Temperature measurement systems using other than the specified mercury-in-glass thermometers shall exhibit the same temperature lag, emergent stem effect, and accuracy as the equivalent mercury-in-glass thermometer. Confirmation of the calibration of these temperature measuring systems shall be made at intervals of not more than six months, and after the system has been replaced or repaired.

9.1.1 The accuracy and the calibration of the electronic circuitry or computer algorithms, or both, shall be verified by the use of a standard precision resistance bench. When performing this verification, no algorithms shall be used to correct

the temperature for lag and the emergent stem effect (see manufacturer's instructions).

9.1.2 Verification of the calibration of temperature measuring devices shall be conducted by distilling toluene in accordance with Group 1 of this test method and comparing the 50 % recovered temperature with that shown in Table 4.⁷

9.1.2.1 If the temperature reading is not within the values shown in Table 4 for the respective apparatus being used (see Note 11 and Table 4), the temperature measurement system shall be considered defective and shall not be used for the test.

NOTE 10—Toluene is used as a verification fluid for calibration; it will yield almost no information on how well an electronic measurement system simulates the temperature lag of a liquid-in-glass thermometer.

9.1.2.2 Reagent grade toluene and hexadecane (cetane), conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society,⁸ shall be used. However, other grades may also 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.

⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1580.

⁸ *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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

TABLE 4 True and Min and Max D86 50 % Recovered Boiling Points (°C)^A

		Manual		Automated	
		Distillation conditions min D86 50 % boiling point	Distillation conditions max D86 50 % boiling point	Distillation conditions min D86 50 % boiling point	Distillation conditions max D86 50 % boiling point
Toluene	ASTM/IP true boiling point	Group 1, 2, and 3			
	110.6	105.9	111.8	108.5	109.7
Hexadecane	ASTM/IP true boiling point	Group 4	Group 4	Group 4	Group 4
	287.0	272.2	283.1	277.0	280.0

^A The manual and automated temperatures show in this table are the values for the 95 % tolerance interval for the 99 % population coverage. The proposed tolerance is approximately 3x sigma. Information on the values in this table can be found in RR:D02-1580.

Note 11—At 101.3 kPa, toluene is shown in reference manuals as boiling at 110.6 °C when measured using a partial immersion thermometer. Because this test method uses thermometers calibrated for total immersion, the results typically will be lower and, depending on the thermometer and the situation, may be different for each thermometer. At 101.3 kPa, hexadecane is shown in reference manuals as boiling at 287.0 °C when measured using a partial immersion thermometer. Because this test method uses thermometers calibrated for total immersion, the results typically will be lower, and, depending on the thermometer and the situation, may be different for each thermometer.

9.1.3 A procedure to determine the magnitude of the temperature lag is described in [Annex A3](#).

9.1.4 A procedure to emulate the emergent stem effect is described in [Appendix X4](#).

9.1.5 To verify the calibration of the temperature measurement system at elevated temperatures, use hexadecane. The temperature measurement system shall indicate, at 50 % recovered, a temperature comparable to that shown in [Table 4](#) for the respective apparatus under Group 4 distillation conditions.

Note 12—Because of the high melting point of hexadecane, Group 4 verification distillations will have to be carried out with condenser temperatures >20 °C.

9.2 Automated Method:

9.2.1 *Level Follower*—For an automated distillation apparatus, the level follower/recording mechanism of the apparatus shall have a resolution of 0.1 % volume or better with a maximum error of 0.3 % volume between the 5 % and 100 % volume points. The calibration of the assembly shall be verified in accordance with manufacturer’s instructions at intervals of not more than three months and after the system has been replaced or repaired.

Note 13—The typical calibration procedure involves verifying the output with the receiver containing 5 % and 100 % volume of material respectively.

9.2.2 *Barometric Pressure*—At intervals of not more than six months, and after the system has been replaced or repaired, the barometric reading of the instrument shall be verified against a barometer, as described in [6.6](#).

10. Procedure

10.1 Record the prevailing barometric pressure.

10.2 *Groups 1 and 2*—Ensure that the sample is conditioned in accordance with [Table 2](#). Fit a low range thermometer

provided with a snug-fitting cork or stopper of silicone rubber, or equivalent polymeric material, tightly into the neck of the sample container and bring the temperature of the sample to the temperature indicated in [Table 3](#).

10.3 *Groups 1, 2, 3, and 4*—Check that the temperature of the sample is as shown in [Table 3](#). Pour the specimen precisely to the 100 mL mark of the receiving cylinder, and transfer the contents of the receiving cylinder as completely as practical into the distillation flask, ensuring that none of the liquid flows into the vapor tube.

Note 14—It is important that the difference between the temperature of the specimen and the temperature of the bath around the receiving cylinder is as small as practically possible. A difference of 5 °C can make a difference of 0.7 mL.

10.3.1 *Groups 3 and 4*—If the sample is not fluid at ambient temperature, it is to be heated to a temperature between 9 °C and 21 °C above its pour point (Test Methods [D97](#), [D5949](#), [D5950](#), or [D5985](#)) prior to analysis. If the sample has partially or completely solidified in the intervening period, it shall be vigorously shaken after melting, and prior to sampling, to ensure homogeneity.

10.3.1.1 If the sample is not fluid at ambient temperatures, disregard the temperature range shown in [Table 3](#) for the receiving cylinder and sample. Prior to analysis, heat the receiving cylinder to approximately the same temperature as the sample. Pour the heated specimen precisely to the 100 mL mark of the receiving cylinder, and transfer the contents of the receiving cylinder as completely as practical into the distillation flask, ensuring that none of the liquid flows into the vapor tube.

Note 15—Any material that evaporates during the transfer will contribute to the loss; any material that remains in the receiving cylinder will contribute to the observed recovery volume at the time of the IBP.

10.4 If the sample can be expected to demonstrate irregular boiling behavior, that is, bumping, add a few boiling chips to the specimen. The addition of a few boiling chips is acceptable for any distillation.

10.5 Fit the temperature sensor through a snug-fitting device, as described in [6.4](#), to mechanically center the sensor in the neck of the flask. In the case of a thermometer, the bulb is centered in the neck and the lower end of the capillary is level with the highest point on the bottom of the inner wall of the

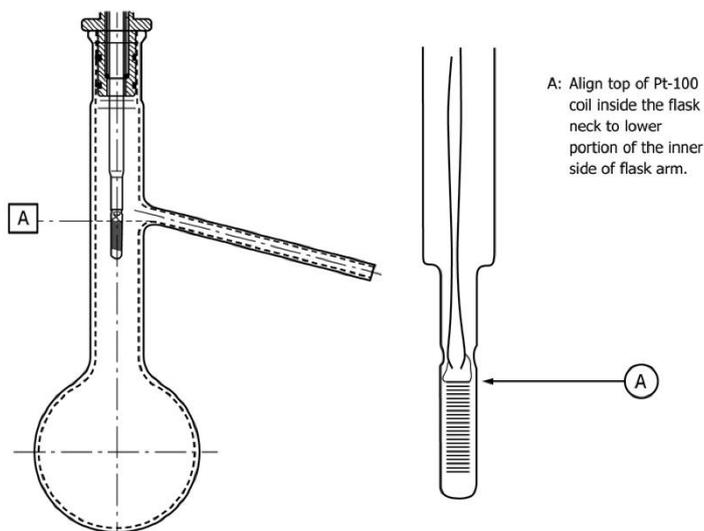


FIG. 6 Example of One Manufacturer's Recommended Placement of Pt-100 Probe Relative to Distillation Flask Sidearm for Automated D86 Distillation Instrument

vapor tube (see Fig. 5). In the case of a thermocouple or resistance thermometer, follow the manufacturer's instructions as to placement (see Fig. 6).

NOTE 16—If vacuum grease is used on the mating surface of the centering device, use the minimum amount of grease that is practical.

10.6 Fit the flask vapor tube, provided with a snug-fitting cork or rubber stopper of silicone, or equivalent polymeric material, tightly into the condenser tube. Adjust the flask in a vertical position so that the vapor tube extends into the condenser tube for a distance from 25 mm to 50 mm. Raise and adjust the flask support board to fit it snugly against the bottom of the flask.

10.7 Place the receiving cylinder that was used to measure the specimen, without drying the inside of the cylinder, into its temperature-controlled bath under the lower end of the condenser tube. The end of the condenser tube shall be centered in the receiving cylinder and shall extend therein for a distance of at least 25 mm, but not below the 100 mL mark.

10.8 Initial Boiling Point:

10.8.1 Manual Method—To reduce evaporation loss of the distillate, cover the receiving cylinder with a piece of blotting paper, or similar material, that has been cut to fit the condenser tube snugly. If a receiver deflector is being used, start the distillation with the tip of the deflector just touching the wall of the receiving cylinder. If a receiver deflector is not used, keep the drip tip of the condenser away from the wall of the receiving cylinder. Note the start time. Observe and record the IBP to the nearest 0.5 °C (1.0 °F). If a receiver deflector is not

being used, immediately move the receiving cylinder so that the tip of the condenser touches its inner wall.

10.8.2 Automated Method—To reduce evaporation loss of the distillate, use the device provided by the instrument manufacturer for this purpose. Apply heat to the distillation flask and contents with the tip of the receiver deflector just touching the wall of the receiving cylinder. Note the start time. Record the IBP to the nearest 0.1 °C (0.2 °F).

10.9 Regulate the heating so that the time interval between the first application of heat and the IBP is as specified in Table 5.

10.10 Regulate the heating so that the time from IBP to 5% recovered is as indicated in Table 5.

10.11 Continue to regulate the heating so that the uniform average rate of condensation from 5% recovered to 5 mL residue in the flask is 4 mL to 5 mL per minute. (Warning—Due to the configuration of the boiling flask and the conditions of the test, the vapor and liquid around the temperature sensor are not in thermodynamic equilibrium. The distillation rate will consequently have an effect on the measured vapor temperature. The distillation rate shall, therefore, be kept as constant as possible throughout the test.)

10.11.1 In the context of this test method, "uniform average rate of condensation" has the following intention. Heating of the boiling flask shall be regulated to maintain as best as possible a uniform flow of condensation, which will then provide the most desired precision for the test. However, some distillation tests can have one or more short-term rates of

TABLE 5 Conditions During Test Procedure

		Group 1	Group 2	Group 3	Group 4
Temperature of cooling bath ^A	°C	0–1	0–5	0–5	0–60
	°F	32–34	32–40	32–40	32–140
Temperature of bath around receiving cylinder	°C	13–18	13–18	13–18	±3
	°F	55–65	55–65	55–65	±5 of charge temperature
Time from first application of heat to initial boiling point, min		5–10	5–10	5–10	5–15
Time from initial boiling point to 5 % recovered, s		60–100	60–100		
Uniform average rate of condensation from 5 % recovered to 5 mL in flask, mL/min		4–5	4–5	4–5	4–5
Time recorded from 5 mL residue to end point, min		5 max	5 max	5 max	5 max

^A The proper condenser bath temperature will depend upon the wax content of the sample and of its distillation fractions. The test is generally performed using one single condenser temperature. Wax formation in the condenser can be deduced from (a) the presence of wax particles in the distillate coming off the drip tip, (b) a higher distillation loss than what would be expected based on the initial boiling point of the specimen, (c) an erratic recovery rate and (d) the presence of wax particles during the removal of residual liquid by swabbing with a lint-free cloth (see 8.3). The minimum temperature that permits satisfactory operation shall be used. In general, a bath temperature in the 0 °C to 4 °C range is suitable for kerosine, Grade No. 1 fuel oil and Grade No. 1-D diesel fuel oil. In some cases involving Grade No. 2 fuel oil, Grade No. 2-D diesel fuel oil, gas oils and similar distillates, it may be necessary to hold the condenser bath temperature in the 38 °C to 60 °C range.

condensation which deviate from the 4 mL/min to 5 mL/min indicated in 10.11 and Table 5, this is a common occurrence for some sample types. The periods of these short-term deviations may last for several percent of material condensed until the temperature slope becomes constant again, and may occur at several periods along the entire condensation range. These deviations will typically correct after the temperature slope again becomes constant. These short-term deviations shall not occur over the entire range of condensation. Typically, these short-term deviations should not occur for more than ten contiguous percent volume. The precision of the temperature readings will be significantly affected during these periods. When the overall calculated average rate of condensation between 5 % recovered and 5 mL residue is within the prescribed rate, the requirement of 10.11 and Table 5 is satisfied. As example, those samples containing a 10 % ethanol-fuel blend or those that exhibit a significant change of temperature slope at points during the distillation can have a short-term rate of condensation which deviates from the 4 mL/min to 5 mL/min indicated in 10.11 and Table 5.

NOTE 17—When testing gasoline samples, it is not uncommon to see the condensate suddenly form non-miscible liquid phases and bead up on the temperature measuring device and in the neck of the boiling flask at a vapor temperature of around 160 °C. This may be accompanied by a sharp (about 3 °C) dip in the vapor temperature and a drop in the recovery rate. The phenomenon, which may be due to the presence of trace water in the sample, may last for 10 s to 30 s before the temperature recovers and the condensate starts flowing smoothly again. This point is sometimes colloquially referred to as the Hesitation Point.

10.12 Repeat any distillation that did not meet the requirements described in 10.9, 10.10, and 10.11.

10.13 If a decomposition point is observed, discontinue the heating and proceed as directed in 10.17.

NOTE 18—Characteristic indications of thermal decomposition are evolution of fumes and erratic, typically decreasing, temperature readings that occur during the final stages of the distillation.

10.14 In the interval between the IBP and the end of the distillation, observe and record data necessary for the calculation and reporting of the results of the test as required by the specification involved, or as previously established for the sample under test. These observed data can include temperature readings at prescribed percentages recovered or percentages recovered at prescribed temperature readings, or both.

10.14.1 *Manual Method*—Record all volumes in the graduated cylinder to the nearest 0.5 mL, and all temperature readings to the nearest 0.5 °C (1.0 °F).

10.14.2 *Automated Method*—Record all volumes in the receiving cylinder to the nearest 0.1 mL, and all temperature readings to the nearest 0.1 °C (0.2 °F).

10.14.3 *Group 1, 2, 3, and 4*—In cases in which no specific data requirements have been indicated, record the IBP and the EP (FBP) or the dry point, or both, and temperature readings at 5 %, 15 %, 85 %, and 95 % recovered, and at each 10 % multiple of volume recovered from 10 to 90, inclusive.

10.14.3.1 *Group 4*—When a high range thermometer is used in testing aviation turbine fuels and similar products, pertinent thermometer readings can be obscured by the centering device. If these readings are required, perform a second distillation in accordance with Group 3. In such cases, reading from a low range thermometer can be reported in place of the obscured high range thermometer readings, and the test report shall so indicate. If, by agreement, the obscured readings are waived, the test report shall so indicate.

10.14.4 When it is required to report the temperature reading at a prescribed percent evaporated or recovered for a sample that has a rapidly changing slope of the distillation curve in the region of the prescribed percent evaporated or recovered reading, record temperature readings at every 1 % recovered. The slope is considered rapidly changing if the change in slope (*C*) of the data points described in 10.14.2 in that particular area is greater than 0.6 (change of slope (*F*) is greater than 1.0) as calculated by Eq 1 (Eq 2).

$$\text{Change of Slope } (C) = \frac{(C_2 - C_1)(V_2 - V_1) - (C_3 - C_2)(V_3 - V_2)}{(V_2 - V_1)(V_3 - V_2)} \quad (1)$$

Change of Slope (F) = (2)

$$(F_2 - F_1)/(V_2 - V_1) - (F_3 - F_2)/(V_3 - V_2)$$

where:

- C_1 = temperature at the volume % recorded one reading prior to the volume % in question, °C,
- C_2 = temperature at the volume % recorded in question, °C,
- C_3 = temperature at the volume % recorded following the volume % in question, °C,
- F_1 = temperature at the volume % recorded one reading prior to the volume % in question, °F,
- F_2 = temperature at the volume % recorded in question, °F,
- F_3 = temperature at the volume % recorded following the volume % in question, °F,
- V_1 = volume % recorded one reading prior to the volume % in question,
- V_2 = volume % recorded at the volume % in question, and
- V_3 = volume % recorded following the volume % in question.

10.15 When the residual liquid in the flask is approximately 5 mL, make a final adjustment of the heat. The time from the 5 mL of liquid residue in the flask to the EP (FBP) shall be within the limits prescribed in Table 5. If this condition is not satisfied, repeat the test with appropriate modification of the final heat adjustment.

NOTE 19—Since it is difficult to determine when there is 5 mL of boiling liquid left in the flask, this time is determined by observing the amount of liquid recovered in the receiving cylinder. The dynamic holdup has been determined to be approximately 1.5 mL at this point. If there are no front end losses, the amount of 5 mL in the flask can be assumed to correspond with an amount of 93.5 mL in the receiving cylinder. This amount has to be adjusted for the estimated amount of front end loss.

10.15.1 If the actual front end loss differs more than 2 mL from the estimated value, the test shall be rerun.

10.16 Observe and record the EP (FBP) or the dry point, or both, as required, and discontinue the heating.

NOTE 20—The end point (final boiling point), rather than the dry point, is intended for general use. The dry point can be reported in connection with special purpose naphthas, such as those used in the paint industry. Also, it is substituted for the end point (final boiling point) whenever the sample is of such a nature that the precision of the end point (final boiling point) cannot consistently meet the requirements given in the precision section.

NOTE 21—Groups 1 and 2, once the final heat adjustment is made, the vapor temperature/thermometer reading will continue to increase. As the distillation nears the end point (final boiling point) the distillation typically achieves dry point first. After the dry point has been achieved the vapor temperature/thermometer reading should continue to increase. The bottom of the flask will be dry but the sides and neck of the flask and the temperature sensor will still have vapor condensate present. The vapor condensate may have the appearance of a white cloud of fumes. This vapor condensate/cloud of fumes should totally engulf the temperature-measuring sensor before the vapor temperature starts to decrease. If these observations do not occur, the end point may not have been reached. It would be advisable to repeat the test adding additional heat to the final heat adjustment. Typically the vapor temperature will continue to rise as the dry point is reached and the vapor cloud engulfs the temperature-measuring sensor. When the end point is near, the rate of temperature increase will slow and level off. Once the endpoint is reached the vapor

temperature will start and continue to decrease. If the vapor temperature starts to decrease but then increases and repeats this cycle while the vapor temperature continues to increase you have added too much heat to the final heat adjustment. If this is the case, it would be advisable to repeat the test lowering final heat setting.

Groups 3 and 4, many Group 3 and 4 samples will have the same distillation characteristics in regards to dry point and endpoint as Groups 1 and 2. With samples that contain higher temperature boiling materials it may not be possible to detect a dry point or an end point before the decomposition point occurs.

10.17 Allow the distillate to drain into the receiving cylinder, after heating has been discontinued.

10.17.1 *Manual Method*—While the condenser tube continues to drain into the graduated cylinder, observe and note the volume of condensate to the nearest 0.5 mL at 2 min intervals until two successive observations agree. Measure the volume in the receiving cylinder accurately, and record it to the nearest 0.5 mL.

10.17.2 *Automated Method*—The apparatus shall continually monitor the recovered volume until this volume changes by no more than 0.1 mL in 2 min. Record the volume in the receiving cylinder accurately to the nearest 0.1 mL.

10.18 Record the volume in the receiving cylinder as percent recovery. If the distillation was previously discontinued under the conditions of a decomposition point, deduct the percent recovered from 100, report this difference as the sum of percent residue and percent loss, and omit the procedure given in 10.19.

10.19 After the flask has cooled and no more vapor is observed, disconnect the flask from the condenser, pour its contents into a 5 mL graduated cylinder, and with the flask suspended over the cylinder, allow the flask to drain until no appreciable increase in the volume of liquid in the cylinder is observed. Measure the volume in the graduated cylinder to the nearest 0.1 mL, and record as percent residue.

10.19.1 If the 5 mL graduated cylinder does not have graduations below 1 mL and the volume of liquid is less than 1 mL, refill the cylinder with 1 mL of a heavy oil to allow a better estimate of the volume of the material recovered.

10.19.1.1 If a residue greater than expected is obtained, and the distillation was not purposely terminated before the EP, check whether adequate heat was applied towards the end of the distillation and whether conditions during the test conformed to those specified in Table 5. If not, repeat test.

NOTE 22—The distillation residues of this test method for gasoline, kerosine, and distillate diesel are typically 0.9 % to 1.2 %, 0.9 % to 1.3 %, and 1.0 % to 1.4 % volume, respectively.

NOTE 23—The test method is not designed for the analysis of distillate fuels containing appreciable quantities of residual material (see 1.2).

10.19.2 *Groups 1, 2, 3, and 4*—Record the volume in the 5 mL graduated cylinder, to the nearest 0.1 mL, as percent residue.

10.20 If the intent of the distillation is to determine the percent evaporated or percent recovered at a predetermined corrected temperature reading, modify the procedure to conform to the instructions described in Annex A4.

10.21 Examine the condenser tube and the side arm of the flask for waxy or solid deposits. If found, repeat the test after making adjustments described in Footnote A of Table 5.

TABLE 6 Approximate Thermometer Reading Correction

Temperature Range		Correction ^A per 1.3 kPa (10 mm Hg) Difference in Pressure	
°C	°F	°C	°F
10–30	50–86	0.35	0.63
30–50	86–122	0.38	0.68
50–70	122–158	0.40	0.72
70–90	158–194	0.42	0.76
90–110	194–230	0.45	0.81
110–130	230–266	0.47	0.85
130–150	266–302	0.50	0.89
150–170	302–338	0.52	0.94
170–190	338–374	0.54	0.98
190–210	374–410	0.57	1.02
210–230	410–446	0.59	1.07
230–250	446–482	0.62	1.11
250–270	482–518	0.64	1.15
270–290	518–554	0.66	1.20
290–310	554–590	0.69	1.24
310–330	590–626	0.71	1.28
330–350	626–662	0.74	1.33
350–370	662–698	0.76	1.37
370–390	698–734	0.78	1.41
390–410	734–770	0.81	1.46

^A Values to be added when barometric pressure is below 101.3 kPa (760 mm Hg) and to be subtracted when barometric pressure is above 101.3 kPa.

11. Calculations

11.1 The percent total recovery is the sum of the percent recovery (see 10.18) and the percent residue (see 10.19). Deduct the percent total recovery from 100 to obtain the percent loss.

11.2 Do not correct the barometric pressure for meniscus depression, and do not adjust the pressure to what it would be at sea level.

NOTE 24—The observed barometric reading does not have to be corrected to a standard temperature and to standard gravity. Even without performing these corrections, the corrected temperature readings for the same sample between laboratories at two different locations in the world will, in general, differ less than 0.1 °C at 100 °C. Almost all data obtained earlier have been reported at barometric pressures that have not been corrected to standard temperature and to standard gravity.

11.3 Correct temperature readings to 101.3 kPa (760 mm Hg) pressure. Obtain the correction to be applied to each temperature reading by means of the Sydney Young equation as given in Eq 3, Eq 4, or Eq 5, as appropriate, or by the use of Table 6. For Celsius temperatures:

$$C_c = 0.0009 (101.3 - P_k) (273 + t_c) \tag{3}$$

$$C_c = 0.00012 (760 - P) (273 + t_c) \tag{4}$$

For Fahrenheit temperatures:

$$C_f = 0.00012 (760 - P) (460 + t_f) \tag{5}$$

where:

- t_c = the observed temperature reading in °C,
- t_f = the observed temperature reading in °F,
- C_c and C_f = corrections to be added algebraically to the observed temperature readings,
- P_k = barometric pressure, prevailing at the time and location of the test, kPa, and
- P = barometric pressure, prevailing at the time and location of the test, mm Hg.

After applying the corrections and rounding each result to the nearest 0.5 °C (1.0 °F) or 0.1 °C (0.2 °F), as appropriate to the apparatus being used, use the corrected temperature readings in all further calculations and reporting.

NOTE 25—Temperature readings are not corrected to 101.3 kPa (760 mm Hg) when product definitions, specifications, or agreements between the parties involved indicate, specifically, that such correction is not required or that correction shall be made to some other base pressure.

11.4 Correct the actual loss to 101.3 kPa (760 mm Hg) pressure when temperature readings are corrected to 101.3 kPa pressure. The corrected loss, L_c , is calculated from Eq 6 or Eq 7, as appropriate, or can be read from the tables presented as Fig. X3.1 or Fig. X3.2.

$$L_c = 0.5 + (L - 0.5) / \{1 + (101.3 - P_k) / 8.00\} \tag{6}$$

$$L_c = 0.5 + (L - 0.5) / \{1 + (760 - P) / 60.0\} \tag{7}$$

where:

- L = observed loss,
- L_c = corrected loss,
- P_k = pressure, kPa, and
- P = pressure, mm Hg.

NOTE 26—Eq 6 and 7 above have been derived from the data in Table A4.3 and Eqs 5 and 6 in Test Method D86 – 95 and earlier versions. It is probable that Eq 6 and 7 shown were the original empirical equations from which the table and equations in the Test Method D86 – 95 and earlier versions were derived.

11.4.1 Calculate the corresponding corrected percent recovery in accordance with the following equation:

$$R_c = R + (L - L_c) \tag{8}$$

where:

- L = percent loss or observed loss,
- L_c = corrected loss,
- R = percent recovery, and
- R_c = corrected percent recovery.

11.5 To obtain the percent evaporated at a prescribed temperature reading, add the percent loss to each of the observed percent recovered at the prescribed temperature readings, and report these results as the respective percent evaporated, that is:

$$P_e = P_r + L \tag{9}$$

where:

- L = observed loss,
- P_e = percent evaporated, and
- P_r = percent recovered.

11.6 To obtain temperature readings at prescribed percent evaporated, and if no recorded temperature data is available within 0.1 volume % of the prescribed percent evaporated, use either of the two following procedures, and indicate on the report whether the arithmetical procedure or the graphical procedure has been used.

11.6.1 *Arithmetical Procedure*—Deduct the observed loss from each prescribed percent evaporated to obtain the corresponding percent recovered. Calculate each required temperature reading as follows:

$$T = T_L + (T_H - T_L) (P_r - P_{rL}) / (P_{rH} - P_{rL}) \tag{10}$$

where:

- P_r = percent recovered corresponding to the prescribed percent evaporated,
 P_{rH} = percent recovered adjacent to, and higher than P_r ,
 P_{rL} = percent recovered adjacent to, and lower than P_r ,
 T = temperature reading at the prescribed percent evaporated,
 T_H = temperature reading recorded at P_{rH} , and
 T_L = temperature reading recorded at P_{rL} .

Values obtained by the arithmetical procedure are affected by the extent to which the distillation graphs are nonlinear. Intervals between successive data points can, at any stage of the test, be no wider than the intervals indicated in 10.18. In no case shall a calculation be made that involves extrapolation.

11.6.2 *Graphical Procedure*—Using graph paper with uniform subdivisions, plot each temperature reading corrected for barometric pressure, if required (see 11.3), against its corresponding percent recovered. Plot the IBP at 0 % recovered. Draw a smooth curve connecting the points. For each prescribed percent evaporated, deduct the distillation loss to obtain the corresponding percent recovered and take from the graph the temperature reading that this percent recovered indicates. Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

NOTE 27—See Appendix X1 for numerical examples illustrating the arithmetical procedure.

11.6.3 In most automated instruments, temperature-volume data are collected at 0.1 volume % intervals or less and stored in memory. To report a temperature reading at a prescribed percent evaporated, neither of the procedures described in 11.6.1 and 11.6.2 have to be used. Obtain the desired temperature directly from the database as the temperature closest to and within 0.1 % volume of the prescribed percent evaporated.

12. Report

12.1 Report the following information (see Appendix X5 for examples of reports):

12.2 Report the barometric pressure to the nearest 0.1 kPa (1 mm Hg).

12.3 Report all volumetric readings in percentages.

12.3.1 *Manual Method*—Report volumetric readings to the nearest 0.5, and all temperature readings to the nearest 0.5° C (1.0 °F).

12.3.2 *Automated Method*—Report volumetric readings to the nearest 0.1, and all temperature readings to the nearest one tenth degree.

12.4 After barometric corrections of the temperature readings have been made, the following data require no further calculation prior to reporting: IBP, dry point, EP (FBP), decomposition point, and all pairs of corresponding values involving percent recovered and temperature readings.

12.4.1 The report shall state if the temperature readings have not been corrected for barometric pressure.

12.5 When the temperature readings have not been corrected to 101.3 kPa (760 mm Hg) pressure, report the percent

residue and percent loss as *observed* in accordance with 10.19 and 11.1, respectively.

12.6 Do not use the corrected loss in the calculation of percent evaporated.

12.7 It is advisable to base the report on relationships between temperature readings and percent evaporated when the sample is a gasoline, or any other product classified under Group 1, or in which the percent loss is greater than 2.0. Otherwise, the report can be based on relationships between temperature readings and percent evaporated or percent recovered. Every report must indicate clearly which basis has been used.

12.7.1 In the manual method, if results are given in percent evaporated versus temperature readings, report if the arithmetical or the graphical procedure was used (see 11.6).

12.8 Report if a drying agent, as described in 7.5.2 or 7.5.3, was used.

12.9 Fig. X1.1 is an example of a tabular report. It shows the percent recovered versus the corresponding temperature reading and versus the corrected temperature reading. It also shows the percent loss, the corrected loss, and the percent evaporated versus the corrected temperature reading.

13. Precision and Bias

13.1 *Precision (Group 1, 2, 3 automated)*—The precision of this test method, as determined by the statistical examination of the interlaboratory test results,⁹ is as follows:

NOTE 28—The precision was derived from data produced by automated D86 apparatus. Typical examples of precision for manual apparatus can be calculated from the information contained in Annex A4 (see A4.10).

NOTE 29—Information on the precision of percent evaporated or percent recovered at a prescribed temperature can be found in Annex A4.

NOTE 30—For naphthas, solvents, and other similar materials where percent recovered are reported and the percent loss is typically less than one percent, the percent recovered temperatures can be considered identical to the percent evaporated temperatures and precision can be calculated as shown for Group 1, 2, 3.

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, would in the long run, in the normal and correct operation of this test method, exceed the values in Table 7 only in one case in twenty.

13.1.2 *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 normal and correct operation of this test method, exceed the values in Table 7 only in one case in twenty.

13.1.3 The precision statements were derived from a 2010 interlaboratory cooperative test program.⁹ Twenty six laboratories participated and analyzed twenty one sample sets comprised of; specification grade gasoline, both conventional and

⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1807. Contact ASTM Customer Service at service@astm.org.

TABLE 7 Repeatability and Reproducibility for Group 1, 2, 3 (Automated)
(Valid Range 20 °C to 260 °C)

Percent Evaporated	Repeatability °C	Reproducibility °C
IBP	2.7	4.7
5	1.4 + 2.8(0.43Sc + 0.24)	2.5 + 2.8(0.43Sc + 0.24)
10	0.9 + 2.8(0.43Sc + 0.24)	1.9 + 2.8(0.43Sc + 0.24)
20	0.9 + 2.8(0.43Sc + 0.24)	2.0 + 2.8(0.43Sc + 0.24)
30	0.8 + 2.8(0.43Sc + 0.24)	1.8 + 2.8(0.43Sc + 0.24)
40	0.9 + 2.8(0.43Sc + 0.24)	2.0 + 2.8(0.43Sc + 0.24)
50	1.0 + 2.8(0.43Sc + 0.24)	1.9 + 2.8(0.43Sc + 0.24)
60	1.1 + 2.8(0.43Sc + 0.24)	2.0 + 2.8(0.43Sc + 0.24)
70	1.5 + 2.8(0.43Sc + 0.24)	2.1 + 2.8(0.43Sc + 0.24)
80	1.1 + 2.8(0.43Sc + 0.24)	2.0 + 2.8(0.43Sc + 0.24)
90	1.8 + 2.8(0.43Sc + 0.24)	2.8 + 2.8(0.43Sc + 0.24)
95	2.0 + 2.8(0.43Sc + 0.24)	3.6 + 2.8(0.43Sc + 0.24)
FBP	3.3	7.1

where:
Sc = slope or rate of change of temperature in degrees Celcius calculated using A4.10.1.

TABLE 8 Repeatability and Reproducibility for Group 4 (Automated)^A

Percent Recovered	Repeatability °C	Reproducibility °C	Valid Range °C
IBP	0.018T	0.055T	145 to 220
5 %	0.0109T	0.03T	160 to 255
10 %	0.0094T	0.022T	160 to 265
20 %	0.00728T	0.0208T	175 to 275
30 %	0.00582T	0.0165T	185 to 285
40 %	0.005T	0.014T	195 to 290
50 %	1.0	3.0	170 to 295
60 %	0.00357T	0.0117T	220 to 305
70 %	0.00355T	0.0125T	230 to 315
80 %	0.00377T	0.0136T	240 to 325
90 %	0.0041T	0.015T	180 to 340
95 %	0.01318(T-140)	0.04105(T-140)	260 to 360
FBP	2.2	7.1	195 to 365

where:
T = percent recovered temperature within valid range prescribed.
^A Refer to Annex A1 for tables of calculated repeatability and reproducibility.

oxygenated, some containing up to 20 % ethanol. The temperature range covered was 20 °C to 220 °C. Information on the type of samples and their average boiling points are in the research report.

13.2 Precision (Group 4)—The precision of this test method, as determined by the statistical examination of the interlaboratory test results,¹⁰ is as follows:

NOTE 31—Information on the precision of percent evaporated or percent recovered at a prescribed temperature can be found in Annex A4.

13.2.1 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 the following values in Table 8 only in one case in twenty.

13.2.2 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 normal and correct operation of this test method, exceed the following values in Table 8 only in one case in twenty.

¹⁰ Supporting data (results of the 2005 Interlaboratory Cooperative Test Program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1621.

13.2.3 The precision statements were derived from a 2005 interlaboratory cooperative test program.¹⁰ Sixteen laboratories participated and analyzed sample sets comprised of; specification grade diesel, with a B5 and B20 biodiesel, specification grade heating oil, aviation turbine fuels, marine fuels, mineral spirits and toluene. The temperature range covered was 145 °C to 365 °C. Information on the type of samples and their average boiling points are in the research report.

13.3 Bias:

13.3.1 Bias—Since there is no accepted reference material suitable for determining the bias for the procedure in these test methods, bias has not been determined.

13.3.2 Relative Bias between Manual and Automated Apparatus—An interlaboratory study⁷ conducted in 2003 using manual and automated apparatus has concluded that there is no statistical evidence to suggest that there is a bias between manual and automated results.

NOTE 32—See A2.1 for information on the application and use of borosilicate and quartz distillation flasks.

14. Keywords

14.1 batch distillation; distillates; distillation; laboratory distillation; petroleum products

ANNEXES

(Mandatory Information)

A1. PRECISION TABLES FOR REPEATABILITY (r) AND REPRODUCIBILITY (R)

A1.1 Tables:

Recovered IBP Temperature (°C)	IBP_GRP4		250	2.35	5.50
	r_D86auto	R_D86auto			
145	2.61	7.98	255	2.40	5.61
150	2.70	8.25	260	2.44	5.72
155	2.79	8.53	265	2.49	5.83
160	2.88	8.80			
165	2.97	9.08			
170	3.06	9.35			
175	3.15	9.63			
180	3.24	9.90			
185	3.33	10.18			
190	3.42	10.45			
195	3.51	10.73			
200	3.60	11.00			
205	3.69	11.28			
210	3.78	11.55			
215	3.87	11.83			
220	3.96	12.10			
Recovered 20 % Temperature (°C)	T20_GRP4		250	2.35	5.50
	r_D86auto	R_D86auto			
175	1.27	3.64	255	1.27	3.64
180	1.31	3.74	260	1.31	3.74
185	1.35	3.85	265	1.35	3.85
190	1.38	3.95			
195	1.42	4.06			
200	1.46	4.16			
205	1.49	4.26			
210	1.53	4.37			
215	1.57	4.47			
220	1.60	4.58			
225	1.64	4.68			
230	1.67	4.78			
235	1.71	4.89			
240	1.75	4.99			
245	1.78	5.10			
250	1.82	5.20			
255	1.86	5.30			
Recovered 5 % Temperature (°C)	T5_GRP4		250	2.35	5.50
	r_D86auto	R_D86auto			
160	1.74	4.80	255	1.89	5.41
165	1.80	4.95	260	1.93	5.51
170	1.85	5.10	265	1.97	5.62
175	1.91	5.25	270	2.00	5.72
180	1.96	5.40			
185	2.02	5.55			
190	2.07	5.70			
195	2.13	5.85			
200	2.18	6.00			
205	2.23	6.15			
210	2.29	6.30			
215	2.34	6.45			
220	2.40	6.60			
225	2.45	6.75			
230	2.51	6.90			
235	2.56	7.05			
240	2.62	7.20			
245	2.67	7.35			
250	2.73	7.50			
255	2.78	7.65			
Recovered 30 % Temperature (°C)	T30_GRP4		250	2.35	5.50
	r_D86auto	R_D86auto			
185	1.08	3.05	255	1.11	3.14
190	1.11	3.14	260	1.13	3.22
195	1.13	3.22	265	1.16	3.30
200	1.16	3.30	270	1.19	3.38
205	1.19	3.38			
210	1.22	3.47			
215	1.25	3.55			
220	1.28	3.63			
225	1.31	3.71			
230	1.34	3.80			
235	1.37	3.88			
240	1.40	3.96			
245	1.43	4.04			
250	1.46	4.13			
255	1.48	4.21			
Recovered 10 % Temperature (°C)	T10_GRP4		250	2.35	5.50
	r_D86auto	R_D86auto			
160	1.50	3.52	255	1.54	4.37
165	1.55	3.63	260	1.57	4.46
170	1.60	3.74	265	1.60	4.54
175	1.65	3.85	270	1.63	4.62
180	1.69	3.96	275	1.66	4.70
185	1.74	4.07			
190	1.79	4.18			
195	1.83	4.29			
200	1.88	4.40			
205	1.93	4.51			
210	1.97	4.62			
215	2.02	4.73			
220	2.07	4.84			
225	2.12	4.95			
230	2.16	5.06			
235	2.21	5.17			
240	2.26	5.28			
245	2.30	5.39			
Recovered 40 % Temperature (°C)	T40_GRP4		250	2.35	5.50
	r_D86auto	R_D86auto			
195	0.98	2.73	255	1.00	2.80
200	1.00	2.80	260	1.03	2.87
205	1.03	2.87	265	1.05	2.94
210	1.05	2.94	270	1.08	3.01
215	1.08	3.01	275	1.10	3.08
220	1.10	3.08			
225	1.13	3.15			
230	1.15	3.22			
235	1.18	3.29			
240	1.20	3.36			
245	1.23	3.43			
250	1.25	3.50			

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255	1.28	3.57	310	1.17	4.22
260	1.30	3.64	315	1.19	4.28
265	1.33	3.71	320	1.21	4.35
270	1.35	3.78	325	1.23	4.42
275	1.38	3.85			
280	1.40	3.92	Recovered 90 %		
285	1.43	3.99	Temperature (°C)		
290	1.45	4.06	180	0.74	2.70
			185	0.76	2.78
Recovered 50 %			190	0.78	2.85
Temperature (°C)			195	0.80	2.93
170–295			200	0.82	3.00
			205	0.84	3.08
Recovered 60 %			210	0.86	3.15
Temperature (°C)			215	0.88	3.23
220	0.79	2.57	220	0.90	3.30
225	0.80	2.63	225	0.92	3.38
230	0.82	2.69	230	0.94	3.45
235	0.84	2.75	235	0.96	3.53
240	0.86	2.81	240	0.98	3.60
245	0.87	2.87	245	1.00	3.68
250	0.89	2.93	250	1.03	3.75
255	0.91	2.98	255	1.05	3.83
260	0.93	3.04	260	1.07	3.90
265	0.95	3.10	265	1.09	3.98
270	0.96	3.16	270	1.11	4.05
275	0.98	3.22	275	1.13	4.13
280	1.00	3.28	280	1.15	4.20
285	1.02	3.33	285	1.17	4.28
290	1.04	3.39	290	1.19	4.35
295	1.05	3.45	295	1.21	4.43
300	1.07	3.51	300	1.23	4.50
305	1.09	3.57	305	1.25	4.58
			310	1.27	4.65
Recovered 70 %			315	1.29	4.73
Temperature (°C)			320	1.31	4.80
230	0.82	2.88	325	1.33	4.88
235	0.83	2.94	330	1.35	4.95
240	0.85	3.00	335	1.37	5.03
245	0.87	3.06	340	1.39	5.10
250	0.89	3.13			
255	0.91	3.19			
260	0.92	3.25	Recovered 95 %		
265	0.94	3.31	Temperature (°C)		
270	0.96	3.38	260	1.58	4.93
275	0.98	3.44	265	1.65	5.13
280	0.99	3.50	270	1.71	5.34
285	1.01	3.56	275	1.78	5.54
290	1.03	3.63	280	1.85	5.75
295	1.05	3.69	285	1.91	5.95
300	1.07	3.75	290	1.98	6.16
305	1.08	3.81	295	2.04	6.36
310	1.10	3.88	300	2.11	6.57
315	1.12	3.94	305	2.17	6.77
			310	2.24	6.98
Recovered 80 %			315	2.31	7.18
Temperature (°C)			320	2.37	7.39
240	0.90	3.26	325	2.44	7.59
245	0.92	3.33	330	2.50	7.80
250	0.94	3.40	335	2.57	8.00
255	0.96	3.47	340	2.64	8.21
260	0.98	3.54	345	2.70	8.42
265	1.00	3.60	350	2.77	8.62
270	1.02	3.67	355	2.83	8.83
275	1.04	3.74	360	2.90	9.03
280	1.06	3.81			
285	1.07	3.88	Recovered FBP		
290	1.09	3.94	Temperature (°C)		
295	1.11	4.01	195–365		
300	1.13	4.08			
305	1.15	4.15			

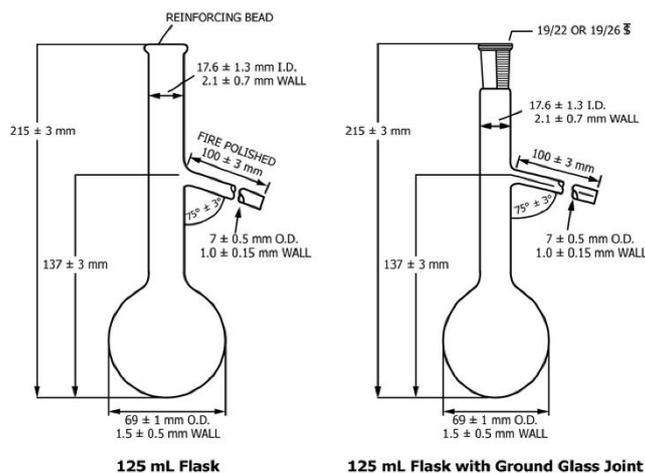


FIG. A2.1 125 mL Flask and 125 mL Flask with Ground Glass Joint

A2. DETAILED DESCRIPTION OF APPARATUS

A2.1 *Distillation Flasks*—Flasks shall be of heat resistant glass, constructed to the dimensions and tolerances shown in Fig. A2.1 and Fig. A2.2. Flasks made of borosilicate glass shall comply with the requirements of Specification E1405. Flasks made of quartz shall be composed of 99.9+ % SiO₂. Flasks may also be constructed with a ground glass joint.

NOTE A2.1—Since the thermal response of borosilicate glass and quartz can be different, consider appropriate adjustments for the initial and final heat regulation to attain the time limits stated in the procedure.

NOTE A2.2—For tests specifying dry point, specially selected flasks with bottoms and walls of uniform thickness are desirable.

A2.1.1 Intralaboratory and interlaboratory data¹¹ for motor gasoline, kerosene, aviation turbine fuel, fuel oil, and diesel fuel were assessed by Practice D6708 indicating that some correction could improve the degree of agreement between quartz and borosilicate flask results. The level of correction could be considered practically not significant. Correction is more probable at the IBP and FBP of both motor gasoline and distillate fuels. Optimizing D86 parameters for motor gasoline and distillate fuels may further minimize the differences in D86 IBP and FBP when using borosilicate versus quartz flask. Bias can conceivably occur for materials and temperatures not studied in this limited program.

A2.1.1.1 For motor gasoline in the temperature range of 25 °C to 220 °C:

$$\text{Borosilicate} = 1.0054 \text{ Quartz} - 0.73$$

A2.1.1.2 For kerosene, aviation turbine fuel, fuel oil, and diesel fuel in the temperature range of 140 °C to 350 °C:

$$\text{Borosilicate} = \text{Quartz} + 0.40$$

A2.2 *Condenser and Condenser Bath*—Typical types of condenser and condenser baths are illustrated in Figs. 1 and 2.

A2.2.1 The condenser shall be made of seamless noncorrosive metal tubing, 560 ± 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0.8 mm to 0.9 mm.

NOTE A2.3—Brass or stainless steel has been found to be a suitable material for this purpose.

A2.2.2 The condenser shall be set so that 393 mm ± 3 mm of the tube is in contact with the cooling medium, with 50 mm ± 3 mm outside the cooling bath at the upper end, and with 114 mm ± 3 mm outside at the lower end. The portion of the tube projecting at the upper end shall be set at an angle of 75° ± 3° with the vertical. The portion of the tube inside the condenser bath shall be either straight or bent in any suitable continuous smooth curve. The average gradient shall be 15° ± 1° with respect to the horizontal, with no 10 cm section having a gradient outside of the 15° ± 3° range. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and the lower end shall be cut off at an acute angle. Provisions shall be made to enable the flow of the distillate to run down the side of the receiving cylinder. This can be accomplished by using a drip-deflector, which is attached to the outlet of the tube. Alternatively, the lower portion of the condenser tube can be curved slightly backward to ensure contact with the wall of the receiving cylinder at a point 25 mm to 32 mm below the top of the receiving cylinder. Fig. A2.3 is a drawing of an acceptable configuration of the lower end of the condenser tube.

¹¹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1753. Contact ASTM Customer Service at service@astm.org.

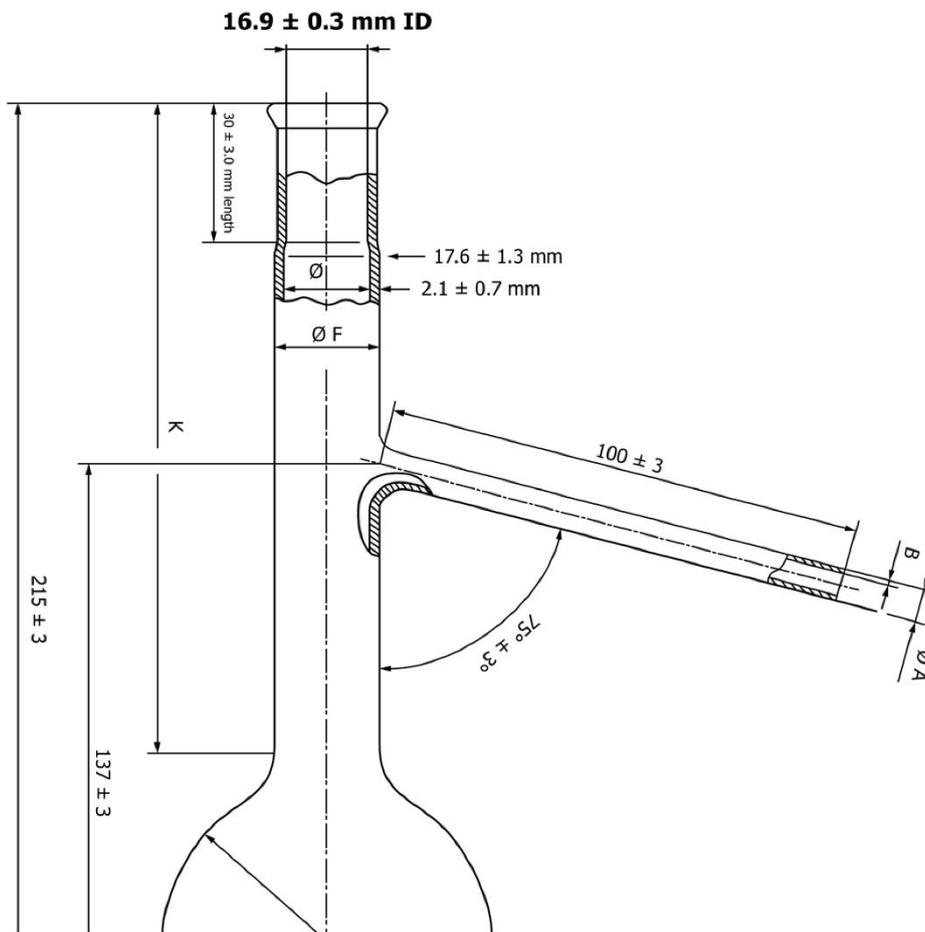


FIG. A2.2 Detail of Upper Neck Section

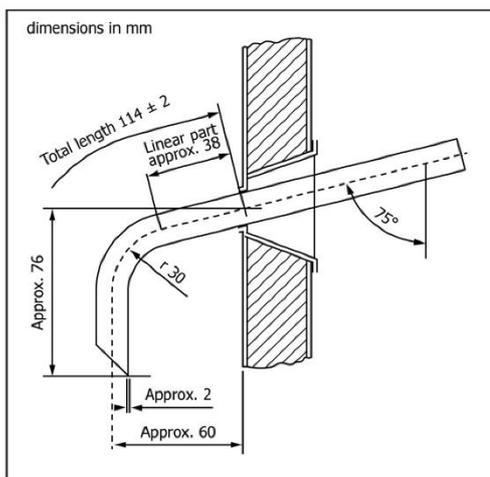
A2.2.3 The volume and the design of the bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single condenser bath may be used for several condenser tubes.

A2.3 Metal Shield or Enclosure for Flask. (Manual units only).

A2.3.1 Shield for Gas Burner (see Fig. 1)—The purpose of this shield is to provide protection for the operator and yet allow easy access to the burner and to the distillation flask

during operation. A typical shield would be 480 mm high, 280 mm long, and 200 mm wide, made of sheet metal of 0.8 mm thickness (22 gauge). The shield shall be provided with at least one window to observe the dry point at the end of the distillation.

A2.3.2 Shield for Electric Heater (see Fig. 2)—A typical shield would be 440 mm high, 200 mm long, and 200 mm wide, made of sheet metal of approximately 0.8 mm thickness (22 gauge) and with a window in the front side. The shield shall



Lower End of Condenser Tube
FIG. A2.3 Lower End of Condenser Tube

be provided with at least one window to observe the dry point at the end of the distillation.

A2.4 Heat Source

A2.4.1 *Gas Burner* (see Fig. 1), capable of bringing over the first drop from a cold start within the time specified and of continuing the distillation at the specified rate. A sensitive manual control valve and gas pressure regulator to give complete control of heating shall be provided.

A2.4.2 *Electric Heater* (see Fig. 2), of low heat retention.

Note A2.4—Heaters, adjustable from 0 W to 1000 W, have been found to be suitable for this purpose.

A2.5 Flask Support

A2.5.1 *Type 1*—Use a Type 1 flask support with a gas burner (see Fig. 1). This support consists of either a ring support of the ordinary laboratory type, 100 mm or larger in diameter, supported on a stand inside the shield, or a platform adjustable from the outside of the shield. On this ring or platform is mounted a hard board made of ceramic or other heat-resistant material, 3 mm to 6 mm in thickness, with a central opening 76 mm to 100 mm in diameter, and outside line dimensions slightly smaller than the inside boundaries of the shield.

A2.5.2 *Type 2*—Use a Type 2 flask support assembly with electric heating (see Fig. 2 as one example). The assembly consists of an adjustable system onto which the electric heater is mounted with provision for placement of a flask support

board (see A2.6) above the electric heater. The whole assembly is adjustable from the outside of the shield.

A2.6 *Flask Support Board*—The flask support board shall be constructed of ceramic or other heat-resistant material, 3 mm to 6 mm in thickness. Flask support boards are classified as A, B, or C, based on the size of the centrally located opening, the dimension of which is shown in Table 3. The flask support board shall be of sufficient dimension to ensure that thermal heat to the flask only comes from the central opening and that extraneous heat to the flask other than through the central opening is minimized. (**Warning** —Asbestos-containing materials shall not be used in the construction of the flask support board.)

A2.7 The flask support board can be moved slightly in different directions on the horizontal plane to position the distillation flask so that direct heat is applied to the flask only through the opening in this board. Usually, the position of the flask is set by adjusting the length of the side-arm inserted into the condenser.

A2.8 Provision shall be made for moving the flask support assembly vertically so that the flask support board is in direct contact with the bottom of the distillation flask during the distillation. The assembly is moved down to allow for easy mounting and removal of the distillation flask from the unit.

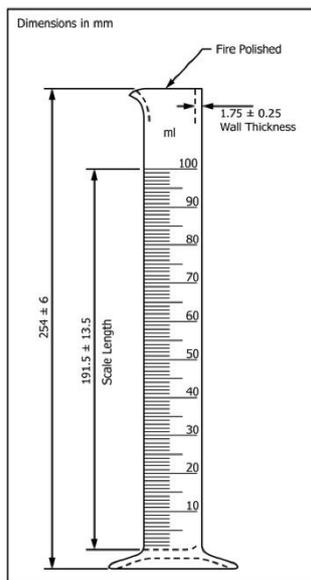
A2.9 *Receiving Cylinders*—The receiving cylinder shall have a capacity to measure and collect 100 mL ± 1.0 mL. The shape of the base shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 13° from the horizontal.

A2.9.1 *Manual Method*—The cylinder shall be graduated at intervals of 1 mL beginning at least at 5 mL and have a graduation at the 100 mL mark. Construction details and tolerances for the graduated cylinder are shown in Fig. A2.4.

A2.9.2 *Automated Method*—The cylinder shall conform to the physical specifications described in Fig. A2.4, except that graduations below the 100 mL mark are permitted, as long as they do not interfere with the operation of the level follower. Receiving cylinders for use in automated units may also have a metal base.

A2.9.3 If required, the receiving cylinder shall be immersed during the distillation to above the 100 mL graduation line in a cooling liquid contained in a cooling bath, such as a tall-form beaker of clear glass or transparent plastic. Alternatively, the receiving cylinder may be placed in a thermostated bath air circulation chamber.

A2.10 *Residue Cylinder*—The graduated cylinder shall have a capacity of 5 mL or 10 mL, with graduations into 0.1 mL subdivisions, beginning at 0.1 mL. The top of the cylinder may be flared, the other properties shall conform to Specification E1272.



Note 1—1 mL graduations – minimum 5 mL to 100 mL

FIG. A2.4 100 mL Graduated Cylinder

A3. DETERMINATION OF THE DIFFERENCE IN LAG TIME BETWEEN AN ELECTRONIC TEMPERATURE MEASUREMENT SYSTEM AND A MERCURY-IN-GLASS THERMOMETER

A3.1 The response time of an electronic temperature measuring device is inherently more rapid than that of a mercury-in-glass thermometer. The temperature measuring device assembly in general use, consisting of the sensor and its casing, or an electronic system and its associated software, or both, is so designed that the temperature measuring system will simulate the temperature lag of the mercury-in-glass thermometer.

A3.2 To determine the difference in lag time between such a temperature measuring system and a mercury-in-glass thermometer, analyze a sample such as gasoline, kerosine, jet fuel, or light diesel fuel with the electronic temperature measurement system in place and in accordance with the procedures described in this test method. In most cases this is the standard distillation step performed with an automated unit.

A3.2.1 Do not use a single pure compound, a very narrow boiling range product, or a synthetic blend of less than six compounds for this test.

A3.2.2 Best results are obtained with a sample that is typical of the sample load of the laboratory. Alternatively, use a full-range mixture with a 5 % to 95 % boiling range of at least 100 °C.

A3.3 Replace the electronic temperature measuring device with a low range or a high range mercury-in-glass thermometer, depending on the boiling range of the sample.

A3.4 Repeat the distillation with this thermometer, and manually record the temperature at the various percent recovered as described in 10.14.

A3.5 Calculate the values for the repeatability for the observed slope ($\Delta T/\Delta V$) for the different readings in the test.

A3.6 Compare the test data obtained using these two temperature measuring devices. The difference at any point shall be equal to, or less than, the repeatability of the method at that point. If this difference is larger, replace the electronic temperature measuring device or adjust the electronics involved, or both.

TABLE A4.1 Precision for Percent Evaporated at a Prescribed Temperature—Gasoline (Consolidated Equation)

Valid Range E70 – E180°C (Automated Apparatus)		
D86 Auto	r	R
	0.00836 (150 – X)	0.0200 (150 – X)

where: X = percent evaporated at the prescribed temperature

A4. PROCEDURE TO DETERMINE THE PERCENT EVAPORATED OR PERCENT RECOVERED AT A PRESCRIBED TEMPERATURE READING

A4.1 Many specifications require specific percentages evaporated or recovered at prescribed temperature readings, either as maxima, minima, or ranges. The procedures to determine these values are frequently designated by the terms Exxx or Rxxx, where xxx is the desired temperature.

Note: A4.1—Regulatory standards on the certification of reformulated gasoline under the complex model procedure require the determination of E200 and E300, defined as the percent evaporated fuel at 93.3 °C (200 °F) and 148.9 °C (300 °F), respectively. E158, the percent evaporated at a distillation temperature of 70 °C (158 °F), is also used in describing fuel volatility characteristics. Other typical temperatures are R 200 for kerosines and R 250 and R 350 for gas oils, where R 200, R 250, and R 350 are the percent recovered fuel at 200 °C, 250 °C, and 350 °C, respectively.

A4.2 Determine the barometric pressure, and calculate the correction to the desired temperature reading using Eq 3, Eq 4, or Eq 5 for $t = xxx^{\circ}\text{C}$ (or $t_f = xxx^{\circ}\text{F}$).

A4.2.1 *Manual Method*—Determine this correction to 0.5 °C (1 °F).

A4.2.2 *Automated Method*—Determine this correction to 0.1 °C (0.2 °F).

A4.3 Determine the expected temperature reading to yield xxx °C (or xxx °F) after the barometric correction. To obtain the expected value, add the absolute value of the calculated correction to the desired temperature if the barometric pressure is above 101.3 kPa. If the barometric pressure is below 101.3 kPa, subtract the absolute value of the calculated correction from the desired temperature.

A4.4 Perform the distillation, as described in Section 10, while taking into account A4.5 and A4.6.

A4.5 Manual Distillation

A4.5.1 In the region between about 10 °C below and 10 °C above the desired expected temperature reading determined in A4.3 record the temperature reading in intervals of 1 volume %.

A4.5.2 If the intent of the distillation is to solely determine the value of Exxx or Rxxx, discontinue the distillation after at least another 2 mL of distillate have been collected. Otherwise, continue the distillation, as described in Section 10, and determine the observed loss, as described in 11.1.

A4.5.2.1 If the intent of the distillation is to determine the value of Exxx and the distillation was terminated after about 2 mL of distillate was collected beyond the desired

temperature, allow the distillate to drain into the receiving graduate. Allow the contents of the flask to cool to below approximately 40 °C and then drain its contents into the receiving graduate. Note the volume of product in the receiving graduate to the nearest 0.5 mL at 2 min intervals until two successive observations agree.

A4.5.2.2 The amount recovered in the receiving graduate is the percent recovery. Determine the amount of observed loss by subtracting the percent recovery from 100.0.

A4.6 Automated Distillation

A4.6.1 In the region between about 10 °C below and 10 °C above the desired expected temperature reading determined in A4.3, collect temperature-volume data at 0.1 % volume intervals or less.

A4.6.2 Continue the distillation, as described in Section 10, and determine the percent loss, as described in 11.1.

A4.7 Calculations

A4.7.1 *Manual Method*—If a volume percent recovered reading is not available at the exact temperature calculated in A4.3, determine the percent recovered by interpolation between the two adjacent readings. Either the linear, as described in 11.6.1, or the graphical procedure, as described in 11.6.2, is permitted. The percent recovered is equal to Rxxx.

A4.7.2 *Automated Method*—Report the observed volume to 0.1 % volume corresponding to the temperature closest to the expected temperature reading. This is the percent recovered, or Rxxx.

A4.7.3 *Manual and Automated Methods*—To determine the value of Exxx, add the observed loss to the percent recovered, Rxxx, as determined in A4.7.1 or A4.7.2 and as described in Eq 9.

A4.7.3.1 As prescribed in 12.6, do not use the corrected loss.

A4.8 *Precision*—The statistical determination of the precision of the volume % evaporated or recovered at a prescribed temperature for automated apparatus were derived according to Practice D6300 from a 2005 interlaboratory program.¹⁰ Table A4.1 shows the consolidated equations for volume percent evaporated for gasoline, Table A4.2 shows the precision for volume percent recovered for diesel. The precision is valid only for the range of temperatures stated. The estimation of

TABLE A4.2 Precision for Percent Recovered at a Prescribed Temperature—Diesel (Rxxx)

Valid Range R200 – R300°C (Automated Apparatus)		
D86 Auto	R200C, R250C, R300C	
	<i>r</i>	R
	1.07	2.66

precision for temperature points outside the stated range can be calculated from the procedures in A4.10 and the precision tables in Annex A1.

A4.9 The statistical determination of the precision of the volume percent evaporated or recovered at a prescribed temperature for manual apparatus has not been directly measured in an interlaboratory program. It can be shown that the precision of the volume percent evaporated or recovered at a prescribed temperature is equivalent to the precision of the temperature measurement at that point divided by the rate of change of temperature versus volume percent evaporated or recovered. The estimation of precision becomes less precise at high slope values.

A4.10 Calculate the slope or rate of change in temperature reading, S_C (or S_F), as described in A4.10.1 and Eq A4.1 and using temperature values bracketing the desired temperature.

A4.10.1 Slope or Rate of Change of Temperature:

NOTE: A4.2—The slope can have a dramatic influence on precision for some samples, typically those containing oxygenates, and the calculated precision obtained using the values in Table A4.3 may not reflect this in all cases. This can be due to the changing composition of the sample, causing the slope to change rapidly over a short interval. This change may occur either during the data increments prior to, or subsequent to, the data point under calculation.

A4.10.1.1 To determine the precision of a result, it is generally necessary to determine the slope or rate of change of the temperature at that particular point. This variable, denoted as S_C or S_F , is equal to the change in temperature, either in °C or in °F, respectively, per percent recovered or evaporated.

A4.10.1.2 The precision of the IBP and EP does not require any slope calculation.

A4.10.1.3 With the exception stated in A4.10.1.2, the slope at any point during the distillation is calculated from the following equations, using the values shown in Table A4.3:

$$S_C \text{ (or } S_F) = (T_U - T_L)/(V_U - V_L) \quad (A4.1)$$

where:

- S_C = the slope, °C/volume %,
- S_F = the slope, °F/volume %,
- T_U = the upper temperature, °C (or °F),
- T_L = the lower temperature, °C (or °F),
- V_U = the volume % recovered or evaporated corresponding to T_U ,
- V_L = the volume % recovered or evaporated corresponding to T_L , and

V_{EP} = the volume % recovered or evaporated corresponding to the end point.

A4.10.1.4 In the event that the distillation end point occurs prior to the 95 % point, the slope at the end point is calculated as follows:

$$S_C \text{ (or } S_F) = (T_{EP} - T_{HR})/(V_{EP} - V_{HR}) \quad (A4.2)$$

where:

- T_{EP} or T_{HR} = the temperature, in °C or °F, at the percent volume recovered indicated by the subscript, and
- V_{EP} or V_{HR} = the volume % recovered.
- Subscript EP* = end point, and
- Subscript HR* = highest reading, either 80 % or 90 %, prior to the end point.

A4.10.1.5 For points between 10 % to 85 % recovered that are not shown in Table A4.3, the slope is calculated as follows:

$$S_C \text{ (or } S_F) = 0.05 (T_{(V+10)} - T_{(V-10)}) \quad (A4.3)$$

A4.10.2 Calculate the repeatability, r , or the reproducibility, R , from the slope, S_C (or S_F) and the data in Tables A4.4 and A4.5.

A4.10.3 Determine the repeatability or reproducibility, or both, of the volume % evaporated or recovered at a prescribed temperature from the following formulas:

$$r_{\text{volume \%}} = r/S_C (S_F) \quad (A4.4)$$

$$R_{\text{volume \%}} = R/S_C (S_F) \quad (A4.5)$$

where:

- $r_{\text{volume \%}}$ = repeatability of the volume percent evaporated or recovered,
- $R_{\text{volume \%}}$ = reproducibility of the volume percent evaporated or recovered,
- r = repeatability of the temperature at the prescribed temperature at the observed percent distilled,
- R = reproducibility of the temperature at the prescribed temperature at the observed percent distilled, and
- $S_C (S_F)$ = rate of change in temperature reading in °C (°F) per the volume percent evaporated or recovered.

A4.10.4 Examples on how to calculate the repeatability and the reproducibility are shown in Appendix X2.

TABLE A4.3 Data Points for Determining Slope, S_C or S_F

Slope at %	IBP	5	10	20	30	40	50	60	70	80	90	95	EP
T _i at %	0	0	0	10	20	30	40	50	60	70	80	90	95
T _U at %	5	10	20	30	40	50	60	70	80	90	90	95	V _{EP}
V _U - V _L	5	10	20	20	20	20	20	20	20	20	10	5	V _{EP} -95

TABLE A4.4 Repeatability and Reproducibility for Group 1

Evaporated Point, %	Manual Repeatability ^A		Manual Reproducibility ^A	
	°C	°F	°C	°F
IBP	3.3	6	5.6	10
5	1.9+0.86S _C	3.4+0.86S _F	3.1+1.74S _C	5.6+1.74S _F
10	1.2+0.86S _C	2.2+0.86S _F	2.0+1.74S _C	3.6+1.74S _F
20	1.2+0.86S _C	2.2+0.86S _F	2.0+1.74S _C	3.6+1.74S _F
30-70	1.2+0.86S _C	2.2+0.86S _F	2.0+1.74S _C	3.6+1.74S _F
80	1.2+0.86S _C	2.2+0.86S _F	2.0+1.74S _C	3.6+1.74S _F
90	1.2+0.86S _C	2.2+0.86S _F	0.8+1.74S _C	1.4+1.74S _F
95	1.2+0.86S _C	2.2+0.86S _F	1.1+1.74S _C	1.9+1.74S _F
FBP	3.9	7	7.2	13

^A S_C or S_F is the average slope (or rate of change) calculated in accordance with A4.10.1. Table A4.4 precision data obtained from RR study on both manual and automated D86 units by North American and IP laboratories.

TABLE A4.5 Repeatability and Reproducibility for Groups 2, 3 and 4 (Manual Method)

	Repeatability ^A		Reproducibility ^A	
	°C	°F	°C	°F
IBP	1.0+0.35S _C	1.9+0.35S _F	2.8+0.93S _C	5.0+0.93S _F
5-95 %	1.0+0.41S _C	1.8+0.41S _F	1.8+1.33S _C	3.3+1.33S _F
FBP	0.7+0.36S _C	1.3+0.36S _F	3.1+0.42S _C	5.7+0.42S _F
% volume at temperature reading	0.7+0.92/S _C	0.7+1.66/S _F	1.5+1.78/S _C	1.53+3.20/S _F

^A S_C or S_F is the average slope (or rate of change) calculated in accordance with A4.10.1. Table A4.5 has been derived from the monographs in Figs. 6 and 7 in D86-97.

APPENDICES

(Nonmandatory Information)

X1. EXAMPLES ILLUSTRATING CALCULATIONS FOR REPORTING OF DATA

X1.1 The observed distillation data used for the calculation of the examples below are shown in the first three columns of Fig. X1.1.

X1.1.1 Temperature readings corrected to 101.3 kPa (760 mm Hg) pressure (see 11.3) are as follows:

$$\text{correction (}^\circ\text{C)} = 0.0009 (101.3 - 98.6) (273 + t_r) \quad (\text{X1.1})$$

$$\text{correction (}^\circ\text{F)} = 0.00012 (760 - 740) (460 + t_r) \quad (\text{X1.2})$$

X1.1.2 Loss correction to 101.3 kPa (see 11.4) are as follows. The data for the examples are taken from Fig. X1.1.

$$\text{corrected loss} = (0.5 + (4.7 - 0.5)) / \quad (\text{X1.3})$$

$$\{1 + (101.3 - 98.6)/8.0\} = 3.6$$

X1.1.3 Recovery correction to 101.3 kPa (see 11.4.1) are as follows:

$$\text{corrected recovery} = 94.2 + (4.7 - 3.6) = 95.3 \quad (\text{X1.4})$$

Sample ID:											Barometric pressure: 98.6 kPa	
Date analyzed:											Analyst:	
Equipment No:												
Remarks:												
	Barometric pressure											
	observed		corrected		procedure							
	98.6 kPa		101.3 kPa		arithmetical/graphical							
	740 mm Hg		760 mm Hg		evaporated							
% recovered	°C	°F	°C	°F	% evaporated	T _{evap} °C	T _{evap} °F					
IBP	25.5	78	26.2	79.2	5	26.7	80.0					
5	33.0	91	33.7	92.7	10	34.1	93.4					
10	39.5	103	40.3	104.5	15	40.7	105.2					
15	46.0	115	46.8	116.2	20	47.3	117.1					
20	54.5	130	55.3	131.5	30	65.7	150.2					
30	74.0	165	74.8	166.7	40	84.9	184.9					
40	93.0	199	93.9	201.0	50	101.9	215.3					
50	108.0	226	108.9	228.0	60	116.9	242.4					
60	123.0	253	124.0	255.1	70	134.1	273.3					
70	142.0	288	143.0	289.4	80	156.0	312.8					
80	166.5	332	167.6	333.6	85	168.4	335.1					
85	180.5	357	181.6	358.9	90	182.8	361.0					
90	200.4	393	201.6	394.8	95	202.4	396.3					
EP	215.0	419	216.2	421.1								
recovery, %	94.2		95.3									
residue, %	1.1		1.1									
loss, %	4.7		3.6									

FIG. X1.1 Example of Test Report

X1.2 Temperature Readings at Prescribed Percent Evaporated

X1.2.1 Temperature reading at 10 % evaporated (4.7 % observed loss = 5.3 % recovered) (see 11.6.1) are as follows:

$$T_{10E} (^{\circ}C) = 33.7 + [(40.3 - 33.7)] \quad (X1.5)$$

$$(5.3 - 5) / (10 - 5) = 34.1^{\circ}C$$

$$T_{10E} (^{\circ}F) = 92.7 + [(104.5 - 92.7)] \quad (X1.6)$$

$$(5.3 - 5) / (10 - 5) = 93.1^{\circ}F$$

X1.2.2 Temperature reading at 50 % evaporated (45.3 % recovered) (see 11.6.1) are as follows:

$$T_{50E} (^{\circ}C) = 93.9 + [(108.9 - 93.9)] \quad (X1.7)$$

$$(45.3 - 40) / (50 - 40) = 101.9^{\circ}C$$

$$T_{50E} (^{\circ}F) = 201 + [(228 - 201)] \quad (X1.8)$$

$$(45.3 - 40) / (50 - 40) = 215.3^{\circ}F$$

X1.2.3 Temperature reading at 90 % evaporated (85.3 % recovered) (see 11.6.1) are as follows:

$$T_{90E} (^{\circ}C) = 181.6 + [(201.6 - 181.6)] \quad (X1.9)$$

$$(85.3 - 85) / (90 - 85) = 182.8^{\circ}C$$

$$T_{90E} (^{\circ}F) = 358.9 + [(394.8 - 358.9)] \quad (X1.10)$$

$$(85.3 - 85) / (90 - 85) = 361.0^{\circ}F$$

X1.2.4 Temperature reading at 90 % evaporated (85.3 % recovered) not corrected to 101.3 kPa pressure (see 11.6.1) are as follows:

$$T_{90E} (^{\circ}C) = 180.5 + [(200.4 - 180.5)] \quad (X1.11)$$

$$(85.3 - 85) / (90 - 85) = 181.7^{\circ}C$$

$$T_{90E} (^{\circ}F) = 357 + [(392 - 357)] \quad (X1.12)$$

$$(85.3 - 85) / (90 - 85) = 359.1^{\circ}F$$

NOTE: X1.1—Results calculated from $^{\circ}C$ data may not correspond exactly to results calculated from $^{\circ}F$ data because of errors in rounding.

X2. EXAMPLES OF CALCULATION OF REPEATABILITY AND REPRODUCIBILITY OF VOLUME % (RECOVERED OR EVAPORATED) AT A PRESCRIBED TEMPERATURE READING

TABLE X2.1 Distillation Data from a Group 1 Sample Manual Distillation

Distillation Point Recovered, mL	Temperature $^{\circ}C$	Temperature $^{\circ}F$	Volume (mL) Recovered at 93.3 $^{\circ}C$ (200 $^{\circ}F$)
			18.0
10	84	183	
20	94	202	
30	103	217	
40	112	233	

Distillation Point Evaporated, mL	Temperature $^{\circ}C$	Temperature $^{\circ}F$	Volume (mL) Evaporated at 93.3 $^{\circ}C$ (200 $^{\circ}F$)
			18.4
10	83	182	
20	94	201	
30	103	217	
40	111	232	

X2.1 Some specifications require the reporting of the volume % evaporated or recovered at a prescribed temperature. Table X2.1 shows the distillation data of a Group 1 sample as obtained by a manual unit.

X2.2 Example Calculation

X2.2.1 For a Group 1 sample exhibiting distillation characteristics as per Table X2.1, as determined by a manual unit, the reproducibility of the volume evaporated, $R_{\text{volume}}\%$, at 93.3 $^{\circ}C$ (200 $^{\circ}F$) is determined as follows:

X2.2.1.1 Determine first the slope at the desired temperature:

$$S_C \% = 0.1 (T_{(20)} - T_{(10)}) \quad (X2.1)$$

$$= 0.1 (94 - 83)$$

$$= 1.1$$

$$S_F \% = 0.1 (T_{(20)} - T_{(10)})$$

$$= 0.1 (201 - 182)$$

$$= 1.9$$

X2.2.2 From Table A4.4, determine the value of R , the reproducibility at the observed percentage distilled. In this case, the observed percentage distilled is 18 % and

$$R = 2.0 + 1.74 (S_C) \quad (X2.2)$$

$$= 2.0 + 1.74 \times 1.1$$

$$= 3.9$$

$$R = 3.6 + 1.74 (S_F)$$

$$= 3.6 + 1.74 \times 1.9$$

$$= 6.9$$

X2.2.3 From the calculated value of R , determine the value of volume, as described in A4.10.

$$R \text{ volume } \% = R / (S_C) \quad (X2.3)$$

$$= 3.9 / 1.1$$

$$= 3.5$$

$$R \text{ volume } \% = R / (S_F)$$

$$= 6.9 / 1.9$$

$$= 3.6$$

Barometric Pressure, kPa		76.1	80.9	84.5	87.3	89.6	91.5	93.1	94.1	95.5	96.4	97.2	97.9	98.4	98.9	99.5	100.0	100.4	100.8	101.2	101.5	102.0	102.4	102.8	103.2	
from through		80.8	84.4	87.2	89.5	91.4	93.0	94.0	95.4	96.3	97.1	97.8	98.3	98.8	99.4	99.9	100.3	100.7	101.1	101.4	101.9	102.3	102.7	103.1	103.5	
Observed Loss	--- Corrected Loss ----->																									
Units		0	0.37	0.35	0.33	0.31	0.29	0.27	0.25	0.23	0.20	0.18	0.16	0.14	0.13	0.11	0.09	0.06	0.04	0.02	-0.00	-0.02	-0.06	-0.09	-0.13	-0.17
		1	0.63	0.65	0.67	0.69	0.71	0.73	0.75	0.78	0.80	0.82	0.84	0.86	0.87	0.89	0.92	0.94	0.96	0.98	1.00	1.03	1.06	1.09	1.13	1.17
		2	0.89	0.95	1.01	1.08	1.14	1.20	1.26	1.33	1.40	1.46	1.52	1.57	1.62	1.68	1.75	1.81	1.87	1.94	2.00	2.08	2.17	2.27	2.38	2.51
		3	1.15	1.25	1.36	1.46	1.57	1.67	1.77	1.88	1.99	2.09	2.19	2.28	2.37	2.47	2.58	2.69	2.79	2.90	3.00	3.13	3.29	3.45	3.63	3.84
		4	1.41	1.56	1.70	1.84	1.99	2.14	2.28	2.43	2.59	2.73	2.87	3.00	3.12	3.26	3.41	3.56	3.70	3.85	4.00	4.18	4.40	4.63	4.89	5.18
		5	1.68	1.86	2.04	2.23	2.42	2.61	2.79	2.98	3.19	3.37	3.55	3.71	3.87	4.05	4.25	4.44	4.62	4.81	5.00	5.23	5.51	5.81	6.14	6.52
		6	1.94	2.16	2.39	2.61	2.84	3.08	3.30	3.53	3.78	4.01	4.23	4.42	4.62	4.84	5.08	5.31	5.53	5.77	6.00	6.28	6.63	6.99	7.40	7.86
		7	2.20	2.46	2.73	3.00	3.27	3.55	3.80	4.08	4.38	4.65	4.90	5.14	5.37	5.63	5.91	6.18	6.44	6.73	7.00	7.33	7.74	8.17	8.65	9.20
		8	2.46	2.76	3.07	3.38	3.70	4.02	4.31	4.63	4.98	5.28	5.58	5.85	6.12	6.41	6.74	7.06	7.36	7.69	8.00	8.38	8.86	9.35	9.90	10.53
		9	2.72	3.07	3.41	3.76	4.12	4.49	4.82	5.18	5.57	5.92	6.26	6.56	6.87	7.20	7.57	7.93	8.27	8.65	9.00	9.43	9.97	10.53	11.16	11.87
		10	2.98	3.37	3.76	4.15	4.55	4.96	5.33	5.73	6.17	6.56	6.94	7.28	7.62	7.99	8.41	8.81	9.19	9.60	10.00	10.48	11.08	11.71	12.41	13.21
		11	3.24	3.67	4.10	4.53	4.97	5.43	5.84	6.28	6.77	7.20	7.61	7.99	8.37	8.78	9.24	9.68	10.10	10.56	11.00	11.53	12.20	12.89	13.67	14.55
		12	3.50	3.97	4.44	4.92	5.40	5.90	6.35	6.83	7.36	7.84	8.29	8.71	9.12	9.57	10.07	10.56	11.02	11.52	12.00	12.59	13.31	14.07	14.92	15.89
		13	3.76	4.27	4.78	5.30	5.83	6.36	6.86	7.39	7.96	8.47	8.97	9.42	9.86	10.36	10.90	11.43	11.93	12.48	13.00	13.64	14.43	15.25	16.17	17.22
		14	4.03	4.58	5.13	5.69	6.25	6.83	7.36	7.94	8.56	9.11	9.64	10.13	10.61	11.15	11.74	12.31	12.85	13.44	14.00	14.69	15.54	16.43	17.43	18.56
		15	4.29	4.88	5.47	6.07	6.68	7.30	7.87	8.49	9.15	9.75	10.32	10.85	11.36	11.93	12.57	13.18	13.76	14.40	15.00	15.74	16.66	17.61	18.68	19.90
		16	4.55	5.18	5.81	6.45	7.10	7.77	8.38	9.04	9.75	10.39	11.00	11.56	12.11	12.72	13.40	14.06	14.68	15.36	16.00	16.79	17.77	18.79	19.94	21.24
		17	4.81	5.48	6.16	6.84	7.53	8.24	8.89	9.59	10.35	11.03	11.66	12.27	12.86	13.51	14.23	14.93	15.59	16.31	17.00	17.84	18.88	19.97	21.19	22.58
		18	5.07	5.78	6.50	7.22	7.96	8.71	9.40	10.14	10.94	11.66	12.35	12.99	13.61	14.30	15.07	15.80	16.50	17.27	18.00	18.89	20.00	21.15	22.44	23.91
		19	5.33	6.08	6.84	7.61	8.38	9.18	9.91	10.69	11.54	12.30	13.03	13.70	14.36	15.09	15.90	16.68	17.42	18.23	19.00	19.94	21.11	22.33	23.70	25.25
		20	5.59	6.39	7.18	7.99	8.81	9.65	10.41	11.24	12.14	12.94	13.71	14.41	15.11	15.88	16.73	17.55	18.33	19.19	20.00	20.99	22.23	23.51	24.95	26.59
Tenths		0.0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
		0.1	0.03	0.03	0.03	0.04	0.04	0.05	0.05	0.06	0.06	0.07	0.07	0.07	0.08	0.08	0.09	0.09	0.10	0.10	0.11	0.11	0.12	0.12	0.13	0.13
		0.2	0.05	0.06	0.07	0.08	0.09	0.09	0.10	0.11	0.12	0.13	0.14	0.14	0.15	0.16	0.17	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.27
		0.3	0.08	0.09	0.10	0.12	0.13	0.14	0.15	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.26	0.27	0.29	0.30	0.32	0.33	0.35	0.38	0.40
		0.4	0.10	0.12	0.14	0.15	0.17	0.19	0.20	0.22	0.24	0.26	0.27	0.29	0.30	0.32	0.33	0.35	0.37	0.38	0.40	0.42	0.45	0.47	0.50	0.54
		0.5	0.13	0.15	0.17	0.19	0.21	0.23	0.25	0.28	0.30	0.32	0.34	0.36	0.37	0.39	0.42	0.44	0.46	0.48	0.50	0.53	0.56	0.59	0.63	0.67
		0.6	0.16	0.18	0.21	0.23	0.26	0.28	0.31	0.33	0.36	0.38	0.41	0.43	0.45	0.47	0.50	0.52	0.55	0.58	0.60	0.63	0.67	0.71	0.75	0.80
		0.7	0.18	0.21	0.24	0.27	0.30	0.33	0.36	0.39	0.42	0.45	0.47	0.50	0.52	0.55	0.58	0.61	0.64	0.67	0.70	0.74	0.78	0.83	0.88	0.94
		0.8	0.21	0.24	0.27	0.31	0.34	0.38	0.41	0.44	0.48	0.51	0.54	0.57	0.60	0.63	0.67	0.70	0.73	0.77	0.80	0.84	0.89	0.94	1.00	1.07
		0.9	0.24	0.27	0.31	0.35	0.38	0.42	0.46	0.50	0.54	0.57	0.61	0.64	0.67	0.71	0.75	0.79	0.82	0.86	0.90	0.95	1.00	1.06	1.13	1.20

FIG. X3.1 Corrected Loss from Observed Loss and Barometric Pressure kPa

X3. TABLES OF CORRECTED LOSS FROM MEASURED LOSS AND BAROMETRIC PRESSURE

X3.1 The table presented as Fig. X3.1 can be used to determine the corrected loss from the measured loss and the barometric pressure in kPa.

X3.2 The table presented as Fig. X3.2 can be used to determine the corrected loss from the measured loss and the barometric pressure in mm Hg.

Barometric Pressure, mm Hg.

from through	571 606	607 633	634 654	655 671	672 685	686 697	698 705	706 715	716 722	723 728	729 733	734 737	738 741	742 745	746 749	750 752	753 755	756 758	759 761	762 764	765 767	768 770	771 773	774 776		
Observed Loss	/ - - Corrected Loss - - - - - >																									
Units	0	0.37	0.35	0.33	0.31	0.29	0.27	0.25	0.23	0.20	0.18	0.16	0.14	0.13	0.11	0.09	0.07	0.05	0.02	-0.00	-0.03	-0.06	-0.09	-0.13	-0.17	
	1	0.63	0.65	0.67	0.69	0.71	0.73	0.75	0.77	0.80	0.82	0.84	0.86	0.87	0.89	0.91	0.93	0.95	0.98	1.00	1.03	1.06	1.09	1.13	1.17	
	2	0.89	0.95	1.01	1.07	1.14	1.20	1.26	1.32	1.39	1.45	1.51	1.57	1.62	1.68	1.74	1.80	1.86	1.93	2.00	2.08	2.17	2.27	2.38	2.50	
	3	1.15	1.25	1.36	1.46	1.56	1.67	1.77	1.87	1.99	2.09	2.19	2.28	2.36	2.46	2.57	2.67	2.77	2.88	3.00	3.13	3.28	3.44	3.63	3.83	
	4	1.41	1.55	1.70	1.84	1.99	2.14	2.27	2.42	2.58	2.72	2.86	2.99	3.11	3.25	3.40	3.54	3.68	3.83	4.00	4.19	4.39	4.62	4.88	5.17	
	5	1.67	1.86	2.04	2.22	2.41	2.61	2.78	2.97	3.18	3.36	3.54	3.70	3.86	4.03	4.23	4.41	4.59	4.79	5.00	5.24	5.50	5.80	6.13	6.50	
	6	1.93	2.16	2.38	2.61	2.84	3.07	3.29	3.52	3.77	3.99	4.21	4.41	4.60	4.82	5.05	5.28	5.50	5.74	6.00	6.29	6.61	6.97	7.38	7.84	
	7	2.19	2.46	2.72	2.99	3.26	3.54	3.79	4.07	4.36	4.63	4.88	5.12	5.35	5.60	5.88	6.15	6.41	6.69	7.00	7.34	7.72	8.15	8.63	9.17	
	8	2.46	2.76	3.07	3.37	3.69	4.01	4.30	4.62	4.96	5.27	5.56	5.83	6.09	6.38	6.71	7.02	7.32	7.64	8.00	8.40	8.84	9.33	9.88	10.50	
	9	2.72	3.06	3.41	3.76	4.11	4.48	4.81	5.17	5.55	5.90	6.23	6.54	6.84	7.17	7.54	7.89	8.23	8.60	9.00	9.45	9.95	10.50	11.13	11.84	
	10	2.98	3.36	3.75	4.14	4.54	4.94	5.31	5.71	6.15	6.54	6.91	7.25	7.58	7.95	8.37	8.76	9.14	9.55	10.00	10.50	11.06	11.68	12.38	13.17	
	11	3.24	3.66	4.09	4.52	4.96	5.41	5.82	6.26	6.74	7.17	7.58	7.96	8.33	8.74	9.19	9.63	10.05	10.50	11.00	11.56	12.17	12.86	13.63	14.51	
	12	3.50	3.96	4.43	4.91	5.39	5.88	6.33	6.81	7.34	7.81	8.26	8.67	9.07	9.52	10.02	10.50	10.96	11.46	12.00	12.61	13.28	14.03	14.88	15.84	
	13	3.76	4.27	4.78	5.29	5.81	6.35	6.83	7.36	7.93	8.44	8.93	9.38	9.82	10.31	10.85	11.37	11.87	12.41	13.00	13.66	14.39	15.21	16.13	17.17	
	14	4.02	4.57	5.12	5.67	6.24	6.82	7.34	7.91	8.53	9.08	9.61	10.09	10.57	11.09	11.68	12.24	12.78	13.36	14.00	14.71	15.51	16.39	17.38	18.51	
	15	4.28	4.87	5.46	6.06	6.66	7.28	7.85	8.46	9.12	9.71	10.28	10.80	11.31	11.88	12.51	13.11	13.68	14.31	15.00	15.77	16.62	17.57	18.63	19.84	
	16	4.54	5.17	5.80	6.44	7.09	7.75	8.35	9.01	9.72	10.35	10.95	11.51	12.06	12.66	13.33	13.98	14.59	15.27	16.00	16.82	17.73	18.74	19.88	21.18	
	17	4.80	5.47	6.14	6.82	7.51	8.22	8.86	9.56	10.31	10.98	11.63	12.22	12.80	13.45	14.16	14.85	15.50	16.22	17.00	17.87	18.84	19.92	21.13	22.51	
	18	5.06	5.77	6.49	7.21	7.94	8.69	9.37	10.11	10.91	11.62	12.30	12.93	13.55	14.23	14.99	15.72	16.41	17.17	18.00	18.93	19.95	21.10	22.38	23.84	
	19	5.32	6.07	6.83	7.59	8.36	9.15	9.88	10.65	11.50	12.25	12.98	13.64	14.29	15.02	15.82	16.59	17.32	18.12	19.01	19.98	21.06	22.27	23.45	24.89	26.51
	20	5.58	6.37	7.17	7.97	8.79	9.62	10.38	11.20	12.09	12.89	13.65	14.35	15.04	15.80	16.64	17.46	18.23	19.08	20.01	21.03	22.17	23.45	24.89	26.51	
Tenths	0.0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
	0.1	0.03	0.03	0.03	0.04	0.04	0.05	0.05	0.05	0.06	0.06	0.07	0.07	0.07	0.08	0.08	0.09	0.09	0.10	0.10	0.11	0.11	0.12	0.13	0.13	
	0.2	0.05	0.06	0.07	0.08	0.08	0.09	0.10	0.11	0.12	0.13	0.14	0.15	0.16	0.17	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.27	0.27	
	0.3	0.08	0.09	0.10	0.11	0.13	0.14	0.15	0.16	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.26	0.27	0.29	0.30	0.32	0.33	0.35	0.38	0.40	
	0.4	0.10	0.12	0.14	0.15	0.17	0.19	0.20	0.22	0.24	0.25	0.27	0.28	0.30	0.31	0.33	0.35	0.36	0.38	0.40	0.42	0.44	0.47	0.50	0.53	
	0.5	0.13	0.15	0.17	0.19	0.21	0.23	0.25	0.27	0.30	0.32	0.34	0.36	0.37	0.39	0.41	0.43	0.45	0.48	0.50	0.53	0.56	0.59	0.63	0.67	
	0.6	0.16	0.18	0.21	0.23	0.25	0.28	0.30	0.33	0.36	0.38	0.40	0.43	0.45	0.47	0.50	0.52	0.55	0.57	0.60	0.63	0.67	0.71	0.75	0.80	
	0.7	0.18	0.21	0.24	0.27	0.30	0.33	0.35	0.38	0.42	0.44	0.47	0.50	0.52	0.55	0.58	0.61	0.64	0.67	0.70	0.74	0.78	0.82	0.88	0.93	
	0.8	0.21	0.24	0.27	0.31	0.34	0.37	0.41	0.44	0.48	0.51	0.54	0.57	0.60	0.63	0.66	0.70	0.73	0.76	0.80	0.84	0.89	0.94	1.00	1.07	
	0.9	0.23	0.27	0.31	0.34	0.38	0.42	0.46	0.49	0.54	0.57	0.61	0.64	0.67	0.71	0.75	0.78	0.82	0.86	0.90	0.95	1.00	1.06	1.13	1.20	

FIG. X3.2 Corrected Loss from Observed Loss and Barometric Pressure mm Hg

X4. PROCEDURE TO EMULATE THE EMERGENT STEM ERROR OF A MERCURY-IN-GLASS THERMOMETER

X4.1 When an electronic or other sensor without an emergent stem error is used, the output of this sensor or the associated data system should emulate the output of a mercury-in-glass thermometer. Based on information supplied by four manufacturers of automated Test Method D86 equipment, the averaged equations shown in X4.2 and X4.3 have been reported to be in use.

X4.1.1 The equations shown in X4.2 have limited applicability and are shown for information purposes only. In addition to the correction for the emergent stem, the electronic sensor and associated data system will also have to emulate the lag in response time observed for mercury-in-glass thermometers.

X4.2 When a low range thermometer would have been used, no stem correction is to be applied below 20°C. Above this temperature, the correction is calculated using the following formula:

$$ASTM\ 7C\ T_{elr} = T_t - 0.000162 \times (T_t - 20^\circ C)^2 \quad (X4.1)$$

X4.3 When a high range thermometer would have been used, no stem correction is to be applied below 35°C. Above this temperature the correction is calculated using the following formula:

$$ASTM\ 8C\ T_{elr} = T_t - 0.000131 \times (T_t - 35^\circ C)^2 \quad (X4.2)$$

where:

- T_{elr} = emulated temperature in °C for low range thermometers,
- T_{elhr} = emulated temperature in °C for high range thermometers, and
- T_t = true temperature in °C.

"Percent Recovered" Report Form

Date:
 Time:
 Operator:

Ambient temperature (°C)
 Atmospheric pressure (kPa)
 Condenser temperature (°C)
 Temperature of the bath around receiving cylinder (°C)

	Percent Recovered	Corrected Temperature Reading (°C)	Time or mL / min
	IBP		
	5		
	10		
	15		
	20		
	25		
	30		
	35		
	40		
	45		
	50		
	55		
	60		
	65		
	70		
	75		
	80		
	85		
	90		
5 ml residue	95		
FBP			

Percent Recovery
 Percent Residue
 Percent Total Recovery
 Percent Loss
 Corrected Percent Recovery Corrected Loss
 Corrected Total Recovery

Comments:

- Ambient temperature at the start of the test
- Ambient barometric pressure at the start of the test
- Volume of condensate observed in the receiving cylinder at any point in the distillation, expressed as a percentage of the charge volume, in connection with simultaneous temperature reading
- Temperature measuring device readings which are corrected to 101, 3 kPa barometric pressure
- Group 1, 2 & 3: 5 to 10 minutes
Group 4: 5 to 15 minutes
- Group 1 & 2: 60 to 100 seconds
- 4 to 5 ml / min uniform average rate from 5% recovered to 5 ml in flask
- Volume of condensate observed in the receiving cylinder when the 5 ml conditions are reached
- Volume of condensate observed in the receiving cylinder when the final boiling point is observed
- Maximum percent recovered
- Volume of residue in the flask expressed as a percentage of the charge volume
- Combined Percent Recovery and Percent Residue in the flask
- Time from 5 ml in flask to FBP = < 5 minutes
- 100 minus the Total Recovery
- Percent Recovery corrected for barometric pressure
- Percent Loss corrected for barometric pressure
- Combined Percent Recovery and Percent Residue in the flask corrected for barometric pressure

FIG. X5.1 Percent Recovered Report Form

X5. EXPLANATORY REPORT FORMS

X5.1 Fig. X5.1 and Fig. X5.2 show report forms.

"Percent Evaporated" Report Form

Laboratory: _____

Date: _____
 Time: _____
 Operator: _____

Ambient temperature (°C) _____
 Atmospheric pressure (kPa) _____
 Condenser temperature (°C) _____
 Temperature of the bath around receiving cylinder (°C) _____

Percent Recovered	Corrected Temperature Reading (°C)	Time or mL / min	Percent Evaporated	Temperature Readings at prescribed percent evaporated (°C)
IBP			IBP	
5			5	
10			10	
15			15	
20			20	
25			25	
30			30	
35			35	
40			40	
45			45	
50			50	
55			55	
60			60	
65			65	
70			70	
75			75	
80			80	
85			85	
90			90	
5 ml residue				
95			95	
FBP			FBP	

Percent Recovery _____
 Percent Residue _____
 Percent Total Recovery _____
 Percent Loss _____
 Corrected Percent Recovery _____

Corrected Loss _____
 Corrected Total Recovery _____

Comments: _____

Ambient temperature at the start of the test

Ambient barometric pressure at the start of the test

Volume of condensate observed in the receiving cylinder at any point in the distillation, expressed as a percentage of the charge volume, in connection with simultaneous temperature reading

Temperature measuring device readings which are corrected to 101,3 kPa barometric pressure

Sum of the percent recovered and the percent loss

Temperature measuring device readings at specified percentages evaporated calculated with arithmetical or graphical procedures

Group 0: 2 to 5 minutes
 Group 1, 2 & 3: 5 to 10 minutes
 Group 4: 5 to 15 minutes

Group 1 & 2: 60 to 100 seconds

Group 0: time from first application of heat to 10% recovered = 3 to 4 minutes
 Group 0, 1, 2, 3 & 4: 4 to 5 mL / min uniform average rate from 5% recovered to 5 mL in flask

Volume of condensate observed in the receiving cylinder when the 5 mL conditions are reached

Volume of condensate observed in the receiving cylinder when the final boiling point is observed

Maximum percent recovered

Volume of residue in the flask expressed as a percentage of the charge volume

Combined Percent Recovery and Percent Residue in the flask

Time from 5 mL in flask to FBP = < 5 minutes

100 minus the Total Recovery

Percent Recovery corrected for barometric pressure

Percent Loss corrected for barometric pressure

Combined Percent Recovery and Percent Residue in the flask corrected for barometric pressure

FIG. X5.2 Percent Evaporated Report Form



D86 – 18

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D86 – 17) that may impact the use of this standard. (Approved Dec. 1, 2018.)

- (1) Updated subsection 7.2.1.1 to clarify the sampling procedure for ambient temperature samples. (2) Updated Table 2 to acknowledge warmer sample temperatures.

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Apéndice 10 Método estándar para punto de inflamación

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.



Designation: D93 – 18



Designation: 34/99

Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester¹

This standard is issued under the fixed designation D93; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

INTRODUCTION

This flash point test method is a dynamic test method which depends on specified rates of heating to be able to meet the precision of the test method. The rate of heating may not in all cases give the precision quoted in the test method because of the low thermal conductivity of some materials. There are flash point test methods with slower heating rates available, such as Test Method D3941 (for paints, resins, and related products, and high viscosity products in the range of 0 °C to 110 °C), where the test conditions are closer to equilibrium.

Flash point values are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods, or with test apparatus different from that specified.

1. Scope*

1.1 These test methods cover the determination of the flash point of petroleum products in the temperature range from 40 °C to 370 °C by a manual Pensky-Martens closed-cup apparatus or an automated Pensky-Martens closed-cup apparatus, and the determination of the flash point of biodiesel in the temperature range of 60 °C to 190 °C by an automated Pensky-Martens closed cup apparatus.

NOTE 1—Flash point determinations above 250 °C can be performed, however, the precision has not been determined above this temperature. For residual fuels, precision has not been determined for flash points above 100 °C. The precision of in-use lubricating oils has not been determined. Some specifications state a D93 minimum flash point below 40 °C, however, the precision has not been determined below this temperature.

1.2 Procedure A is applicable to distillate fuels (diesel, biodiesel blends, kerosine, heating oil, turbine fuels), new and in-use lubricating oils, and other homogeneous petroleum liquids not included in the scope of Procedure B or Procedure C.

1.3 Procedure B is applicable to residual fuel oils, cutback residua, used lubricating oils, mixtures of petroleum liquids with solids, petroleum liquids that tend to form a surface film under test conditions, or are petroleum liquids of such kinematic viscosity that they are not uniformly heated under the stirring and heating conditions of Procedure A.

1.4 Procedure C is applicable to biodiesel (B100). Since a flash point of residual alcohol in biodiesel is difficult to observe by manual flash point techniques, automated apparatus with electronic flash point detection have been found suitable.

1.5 These test methods are applicable for the detection of contamination of relatively nonvolatile or nonflammable materials with volatile or flammable materials.

1.6 The values stated in SI units are to be regarded as the standard.

1.6.1 *Exception*—The values given in parentheses are for information only.

NOTE 2—It has been common practice in flash point standards for many decades to alternately use a C-scale or an F-scale thermometer for temperature measurement. Although the scales are close in increments, they are not equivalent. Because the F-scale thermometer used in this procedure is graduated in 5 °F increments, it is not possible to read it to the 2 °C equivalent increment of 3.6 °F. Therefore, for the purposes of application of the procedure of the test method for the separate temperature scale thermometers, different increments must be used. In this test method, the following protocol has been adopted: When a temperature is intended to be a converted equivalent, it will appear in parentheses following the SI unit, for example 370 °C (698 °F). When a temperature

¹ These test methods are under the joint jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and are the direct responsibility of Subcommittee D02.08 on Volatility. In the IP, these test methods are under the jurisdiction of the Standardization Committee.

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

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is intended to be a rationalized unit for the alternate scale, it will appear after “or,” for example, 2 °C or 5 °F.

1.7 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific warning statements, see 6.4, 7.1, 9.3, 9.4, 11.1.2, 11.1.4, 11.1.8, 11.2.2, and 12.1.2.

1.8 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

D56 Test Method for Flash Point by Tag Closed Cup Tester
D3941 Test Method for Flash Point by the Equilibrium Method With a Closed-Cup Apparatus

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

E1 Specification for ASTM Liquid-in-Glass Thermometers

E300 Practice for Sampling Industrial Chemicals

E502 Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods

2.2 ISO Standards³

Guide 34 General requirements for the competence of reference material producers

Guide 35 Reference material—General and statistical principles for certification

3. Terminology

3.1 Definitions:

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.

3.1.2 *biodiesel blends, n*—a blend of biodiesel fuel with petroleum-based diesel fuel.

3.1.3 *dynamic, adj*—in petroleum products—in petroleum product flash point test methods—the condition where the vapor above the test specimen and the test specimen are not in temperature equilibrium at the time that the ignition source is applied.

3.1.3.1 *Discussion*—This is primarily caused by the heating of the test specimen at the constant prescribed rate with the vapor temperature lagging behind the test specimen temperature.

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, <http://www.ansi.org>.

3.1.4 *equilibrium, n*—in petroleum products—in petroleum product flash point test methods—the condition where the vapor above the test specimen and the test specimen are at the same temperature at the time the ignition source is applied.

3.1.4.1 *Discussion*—This condition may not be fully achieved in practice, since the temperature may not be uniform throughout the test specimen, and the test cover and shutter on the apparatus can be cooler.

3.1.5 *flash point, n*—in flash point test methods, the lowest temperature of the test specimen, adjusted to account for variations in atmospheric pressure from 101.3 kPa, at which application of an ignition source causes the vapors of the test sample to ignite under specified conditions of test.

4. Summary of Test Method

4.1 A brass test cup of specified dimensions, filled to the inside mark with test specimen and fitted with a cover of specified dimensions, is heated and the specimen stirred at specified rates, using one of three defined procedures (A, B, or C). An ignition source is directed into the test cup at regular intervals with simultaneous interruption of the stirring, until a flash is detected (see 11.1.8). The flash point is reported as defined in 3.1.5.

5. Significance and Use

5.1 The flash point temperature is one measure of the tendency of the test specimen to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties which must be considered in assessing the overall flammability hazard of a material.

5.2 Flash point is used in shipping and safety regulations to define *flammable* and *combustible* materials. One should consult the particular regulation involved for precise definitions of these classifications.

5.3 These test methods should be used to measure and describe the properties of materials, products, or assemblies in response to heat and an ignition source under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of these test methods may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

5.4 These test methods provide the only closed cup flash point test procedures for temperatures up to 370 °C (698 °F).

6. Apparatus

6.1 *Pensky-Martens Closed Cup Apparatus (manual)*—This apparatus consists of the test cup, test cover and shutter, stirring device, heating source, ignition source device, air bath, and top plate described in detail in Annex A1. The assembled manual apparatus, test cup, test cup cover, and test cup assembly are illustrated in Figs. A1.1-A1.4, respectively. Dimensions are listed respectively.

6.2 *Pensky-Martens Closed Cup Apparatus (Automated)*⁴—This apparatus is an automated flash point instrument that is capable of performing the test in accordance with Section 11 (Procedure A), Section 12 (Procedure B), and 13 (Procedure C) of these test methods. The apparatus shall use the test cup, test cover and shutter, stirring device, heating source, and ignition source device described in detail in Annex A1.

6.3 *Temperature Measuring Device*—Thermometer having a range as shown in Table 1 and conforming to the requirements prescribed in Specification E1 or in Annex A3, or an electronic temperature measuring device, such as resistance thermometers or thermocouples. The device shall exhibit the same temperature response as the mercury thermometers.

6.4 *Ignition Source*—Natural gas flame, bottled gas flame, and electric ignitors (hot wire) have been found acceptable for use as the ignition source. The gas flame device described in detail in Fig. A1.4 requires the use of the pilot flame described in A1.1.2.3. The electric ignitors shall be of the hot-wire type and shall position the heated section of the ignitor in the aperture of the test cover in the same manner as the gas flame device. (Warning—Gas pressure supplied to the apparatus should not be allowed to exceed 3 kPa (12 in.) of water pressure.)

6.5 *Barometer*—With accuracy of ±0.5 kPa.

NOTE 3—The barometric pressure used in this calculation is the ambient pressure for the laboratory at the time of the test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings and would not give the correct reading for this test.

7. Reagents and Materials

7.1 *Cleaning Solvents*—Use suitable solvent capable of cleaning out the specimen from the test cup and drying the test cup and cover. Some commonly used solvents are toluene and acetone. (Warning—Toluene, acetone, and many solvents are flammable and a health hazard. Dispose of solvents and waste material in accordance with local regulations.)

8. Sampling

8.1 Obtain a sample in accordance with instructions given in Practices D4057, D4177, or E300.

8.2 Ensure at least 75 mL of sample is available so there is sufficient material for the test. When obtaining a sample of residual fuel oil, the sample container shall be from 85 % to

95 % full. For other types of samples, the size of the container shall be chosen such that the container is not more than 85 % full or less than 50 % full prior to any sample aliquot being taken. For biodiesel (B100) samples, a typical one liter container filled to 85 % volume is recommended.

8.3 Successive test specimens can be taken from the same sample container. Repeat tests have been shown to be within the precisions of the method when the second specimen is taken with the sample container at least 50 % filled. The results of flash point determinations can be affected if the sample volume is less than 50 % of sample container capacity.

8.4 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily, to prevent loss of volatile material or possible introduction of moisture, or both. Avoid storage of samples at temperatures in excess of 35 °C or 95 °F. Samples for storage shall be capped tightly with inner seals. Do not make a transfer unless the sample temperature is at least the equivalent of 18 °C or 32 °F below the expected flash point.

8.5 Do not store samples in gas-permeable containers, since volatile material may diffuse through the walls of the enclosure. Samples in leaky containers are suspect and not a source of valid results.

8.6 Samples of very viscous materials shall be heated in their containers, with lid/cap slightly loosened to avoid buildup of dangerous pressure, at the lowest temperature adequate to liquefy any solids, not exceeding 28 °C or 50 °F below the expected flash point, for 30 min. If the sample is then not completely liquefied, extend the heating period for additional 30 min periods as necessary. Then gently agitate the sample to provide mixing, such as orbiting the container horizontally, before transferring to the specimen cup. No sample shall be heated and transferred unless its temperatures is more than 18 °C or 32 °F below its expected flash point. When the sample has been heated above this temperature, allow the sample to cool until its temperature is at least 18 °C or 32 °F below the expected flash point before transferring.

NOTE 4—Volatile vapors can escape during heating when the sample container is not properly sealed.

NOTE 5—Some viscous samples may not completely liquefy even after prolonged periods of heating. Care should be exercised when increasing the heating temperature to avoid unnecessary loss of volatile vapors, or heating the sample too close to the flash point.

8.7 Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted, but it shall not be heated for

⁴ Supporting data regarding a variant of the cover locking mechanism have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1706. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Temperature Measuring Device

Temperature Range	Thermometer Number	Temperature Range	Thermometer Number
	ASTM		IP
-5 °C to 110 °C (20 °F to 230 °F)	9C (9F)	-5 °C to 110 °C	15C
+10 °C to 200 °C (50 °F to 392 °F)	88C (88F)	+20 °C to 150 °C	101C
+90 °C to 370 °C (200 °F to 700 °F)	10C (10F)	+90 °C to 370 °C	16C

prolonged periods or greater than a temperature of 18 °C or 32 °F below its expected flash point.

NOTE 6—If the sample is suspected of containing volatile contaminants, the treatment described in 8.6 and 8.7 should be omitted.

9. Preparation of Apparatus

9.1 Support the manual or automated apparatus on a level steady surface, such as a table.

9.2 Tests are to be performed in a draft-free room or compartment.

NOTE 7—A shield, of the approximate dimensions 460 mm (18 in.) square and 610 mm (24 in.) high, or other suitable dimensions, and having an open front is recommended to prevent drafts from disturbing the vapors above the test cup.

NOTE 8—With some samples whose vapors or products of pyrolysis are objectionable, it is permissible to place the apparatus along with a draft shield in a ventilation hood, the draft of which is adjustable so that vapors can be withdrawn without causing air currents over the test cup during the ignition source application period.

9.3 Prepare the manual apparatus or the automated apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment. (**Warning**—Gas pressure should not be allowed to exceed 3 kPa (12 in.) of water pressure.)

9.4 Thoroughly clean and dry all parts of the test cup and its accessories before starting the test, to ensure the removal of any solvent which had been used to clean the apparatus. Use suitable solvent capable of removing all of the specimen from the test cup and drying the test cup and cover. Some commonly used solvents are toluene and acetone. (**Warning**—Toluene, acetone, and many solvents are flammable. Health hazard. Dispose of solvents and waste material in accordance with local regulations.)

10. Verification of Apparatus

10.1 Adjust the automated flash point detection system (when used) in accordance with the manufacturer's instructions.

10.2 Verify that the temperature measuring device is in accordance with 6.3.

10.3 Verify the performance of the manual apparatus or the automated apparatus at least once per year by determining the flash point of a certified reference material (CRM) such as those listed in Annex A4, which is reasonably close to the expected temperature range of the samples to be tested. The material shall be tested according to Procedure A of these test methods and the observed flash point obtained in 11.1.8 or 11.2.2 shall be corrected for barometric pressure (see Section 14). The flash point obtained shall be within the limits stated in Table A4.1 for the identified CRM or within the limits calculated for an unlisted CRM (see Annex A4).

10.4 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWSs) can be determined along with their control limits. These secondary materials can then be utilized for more frequent performance checks (see Annex A4).

10.5 When the flash point obtained is not within the limits stated in 10.3 or 10.4, check the condition and operation of the apparatus to ensure conformity with the details listed in Annex A1, especially with regard to tightness of the lid (A1.1.2.2), the action of the shutter, the position of the ignition source (A1.1.2.3), and the angle and position of the temperature measuring device (A1.1.2.4). After any adjustment, repeat the test in 10.3 using a fresh test specimen, with special attention to the procedural details prescribed in these test methods.

10.6 The numerical values obtained during the verification check (10.3) shall not be used to provide a bias statement, nor shall they be used to make any correction to the flash points subsequently determined using the apparatus.

PROCEDURE A

11. Procedure

11.1 Manual Apparatus:

11.1.1 Ensure that the sample container is filled to the volume capacity requirement specified in 8.2. Fill the test cup with the test specimen to the filling mark inside of the test cup. The temperature of the test cup and test specimen shall be at least 18 °C or 32 °F below the expected flash point. If too much test specimen has been added to the test cup, remove the excess using a syringe or similar device for withdrawal of fluid. Place the test cover on the test cup and place the assembly into the apparatus. Be sure the locating or locking device is properly engaged. If the temperature measuring device is not already in place, insert the device into its holder.

11.1.2 Light the test flame, and adjust it to a diameter of 3.2 mm to 4.8 mm (0.126 in. to 0.189 in.), or switch on the electric igniter and adjust the intensity in accordance with the manufacturer's instructions. (**Warning**—Gas pressure should not be allowed to exceed 3 kPa (12 in.) of water pressure.) (**Warning**—Exercise care when using a gas test flame. If it should be extinguished it will not ignite the vapors in the test cup, and the gas for the test flame that then enters the vapor space can influence the result.) (**Warning**—The operator should exercise and take appropriate safety precautions during the initial application of the ignition source, since test specimens containing low-flash material can give an abnormally strong flash when the ignition source is first applied.) (**Warning**—The operator should exercise and take appropriate safety precautions during the performance of these test methods. The temperatures attained during these test methods, up to 370 °C (698 °F), are considered hazardous.) (**Warning**—As a safety practice, when using automated or manual apparatus, it is strongly advised, before heating the test cup and specimen, to dip the ignitor to check for the presence of unexpected volatile material.)

11.1.3 Apply the heat at such a rate that the temperature, as indicated by the temperature measuring device, increases 5 °C to 6 °C (9 °F to 11 °F)/min.

11.1.4 Turn the stirring device at 90 r/min to 120 r/min, stirring in a downward direction. (**Warning**—Meticulous attention to all details relating to the ignition source, size of test flame or intensity of the electric ignitor, rate of temperature

increase, and rate of dipping the ignition source into the vapor of the test specimen is desirable for good results.)

11.1.5 Application of Ignition Source:

11.1.5.1 If the test specimen is expected to have a flash point of 110 °C or 230 °F or below, apply the ignition source when the temperature of the test specimen is 23 °C ± 5 °C or 41 °F ± 9 °F below the expected flash point and each time thereafter at a temperature reading that is a multiple of 1 °C or 2 °F. Discontinue the stirring of the test specimen and apply the ignition source by operating the mechanism on the test cover which controls the shutter so that the ignition source is lowered into the vapor space of the test cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its upward position.

11.1.5.2 If the test specimen is expected to have a flash point above 110 °C or 230 °F, apply the ignition source in the manner described in 11.1.5.1 at each temperature increase of 2 °C or 5 °F, beginning at a temperature of 23 °C ± 5 °C or 41 °F ± 9 °F below the expected flash point. (**Warning**—As a safety practice, when using automated or manual apparatus, it is strongly advised that, for an expected flash point above 130 °C, to dip the ignitor every 10 °C throughout the test until the sample temperature reaches 28 °C below the expected flash point and then follow the prescribed dipping procedure. This practice has been shown to reduce the possibility of a fire, and, on average, not to significantly affect the result. A limited study⁵ has shown that this dipping practice has no observable effect on test method repeatability.)

11.1.6 When testing materials to determine if volatile material contamination is present, it is not necessary to adhere to the temperature limits for initial ignition source application as stated in 11.1.5.

11.1.7 When testing materials where the expected flash point temperature is not known, bring the material to be tested and the tester to a temperature of 15 °C ± 5 °C or 60 °F ± 10 °F. When the material is known to be very viscous at this temperature, heat the specimen to a starting temperature as described in 8.6. Apply the ignition source, in the manner described in 11.1.5.1, beginning at least 5 °C or 10 °F higher than the starting temperature.

Note: 9—Flash Point results determined in an “unknown expected flash point mode” should be considered approximate. This value can be used as the expected flash point when a fresh specimen is tested in the standard mode of operation.

11.1.8 Record as the observed flash point the reading on the temperature measuring device at the time ignition source application causes a distinct flash in the interior of the test cup. The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the entire surface of the test specimen. (**Warning**—For certain mixtures containing halogenated hydrocarbons, such as, methylene chloride or trichloroethylene, no distinct flash, as defined, is observed. Instead a significant enlargement of the test flame (not halo effect) and change in color of the test flame from blue to yellowish-orange occurs. Continued heating and testing of

these samples above ambient temperature can result in significant burning of vapors outside the test cup, and can be a potential fire hazard. See Appendix X1 and Appendix X2 for more information.)

11.1.9 When the ignition source is a test flame, the application of the test flame can cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash and shall be ignored.

11.1.10 When a flash point is detected on the first application, the test shall be discontinued, the result discarded, and the test repeated with a fresh test specimen. The first application of the ignition source with the fresh test specimen shall be 23 °C ± 5 °C or 41 °F ± 9 °F below the temperature at which a flash point was detected on the first application.

11.1.11 When a flash point is detected at a temperature which is greater than 28 °C or 50 °F above the temperature of the first application of the ignition source, or when a flash point is detected at a temperature which is less than 18 °C or 32 °F above the temperature of the first application of the ignition source, the result shall be considered approximate, and the test repeated with a fresh test specimen. Adjust the expected flash point for this next test to the temperature of the approximate result. The first application of the ignition source with the fresh test specimen shall be 23 °C ± 5 °C or 41 °F ± 9 °F below the temperature at which the approximate result was found.

11.1.12 When the apparatus has cooled down to a safe handling temperature, less than 55 °C (130 °F), remove the test cover and the test cup and clean the apparatus as recommended by the manufacturer.

Note: 10—Exercise care when cleaning and positioning the lid assembly so not to damage or dislocate the flash detection system or temperature measuring device. See the manufacturer’s instructions for proper care and maintenance.

11.2 Automated Apparatus:

11.2.1 The automated apparatus shall be capable of performing the procedure as described in 11.1, including control of the heating rate, stirring of the test specimen, application of the ignition source, detection of the flash point, and recording the flash point.

11.2.2 Start the automated apparatus in accordance with the manufacturer’s instructions. (**Warning**—Failure to install the sample temperature measuring device correctly, when using automated apparatus, can result in uncontrolled heating of the test portion and potentially a fire. Some automated apparatus include provisions to avoid this occurrence.) The apparatus shall follow the procedural details described in 11.1.3 through 11.1.8.

PROCEDURE B

12. Procedure

12.1 Manual Apparatus:

12.1.1 Ensure that the sample container is filled to the volume capacity requirement specified in 8.2. Fill the test cup with the test specimen to the filling mark inside of the test cup. The temperature of the test cup and test specimen shall be at least 18 °C or 32 °F below the expected flash point. If too much test specimen has been added to the test cup, remove the excess using a syringe or similar device for withdrawal of fluid. Place

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1652. Contact ASTM Customer Service at service@astm.org.

the test cover on the test cup and place the assembly into the apparatus. Be sure the locating or locking device is properly engaged. If the temperature measuring device is not already in place, insert the device into its holder.

12.1.2 Light the test flame and adjust it to a diameter of 3.2 mm to 4.8 mm (0.126 in. to 0.189 in.), or switch on the electric igniter and adjust the intensity in accordance with the manufacturer's instructions. (**Warning**—Gas pressure should not be allowed to exceed 3 kPa (12 in.) of water pressure.) (**Warning**—Exercise care when using a gas test flame. If it should be extinguished it will not ignite the vapors in the test cup and the gas for the test flame that then enters the vapor space can influence the result.) (**Warning**—The operator should exercise and take appropriate safety precautions during the initial application of the ignition source, since test specimens containing low-flash material may give an abnormally strong flash when the ignition source is first applied.) (**Warning**—The operator should exercise and take appropriate safety precautions during the performance of these test methods. The temperatures attained during these test methods, up to 370 °C (698 °F), are considered hazardous.)

12.1.3 Turn the stirring device at 250 r/min \pm 10 r/min, stirring in a downward direction.

12.1.4 Apply the heat at such a rate that the temperature as indicated by the temperature measuring device increases 1 °C to 1.6 °C (2 °F to 3 °F)/min.

12.1.5 Proceed as prescribed in Section 11, with the exception of the preceding requirements for rates of stirring and heating.

12.2 Automated Apparatus:

12.2.1 The automated apparatus shall be capable of performing the procedure as described in 12.1, including control of the heating rate, stirring of the test specimen, application of the ignition source, detection of the flash point, and recording the flash point.

12.2.2 Start the automated apparatus in accordance with the manufacturer's instructions. The apparatus shall follow the procedural details in accordance with 12.1.3 through 12.1.5.

Procedure C

13. Procedure

13.1 *Automated Apparatus*—Ensure that the apparatus is equipped with an electronic measuring system for the detection of the flash point.

13.2 Ensure that the sample container is filled to the volume capacity requirement specified in 8.2. Fill the test cup with the test specimen to the filling mark inside of the test cup. The temperature of the test cup and test specimen shall be at least 24 °C below the expected flash point. If too much test specimen has been added to the test cup, remove the excess using a syringe or similar device for withdrawal of fluid. Place the test cover on the test cup and place the assembly into the apparatus. Be sure the locating or locking device is properly engaged. If the temperature measuring device is not already in place, insert the device into its holder.

13.3 Light the test flame, and adjust it to a diameter of 3.2 mm to 4.8 mm (0.126 in. to 0.189 in.) or switch on the

electric igniter and adjust the intensity in accordance with the manufacturer's instructions. (**Warning**—Gas pressure should not be allowed to exceed 3 kPa (12 in. of water pressure.) (**Warning**—Exercise care when using a gas test flame. If it should be extinguished it will not ignite the vapors in the test cup, and the gas for the test flame that then enters the vapor space can influence the result.) (**Warning**—The operator should exercise and take appropriate safety precautions during the initial application of the ignition source, since test specimens containing low-flash material can give an abnormally strong flash when the ignition source is first applied.) (**Warning**—The operator should exercise and take appropriate safety precautions during the performance of these test methods. The temperatures attained during these test methods, up to 370 °C (698 °F), are considered hazardous.)

13.4 Apply the heat at such a rate that the temperature as indicated by the temperature measuring device increases 3.0 °C/min \pm 0.5 °C/min.

13.5 Turn the stirring device at 90 r/min to 120 r/min, stirring in a downward direction. (**Warning**—Meticulous attention to all details relating to the ignition source, size of test flame, rate of temperature increase, and rate of dipping the ignition source into the vapor of the test specimen is desirable for good results.)

13.6 *Application of Ignition Source*—The first test on the sample shall use an expected flash point of 100 °C.

13.7 Apply the ignition source when the temperature of the test specimen is approximately 24 °C below the expected flash point and each time thereafter at a temperature reading that is a multiple of 2 °C. Discontinue the stirring of the test specimen and apply the ignition source by operating the mechanism on the test cover which controls the shutter so that the ignition source is lowered into the vapor space of the test cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its upward position.

13.8 Record as the flash point the reading on the temperature measuring device at the time the ignition source application causes a distinct flash in the interior of the test cup which is detected by the electronic device.

13.9 The application of the test flame can cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash and shall be ignored.

13.10 When a flash point is detected on the first application, the test shall be discontinued, the result discarded, and the test repeated with a fresh test specimen. The first application of the ignition source with the fresh test specimen shall be approximately 24 °C below the temperature at which a flash point was detected on the first application.

13.11 When a flash point is detected at a temperature which is greater than 30 °C above the temperature of the first application of the ignition source, or when a flash point is detected at a temperature which is less than 16 °C above the temperature of the first application of the ignition source, the result shall be considered approximate, and the test repeated with a fresh test specimen. Adjust the expected flash point for this next test to the temperature of the approximate result. The

first application of the ignition source with the fresh test specimen shall be approximately 24 °C below the temperature at which the approximate result was found.

13.12 When the apparatus has cooled down to a safe handling temperature, less than 55 °C, remove the test cover and the test cup and clean the apparatus as recommended by the manufacturer.

NOTE 11—Exercise care when cleaning and positioning the lid assembly so not to damage or dislocate the flash detection system or temperature measuring device. See the manufacturer’s instructions for proper care and maintenance.

PRECISION, CALCULATION, AND REPORT FOR PROCEDURES A, B, OR C

14. Calculation

14.1 Observe and record the ambient barometric pressure (see Note 3) at the time of the test. When the pressure differs from 101.3 kPa (760 mm Hg), correct the flash point as follows:

$$\text{Corrected flash point} = C + 0.25 (101.3 - K) \quad (1)$$

$$\text{Corrected flash point} = F + 0.06 (760 - P) \quad (2)$$

$$\text{Corrected flash point} = C + 0.033 (760 - P) \quad (3)$$

where:

- C = observed flash point, °C,
- F = observed flash point, °F,
- P = ambient barometric pressure, mm Hg, and
- K = ambient barometric pressure, kPa.

14.2 After correction for barometric pressure, round the temperature to the nearest 0.5 °C (1 °F) and record.

15. Report

15.1 Report the corrected flash point as the ASTM D93, Procedure A or Procedure B or Procedure C Pensky-Martens Closed Cup Flash Point of the test specimen.

16. Precision and Bias (Procedure A)

16.1 *Precision*—The precision of this procedure as determined by the statistical examination of the interlaboratory test results, is as follows:

16.1.1 *Repeatability*—The difference between successive 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 the following values in 1 case in 20.

$$r = AX, \quad (4)$$

$$A = 0.029,$$

X = mean result in °C, and

r = repeatability.

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

$$R = BX, \quad (5)$$

$$B = 0.071,$$

X = mean result in °C, and

R = reproducibility.

16.1.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in these test methods, bias has not been determined.

16.1.4 *Relative Bias*—Statistical evaluation of the data did not detect any significant difference between the reproducibility variances of manual and automated Pensky-Martens flash point results for the samples studied. Evaluation of the data did not detect any significant difference between averages of manual and automated Pensky-Martens flash point for the samples studied with the exception of cycle oil and fuel oil which showed some bias. In any case of dispute, the manual procedure shall be considered the referee test.

NOTE 12—The precision statements were derived on clear liquids only. Refer to the research report⁶ for information regarding relative bias and types of samples. Additional studies are in progress concerning relative bias.

16.1.5 The precision data were developed from a combined 1991 ASTM cooperative test program⁶ using 5 samples of fuel and lubricating oils (Twelve laboratories participated with the manual apparatus and 21 laboratories participated with the automated equipment) and a 1994 IP cooperative test program using 12 fuel samples and 4 pure chemicals. (Twenty-six laboratories participated with manual and automated equipment. The apparatus used either a gas test flame or an electric resistance (hot wire) device for the ignition source. Information on the type of samples and their average flash point are in the research report.⁶

17. Precision and Bias (Procedure B)

17.1 *Precision*—The precision of this procedure, as determined by the statistical examination of the interlaboratory test results, is as follows:

17.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following value in 1 case in 20:

- Residual fuel oil 2 °C
- Other types 5 °C

17.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following value only in 1 case in 20:

- Residual fuel oil 6 °C
- Other types 10 °C

NOTE 13—The precisions of these standards were derived from interlaboratory studies conducted in degrees Celsius.

⁶Supporting data (the results of the 1991 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:S15-1008. Contact ASTM Customer Service at service@astm.org.

17.1.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in these test methods, bias has not been determined.

17.1.4 The precision data for residual fuel oils were developed in a 1996 cooperative test program conducted by the IP using 12 samples of residual fuel and 40 laboratories worldwide using both the manual and automated apparatus. Information on the type of samples and their average flash point are in the research report.

17.1.5 The precision data for other sample types in Procedure B is not known to have been developed in accordance with RR:D02-1007.

NOTE 14—Procedure B was not tested in the 1991 interlaboratory program.

18. Precision and Bias (Procedure C)⁷

18.1 *Precision*—The precision of this procedure, as determined by the statistical examination of the interlaboratory test results, is as follows:

18.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following value in 1 case in 20:

8.4 °C

18.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in 1 case in 20:

14.7 °C

NOTE 15—The precisions of these standards were derived from interlaboratory studies conducted in degrees Celsius.

18.1.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in these test methods, bias has not been determined.

18.1.4 The precision data for biodiesel were developed in a 2008 cooperative interlaboratory test program⁷ using 9 samples of biodiesel (B100) of various source and 17 samples of the same biodiesel dosed with concentrations of alcohol from 0.1 % to 0.3 %. Various automated apparatus in 11 laboratories participated. The precision was calculated on the flash point range from 60 °C to 190 °C. The alcohol concentrations were verified in separate laboratories using EN 14110. Information on the type of samples and their average flash point are in the research report.

19. Keywords

19.1 automated flash point; automated Pensky-Martens closed cup; flammability; flash point; Pensky-Martens closed cup

⁷ Supporting data (the results of the 2008 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1683. Contact ASTM Customer Service at service@astm.org.

ANNEXES

(Mandatory Information)

A1. APPARATUS SPECIFICATIONS⁴

A1.1 A typical assembly of the apparatus, gas heated, is shown in Fig. A1.1. The apparatus shall consist of a test cup, cover, and stove conforming to the following requirements:

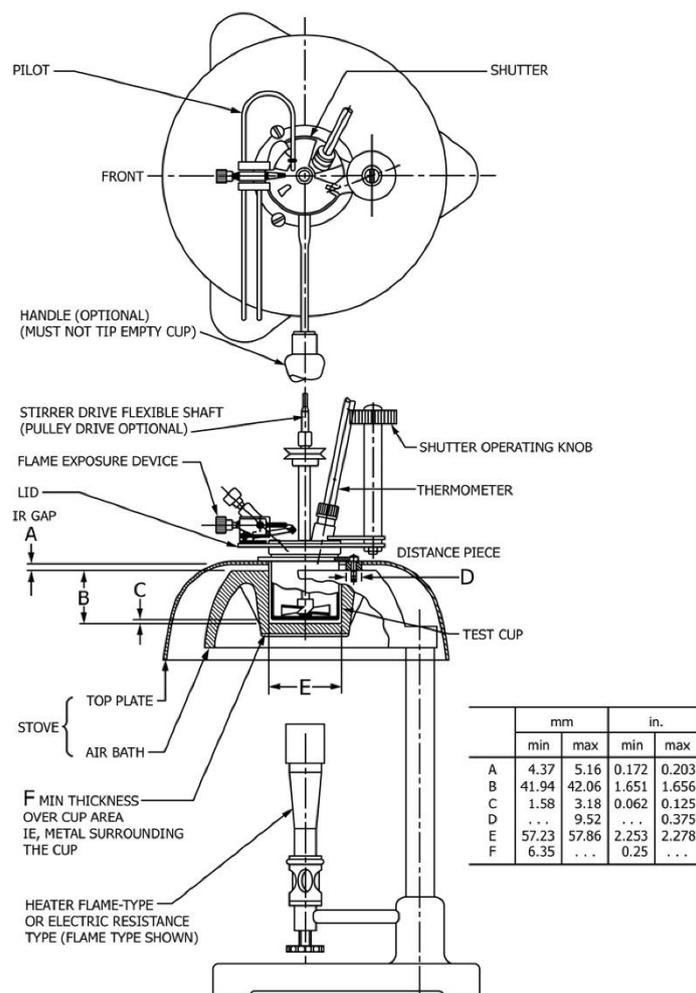
A1.1.1 *Cup*—The cup shall be of brass, or other nonrusting metal of equivalent heat conductivity, and shall conform to the dimensional requirements in Fig. A1.2. The flange shall be equipped with devices for locating the position of the cup in the stove. A handle attached to the flange of the cup is a desirable accessory. The handle shall not be so heavy as to tip over the empty cup.

A1.1.2 *Cover*:

A1.1.2.1 *Cover Proper*—The cover shown in Fig. A1.3 shall be of brass (A1.1.1) and shall have a rim projecting downward almost to the flange of the cup. The rim shall fit the outside of the cup with a clearance not exceeding 0.36 mm (0.014 in.) on the diameter. There shall be a locating or locking device, or both, engaging with a corresponding device on the cup. The upper edge of the cup shall be in close contact with the inner face of the cover throughout its circumference.

A1.1.2.2 *Shutter*—The cover shall be equipped with a brass shutter (Fig. A1.1 and Fig. A1.4), approximately 2.4 mm ($\frac{3}{32}$ in.) thick, operating on the plane of the upper surface of the cover. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the cover between two stops, so placed, that when in one extreme position, the openings *A*, *B*, and *C* in the cover are completely closed, and when in the other extreme position, these openings are completely opened. The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme, the three cover openings shall be exactly open and the tip of the exposure tube shall be fully depressed.

A1.1.2.3 *Flame-Ignition Device*—The flame-ignition device (Fig. A1.4) shall have a tip with an opening 0.69 mm to 0.79 mm (0.027 in. to 0.031 in.) in diameter. This tip shall be made preferably of stainless steel, although it may be fabricated of other suitable metals. The flame-exposure device shall be equipped with an operating mechanism which, when the shutter is in the open position, depresses the tip so that the



NOTE 1—Lid assembly can be positioned either right or left-handed.

FIG. A1.1 Pensky-Martens Closed Flash Tester

center of the orifice is between the planes of the under and upper surfaces of the cover proper at a point on a radius passing through the center of the larger opening A (Fig. A1.3). An electric ignitor is also suitable. The electric ignitors shall be of the electric resistance (hot-wire) type and shall position the heated section of the ignitor in the aperture of the test cover in the same manner as the gas flame device.

A1.1.2.4 *Pilot Flame*—A pilot flame shall be provided for automatic relighting of the exposure flame. A bead 4 mm (5/32 in.) in diameter can be mounted on the cover so that the

size of the test flame can be regulated by comparison. The tip of the pilot flame shall have an opening the same size as the tip of the flame exposure device (0.69 mm to 0.79 mm (0.027 in. to 0.031 in.) in diameter).

A1.1.2.5 *Stirring Device*—The cover shall be equipped with a stirring device (Fig. A1.4) mounted in the center of the cover and carrying two 2-bladed metal propellers. In Fig. A1.4 lower propeller is designated by the letters L, M, and N. This propeller shall measure approximately 38 mm from tip to tip, with each of its two blades 8 mm in width with a pitch of 45°.

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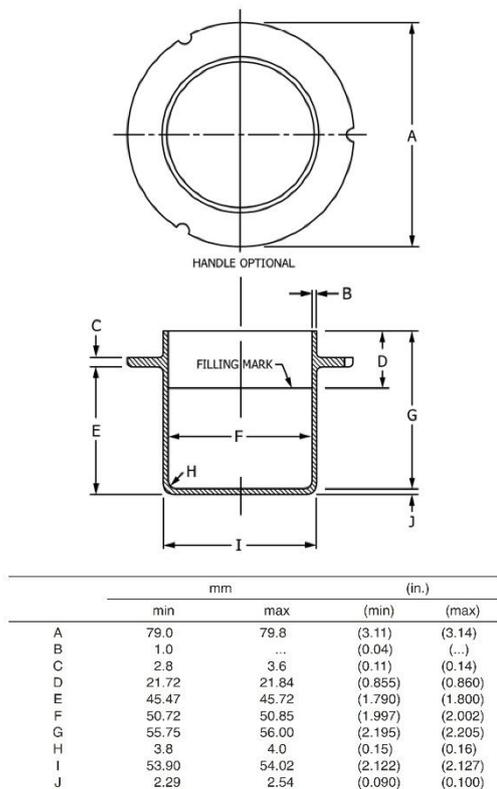


FIG. A1.2 Test Cup

The upper propeller is designated by the letters A, C, and G. This propeller measures approximately 19 mm, tip to tip, each of its two blades is also 8 mm in width with a pitch of 45°. Both propellers are located on the stirrer shaft in such a manner that, when viewed from the bottom of the stirrer, the blades of one propeller are at 0° and 180° while the blades of the other propeller are at 90° and 270°. A stirrer shaft may be coupled to the motor by a flexible shaft or a suitable arrangement of pulleys.

A1.1.2.6 *Stove*—Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. The stove shall consist of an air bath and a top plate on which the flange of the cup rests.

A1.1.2.7 *Air Bath*—The air bath shall have a cylindrical interior and shall conform to the dimensional requirements in Fig. A1.1. The air bath may be either a flame or electrically heated metal casting (A1.1.2.8), or an electric-resistance element (A1.1.2.9). In either case, the air bath must be suitable for use at the temperatures to which it will be subjected without deformation.

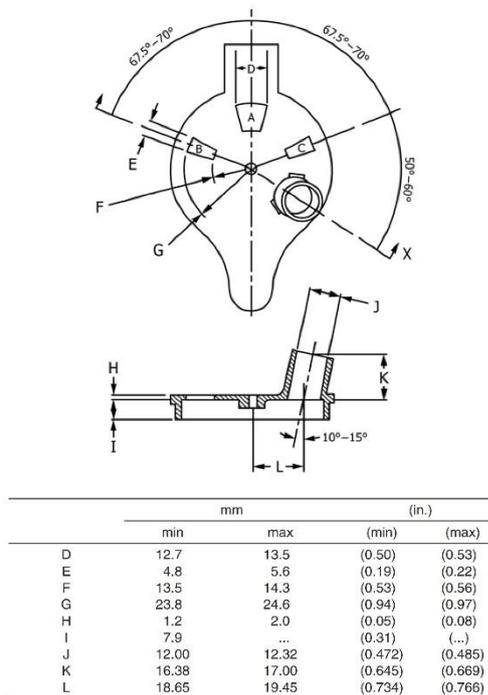
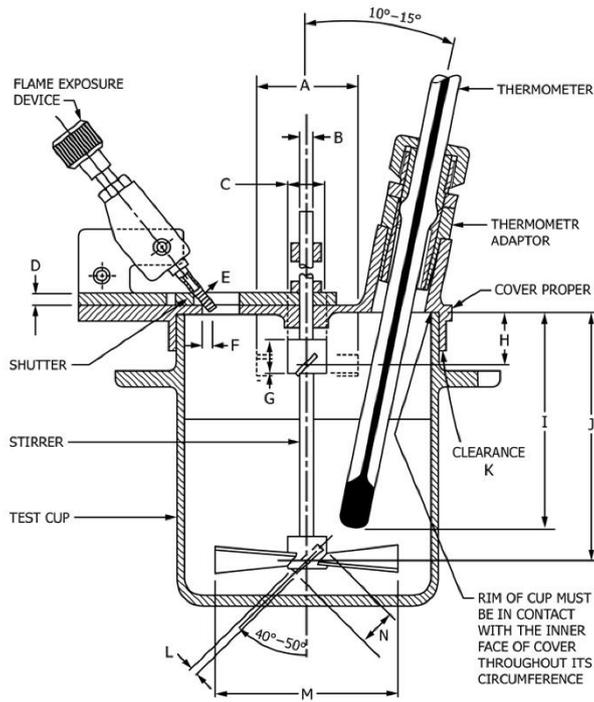


FIG. A1.3 Cover Proper

A1.1.2.8 *Heater, Flame or Electric*—If the heating element is a flame or an electric heater, it shall be so designed and used that the temperatures of the bottom and the walls are approximately the same. In order that the air bath internal surfaces should be at a uniform temperature, it should not be less than 6.4 mm (1/4 in.) in thickness unless the heating element is designed to give equal heat flux densities over all the wall and bottom surfaces.

A1.1.2.9 *Heater, Electric Resistance*—If the heater is of the electric resistance type, it shall be constructed so that all parts of the interior surface are heated uniformly. The wall and bottom of the air bath shall not be less than 6.4 mm (1/4 in.) in thickness unless the resistance heating elements are distributed over at least 80 % of the wall and all the bottom of the air bath. A heater having such a distribution shall have the heating elements positioned at least 4.0 mm (5/32 in.) away from the internal surface of the air bath in conjunction with a minimum thickness of 1.58 mm (1/16 in.) for the wall and bottom of the air bath.

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	mm		(in.)	
	min	max	(min)	(max)
A	18.3	19.8	(0.72)	(0.78)
B	2.38	3.18	(0.094)	(0.125)
C	7.6	8.4	(0.30)	(0.33)
D	2.0	2.8	(0.08)	(0.11)
E	0.69	0.79	(0.027)	(0.031)
F	2.0	2.8	(0.08)	(0.11)
G	6.4	10.4	(0.25)	(0.41)
H	9.6	11.2	(0.38)	(0.44)
I ^A	43.0	46.0	(1.69)	(1.81)
J	50.0	51.6	(1.97)	(2.03)
K	...	0.36	(...)	(0.014)
L	1.22	2.06	(0.048)	(0.08)
M	31.8	44.4	(1.25)	(1.75)
N	7.6	8.4	(0.30)	(0.33)

^A Includes tolerance for length of thermometer given in Specification E1.

FIG. A1.4 Test Cup and Cover Assembly

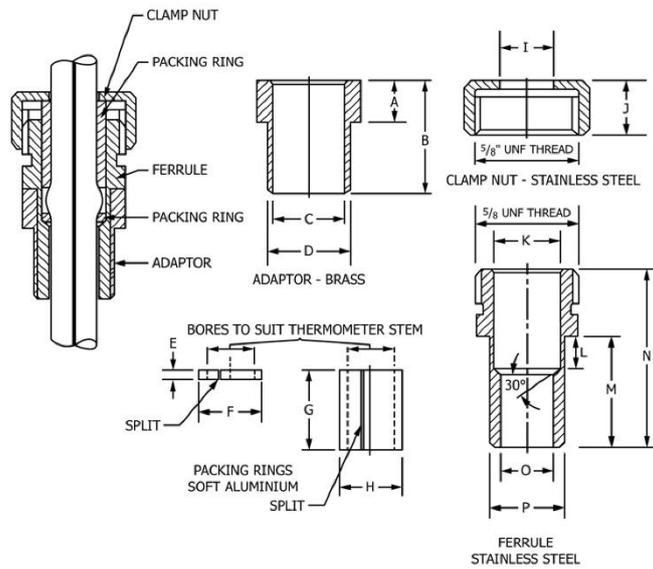
A1.1.2.10 *Top Plate*—The top plate shall be of metal, and shall be mounted with an air gap between it and the air bath. It may be attached to the air bath by means of three screws and

spacing bushings. The bushings should be of proper thickness to define an air gap of 4.8 mm ($\frac{3}{16}$ in.), and they shall be not more than 9.5 mm ($\frac{3}{8}$ in.) in diameter.

A2. MANUFACTURING STANDARDIZATION OF THERMOMETER AND FERRULE

A2.1 The low-range thermometer, which conforms also to the specification for the cup thermometer in the tag closed tester (Test Method D56) and which frequently is fitted with a metal ferrule intended to fit the collar on the cover of the tag flash tester, can be supplemented by an adapter (Fig. A2.1) to be used in the larger diameter collar of the Pensky-Martens apparatus. Differences in dimensions of these collars, which do not affect test results, are a source of unnecessary trouble to manufacturers and suppliers of instruments, as well as to users.

A2.2 Dimensional requirements are shown in Fig. A2.1. Conformity to these requirements is not mandatory, but is desirable to users as well as suppliers of Pensky-Martens testers.



	mm		(in.)	
	min	max	(min)	(max)
A	6.20	6.50	(0.244)	(0.256)
B	17.0	18.0	(0.67)	(0.71)
C	9.80	9.85	(0.386)	(0.388)
D	11.92	12.24	(0.469)	(0.482)
E	1.40	1.65	(0.055)	(0.065)
F	8.56	8.61	(0.337)	(0.339)
G	12.4	13.0	(0.49)	(0.57)
H	8.56	8.61	(0.337)	(0.339)
I	8.1	8.6	(0.32)	(0.34)
J	9.9	10.7	(0.39)	(0.42)
K	8.64	8.69	(0.340)	(0.342)
L	5.1	5.6	(0.20)	(0.22)
M	17.0	17.5	(0.67)	(0.69)
N	27.4	28.2	(1.08)	(1.11)
O	7.11	7.16	(0.280)	(0.282)
P	9.73	9.78	(0.383)	(0.385)

FIG. A2.1 Dimensions for Thermometer Adapter, Ferrule, and Packing Ring

A3. THERMOMETER SPECIFICATIONS

A3.1 See Fig. A3.1 and Tables A3.1-A3.4.

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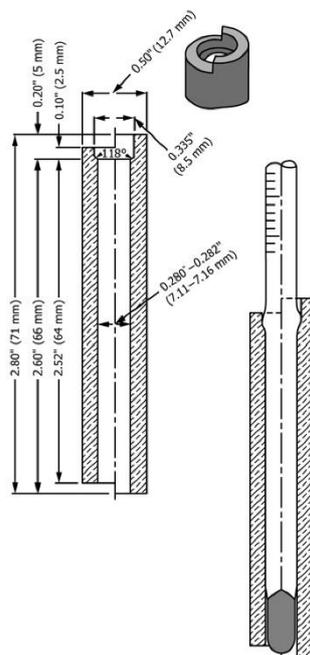


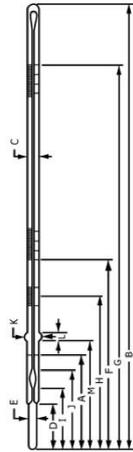
FIG. A3.1 Test Gage for Checking Enlargements on Thermometers

TABLE A3.1 IP Thermometer Specifications

NOTE 1—The stem shall be made with an enlargement having a diameter of 1.5 mm to 2.0 mm greater than the stem and a length of 3 mm to 5 mm, the bottom of the enlargement being 64 mm to 66 mm from the bottom of the bulb. These dimensions shall be measured with the test gage shown in Fig. A3.1.

Name	IP 15C	IP 16C	IP 101C
	Pensky-Martens Low	Pensky-Martens High	Pensky-Martens Medium
Range	-5 °C to +110 °C	90 °C to 370 °C	20 °C to 150 °C
Graduation	0.5 °C	2 °C	1 °C
Immersion, mm	57	57	57
Overall length ±5 mm	290	280 ± 10	290
Stem diameter, mm	6.0 to 7.0	6.0 to 7.0	6.0 to 7.0
Bulb shape	cylindrical	cylindrical	cylindrical
Bulb length, mm	9 to 13	7 to 10	9 to 13
Bulb diameter, mm	not less than 5.5 and not greater than stem	not less than 4.5 and not greater than stem	not less than 5.5 and not greater than stem
Length of graduated portion, mm	140 to 175	143 to 180	140 to 175
Distance bottom of bulb to, mm	0 °C 85 to 95	90 °C 80 to 90	20 °C 85 to 95
Longer lines at each	1 °C and 5 °C	10 °C and 20 °C	5 °C
Figured at each	5 °C	20 °C	5 °C
Expansion chamber	required	required	required
Top finish	ring	ring	ring
Scale error not to exceed ±	0.5 °C	1 °C to 260 °C 2 °C above 260 °C	1 °C
See notes	1 and Table A3.2 for emergent stem temperatures	1 and Table A3.2 for emergent stem temperatures	1 and Table A3.2 for emergent stem temperatures

TABLE A3.2 Specifications for ASTM Thermometers
All dimensions are in millimetres.
See Table A3.3 for Standardization Temperature.



ASTM Number and Name	Range	For Test Interval	Graduations			Scale Error, max	Special Inscription	Expansion Chamber	Stem OD	Bulb		Scale Location			Ice Point Scale		Contraction Chamber		Stem Enlargement	
			Sub-divisions	Long Lines at Each	Number at Each					Length	OD	Bottom of Bulb to Line at	Distance to Line at	Bottom of Bulb to Line at	Range	Bottom of Bulb to Ice Point	Distance to Bottom, min	Distance to Top, max	OD	Length
9C-62	-5 °C to +110 °C		0.5 °C	1 °C	5 °C	0.5 °C	ASTM	160 °C	6.0	9.0	0 °C	85	100 °C							
Pensky- Martens Low- Temp Tester 9F-62	(20 °F to 230 °F)		1 °F	5 °F	10 °F	1 °F	9C or 9F 57 mm IMM	320 °F	7.0	13	32 °F	99	212 °F							
10C-62 Pensky- Martens High- Temp Tester 10F-62	(200 °F to 700 °F)		2 °C	10 °C	20 °C	2 °C	ASTM 10C or 10F 57 mm IMM	350 °F	6.0	8.0	110 °C	86 99	360 °C 680 °F							

1 The length of the enlargement, and the distance from the bottom of the enlargement to the bottom of the bulb shall be measured with the test gage shown in Fig. A3.1.
 a Scale error: 1 °C up to 260 °C; 2 °C over 260 °C.
 c An expansion chamber is provided for relief of gas pressure to avoid distortion of the bulb at higher temperatures. It is not for the purpose of joining mercury separations; and under no circumstances should the thermometer be heated above the highest temperature reading.
 d Scale error: 2.5 °F up to 500 °F; 3.5 °F over 500 °F.

TABLE A3.3 Standardization Temperatures

NOTE 1—The emergent column temperatures are those attained when using the thermometers in the test equipment for which the thermometers were originally designed. In some cases these temperatures are markedly different from those realized during standardization.

Temperature	Average Temperature of Emergent Column	Temperature	Average Temperature of Emergent Column	Temperature	Average Temperature of Emergent Column	Temperature	Average Temperature of Emergent Column
Thermometer 9C (-5 °C to + 100 °C)		Thermometer 9F (20 °F to 230 °F)		Thermometer 10C (90 °C to 370 °C)		Thermometer 10F (200 °F to 700 °F)	
0 °C	19 °C	32 °F	66 °F	100 °C	61 °C	212 °F	141 °F
35 °C	28 °C	100 °F	86 °F	200 °C	71 °C	390 °F	159 °F
70 °C	40 °C	160 °F	106 °F	300 °C	87 °C	570 °F	180 °F
105 °C	50 °C	220 °F	123 °F	370 °C	104 °C	700 °F	220 °F
IP 15C (-7 °C to 110 °C)		IP 15F (20 °F to 230 °F)		IP 16C (90 °C to 370 °C)		IP 16F (20 °F to 700 °F)	
0 °C	19 °C	32 °F	66 °F	100 °C	61 °C	200 °F	140 °F
20 °C	20 °C	70 °F	70 °F	150 °C	65 °C	300 °F	149 °F
40 °C	31 °C	100 °F	86 °F	200 °C	71 °C	400 °F	160 °F
70 °C	40 °C	150 °F	104 °F	250 °C	78 °C	500 °F	175 °F
100 °C	48 °C	212 °F	118 °F	300 °C	87 °C	600 °F	195 °F
				350 °C	99 °C	700 °F	220 °F

TABLE A3.4 Specifications for Medium-Range Pensky-Martens

ASTM No. 88F (88C) Vegetable Oil Flash Thermometer	Medium-Range Pensky-Martens	
Name	5	
Reference Fig. No.	5	
Range	10 °C–200 °C	50 °F–392 °F
For test at		
A Immersion, mm	57	
Graduations:		
Subdivisions	1 °C	2 °F
Long lines at each	5 °C	10 °F
Numbers at each	10 °C	20 °F
Scale error, max	1 °C	2 °F
Special inscription	ASTM 88F (88C) 57 mm IMM	
Expansion chamber:		
Permit heating to	250 °C	490 °F
B Total length, mm	282 to 292	
C Stem OD, mm	6.0 to 7.0	
D Bulb length, mm	9	
E Bulb OD, mm	>stem	
Scale location:		
Bottom of bulb to line at	10 °C	50 °F
F Distance, mm	75 to 90	
G Length of graduated portion, mm	143 to 177	
Ice-point scale:		
Range		
H Bottom of bulb to ice-point, mm		
Contraction chamber:		
I Distance to bottom, min, mm		
J Distance to top, max, mm		
Stem enlargement:		
K OD, mm	7.5 to 8.5	
L Length, mm	2.5 to 5.0 ^A	
M Distance to bottom, mm	64 to 66	

^A Bulb OD shall be greater than 4.5 mm and less than the outside diameter of the stem (C).

A4. VERIFICATION OF APPARATUS PERFORMANCE

A4.1 *Certified Reference Material (CRM)*—CRM is a stable, pure (99 + mole % purity) hydrocarbon or other stable petroleum product with a method-specific flash point established by a method-specific interlaboratory study following ASTM RR:D02-1007 guidelines or ISO Guide 34 and 35.

A4.1.1 Values of the flash point corrected for barometric pressure for some reference materials and their typical limits are given in Table A4.1⁸ (see Note A4.1). Suppliers of CRMs will provide certificates stating the method-specific flash point for each material of the current production batch. Calculation of the limits for these other CRMs can be determined from the reproducibility value of these test methods multiplied by 0.7. This value provides a nominal coverage of at least 90 % with 95 % confidence.

Note: A4.1—Materials, purities, flash point values, and limits stated in Table A4.1 were developed in an ASTM interlaboratory program to determine suitability of use for verification fluids in flash point test methods. Other materials, purities, flash point values, and limits can be suitable when produced according to the practices of ASTM RR:D02-1007 or ISO Guides 34 and 35. Certificates of performance of such

⁸ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:S15-1010. Contact ASTM Customer Service at service@astm.org.

TABLE A4.1 D93 Typical Flash Point Values and Typical Limits for CRM

Note 1—Supporting data for the interlaboratory study to generate the flash point in Table A4.1 can be found in research report RR:S15-1010.⁸

Hydrocarbon	Purity, mole %	Flash Point, °C	Tolerance Limits (0.7R), °C
<i>n</i> -decane	99 +	52.8	2.6
<i>n</i> -undecane	99 +	68.7	3.4
<i>n</i> -tetradecane	99 +	109.3	5.4
<i>n</i> -hexadecane	99 +	133.9	6.7

materials should be consulted before use, as the flashpoint value will vary dependent on the composition of each CRM batch.

A4.2 *Secondary Working Standard (SWS)*—SWS is a stable, pure (99 + mole % purity) hydrocarbon, or other petroleum product whose composition is known to remain appreciably stable.

A4.2.1 Establish the mean flash point and the statistical control limits (3σ) for the SWS using standard statistical techniques. (See ASTM MNL 7).⁹

⁹ *Manual on Presentation of Data and Control Chart Analysis, ASTM MNL, 6th ed.*, ASTM International, W. Conshohocken, 1990.

APPENDIXES
(Nonmandatory Information)
X1. FLASH POINT MASKING PHENOMENON

X1.1 A condition during flash point testing can occur with certain mixtures whereby the nonflammable component of the sample tends to inert the vapor space above the liquid, thus preventing a flash. Under this condition, the flash point of the material is masked resulting in the reporting of incorrect high flash point or no flash point.

X1.2 This flash point masking phenomenon most frequently occurs with ignitable liquids that contain certain halogenated hydrocarbons such as dichloromethane (methylene chloride) and trichloroethylene.

X1.3 Under this condition, no distinct flash as defined in 3.1.5 of these test methods is observed. Instead a significant enlargement of the test flame and a change in the color of the

test flame from blue to yellow-orange laminar flame is observed.

X1.4 Under this condition, continued heating and testing for flash point at temperatures above ambient temperature, have resulted in significant burning of the ignitable vapor outside the test cup, often above the test flame. This can be a potential fire hazard if not recognized.

X1.5 It is recommended that if this condition is encountered during the flash point testing of these type of materials, testing should be discontinued.

X1.6 Further commentaries regarding flash point test and flammability of mixtures can be found in Test Method B502.

X2. FLASH POINT TEST AND FLAMMABILITY OF MIXTURES

X2.1 While the flash point can be used to indicate the flammability of liquid materials for certain end uses, flash point does not represent the minimum temperature at which a material can evolve flammable vapors.

X2.2 There are instances with pure materials where the absence of a flash point does not ensure freedom from flammability. Included in this category are materials that require large diameters for flash propagation, such as trichloroethylene. This material will not propagate a flame in apparatus the size of a flash point tester, however, its vapors are flammable and will burn when ignited in apparatus of adequate size.

X2.3 When a liquid contains flammable and nonflammable components, there are cases where this liquid can evolve

flammable vapors under certain conditions and yet will not exhibit a close-cup flash point. This phenomenon is noted when a nonflammable component is sufficiently volatile and present in sufficient quantity to inert the vapor space of the closed cup, thus preventing a flash. In addition, there are certain instances where an appreciable quantity of the nonflammable component will be present in the vapor, and the material will exhibit no flash point.

X2.4 Liquids containing a highly volatile nonflammable component or impurity, which exhibit no flash point because of the influence of the nonflammable material, may form flammable mixtures if totally flash vaporized in air in the proper proportions.

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D93 – 16a) that may impact the use of this standard. (Approved July 1, 2018.)

(1) Revised subsection 9.2, removing the statement on reliability of tests made in a laboratory hood.

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ANEXO 4

**FACULTAD INGENIERÍA QUÍMICA
CARRERA INGENIERÍA QUÍMICA
UNIDAD DE TITULACIÓN**

Guayaquil, 14 de agosto del 2019

**Ing. Luis Alberto Bonilla Abarca
Director De La Carrera De Ingeniería Química
Facultad De Ingeniería Química
Universidad De Guayaquil**

Ciudad. -

De mis consideraciones:

Envío a Ud. el Informe correspondiente a la tutoría realizada al Trabajo de Titulación "OBTENCIÓN DE ÉSTER METÍLICO (BIODIESEL), MEDIANTE REACCIÓN DE TRANSESTERIFICACIÓN DEL ACEITE DE MORINGA OLEÍFERA" de los estudiantes **Guerrero De La A Bryan Nivaldo y Kocher Solano Karla Nicole**, indicando que han cumplido con todos los parámetros establecidos en la normativa vigente:

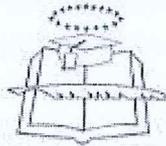
- El trabajo es el resultado de una investigación.
- El estudiante demuestra conocimiento profesional integral.
- El trabajo presenta una propuesta en el área de conocimiento.
- El nivel de argumentación es coherente con el campo de conocimiento.

Adicionalmente, se adjunta el certificado de porcentaje de similitud y la valoración del trabajo de titulación con la respectiva calificación.

Dando por concluida esta tutoría de trabajo de titulación, **CERTIFICO**, para los fines pertinentes, que los estudiantes están aptos para continuar con el proceso de revisión final.

Atentamente,

Ing. Carmen Llerena Ramírez
CI: 0913777058



Universidad de Guayaquil

ANEXO 11

**FACULTAD DE INGENIERÍA QUÍMICA
CARRERA DE INGENIERÍA QUÍMICA
UNIDAD DE TITULACIÓN**

Guayaquil, 25 de agosto de 2019.

CERTIFICACIÓN DEL TUTOR REVISOR

Habiendo sido nombrado la Ing. Sandra Peña Murillo, MSc. tutor revisor del trabajo de titulación **"OBTENCIÓN DE ÉSTER METÁLICO (BIODIESEL), MEDIANTE REACCIÓN DE TRANSESTERIFICACIÓN DEL ACEITE DE MORINGA OLEÍFERA"**, certifico que el presente trabajo de titulación, elaborado por *Guerrero De La a Bryan Nivaldo; y Kocher Solano Karla Nicole*, con mi respectiva supervisión como requerimiento parcial para la obtención del título de Ingeniero Químico, en la Carrera de Ingeniería Química, Facultad de Ingeniería Química, ha sido REVISADO Y APROBADO en todas sus partes, encontrándose apto para su sustentación.

Ing. Sandra Peña Murillo, MSc.

C.I. 0917228801



ANEXO 6

Universidad de Guayaquil

FACULTAD INGENIERÍA QUÍMICA
CARRERA INGENIERÍA QUÍMICA
UNIDAD DE TITULACIÓN

CERTIFICADO PORCENTAJE DE SIMILITUD

Habiendo sido nombrado Ing. Carmen Emperatriz Llerena Ramírez, tutor del trabajo de titulación certifico que el presente trabajo de titulación ha sido elaborado por Guerrero De La A Bryan Nivaldo y Kocher Solano Karla Nicole, con mi respectiva supervisión como requerimiento parcial para la obtención del título de Ingeniero Químico.

Se informa que el trabajo de titulación: "OBTENCIÓN DE ÉSTER METÁLICO (BIODIESEL), MEDIANTE REACCIÓN DE TRANSESTERIFICACIÓN DEL ACEITE DE MORINGA OLEÍFERA", ha sido orientado durante todo el periodo de ejecución en el programa anti plagio (URKUND quedando el 2% de coincidencia.

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