



A Honeywell Company

Smoke Point Determination of Kerosene and Aviation Turbine Fuel Using an Automated Method

UOP Method 1000-13

Scope

This method is for determining the smoke point of kerosene and aviation turbine fuel utilizing an automated instrument with a camera capable of digitalizing the flame image. Smoke point is defined as the maximum height, in millimeters, of a smokeless flame of fuel burned in a wick-fed lamp of specified design. In general, higher aromatic concentration of a fuel will produce a shorter flame height, while low aromatic concentration will produce a higher flame height with lower smoke-producing tendency. The automated method demonstrates improved precision and removes analyst subjectivity of visually observing flame height by manual smoke point methods.

References

ASTM Method D1322, "Standard Test Method for Smoke Point of Kerosine and Aviation Turbine Fuel" www.astm.org

ASTM Practice D4057 "Practice for Manual Sampling of Petroleum and Petroleum Products" www.astm.org

ASTM Practice D6299, "Standard Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance" www.astm.org

UOP Method 666, "Precision Statements in UOP Methods"

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

An automated smoke point analyzer is set up according to the manufacturer's specifications. The sample is burned in an enclosed wick-fed lamp, calibrated against reference blends of known smoke point. A digital camera, capable of resolution to the nearest 0.05 mm, captures images of the flame while the flame height is being adjusted. Three consecutive measurements are recorded with the results averaged, multiplied by the appropriate correction factor, and reported to the nearest 0.1 mm.

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).
--

© COPYRIGHT 2012, 2013 UOP LLC. All rights reserved.

Nonconfidential UOP Methods are available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, USA. The UOP Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Automated smoke point analyzer, Model SP 10, AD systems. Follow the manufacturer's recommendations for maintaining a supply of spare parts and consumables, such as wicks. Any use of an alternative instrument must be tested to determine if it provides equivalent sensitivity and precision.

Wick extractor, AD systems, Cat. No. NA001-102

Wick trimmer set, AD systems, Cat. No. AK220-001

Wick tube, AD systems, Cat. No. AD220-002

Barometer, accuracy of at least ± 0.5 kPa, Traceable Precision Dial Barometer, VWR, Cat. No. 15551-024

Candle, AD systems, Cat. No. AD220-001

Desiccator, with plate, Fisher Scientific, Cat. No. 08-615B

Graduated cylinder, VWR, Cat. No. 89000-266

Pipet, volumetric, Class A, 5-, 10-, and 20-mL, respectively, VWR, Cat. Nos. 89045-064, -074, -76, respectively

Razor blade, VWR, Cat. No. 55411-050

Soxhlet extraction assembly, borosilicate glass, comprised of a 40-mm ID extraction tube with top 40/50 joint, 250-mL flask and condenser, Fisher Scientific, Cat. No. 09-556B

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

The following items are required to perform the analysis. Additional reagents and materials may be required depending on the specific instrument used. Where different reagents are specified in the instrument manual, follow the manufacturer's recommendations.

Desiccant, Drierite, indicating, 10- to 20-mesh, Fisher Scientific, Cat. No. 07-578-4A

Extracted wicks, AD systems, Cat. No. AC220-001

Methanol, anhydrous, Burdick & Jackson, Cat. No. AS230-1,

Toluene, anhydrous, Burdick & Jackson, Cat. No. AS347-1

2,2,4-Trimethylpentane, Burdick & Jackson, Cat. No. LP362-4

Procedure

The analyst is expected to be familiar with general laboratory practices and the equipment being used. Dispose of used reagents, materials, and samples in an environmentally safe manner according to local regulations.

Preparation of Apparatus

Set up the SP 10 automated smoke point analyzer and candle according to the manufacturer's instructions.

1. First, the automated smoke point instrument must be placed on a smooth and level surface in a draft-free room. If the room is not completely draft-free, a box must be constructed of heat resistant material, open at the front, painted dull black, with a height no less than 150 mm above the top of the chimney in order to eliminate any excess movement of the flame caused by a draft.
2. Extract all the wicks for a minimum of 25 cycles in a Soxhlet Extractor, using a mixture of equal volumes of toluene and anhydrous methanol.
3. Allow the wicks to partially dry in a hood.
4. Place the wicks in an oven (explosion proof and/or forced draft) for 30 minutes at 100-110°C. Once the wicks are dry, place them in a desiccator until used.

Calibration of Apparatus

Calibrate the instrument by testing the seven reference fuel blends listed in Table 1, using the steps listed in *Smoke Point Determination*. Each calibration test with the corresponding barometric pressure observed at the time of analysis is stored in the instrument's calibration database. References only need to be run when a change of more than ± 7.0 hPa occurs in the barometric pressure from values already stored in the database. Smoke point values of reference blends are used in calculating the correction factor, which is determined by the instrument. Accurately prepare the reference fuel blends, in agreement with the compositions provided in Table 1, utilizing calibrated pipettes or burettes.

- The instrument accepts pressure entries in hectopascals (hPa), rather than the more common kilopascals (kPa). Convert kPa to hPa using Equation 1:

$$H = 10 K \quad (1)$$

where:

H = pressure in hPa

K = pressure in kPa

Table 1
Reference Fuel Blends for Calibration

2,2,4-Trimethylpentane, liquid volume-%	Toluene, liquid volume-%	Smoke point height at 101.3 kPa
60	40	14.7
75	25	20.3
80	20	22.7
85	15	25.8
90	10	30.2
95	5	35.4
100	0	42.8

This calculation is performed by the instrument software. The calculation is shown in Equation 2 for reference:

$$F = \frac{\frac{A}{B} + \frac{C}{D}}{2} \quad (2)$$

where:

- A** = the standard smoke point of the first reference fuel blend
- B** = the smoke point determined for the first reference fuel blend
- C** = the standard smoke point of the second reference fuel blend
- D** = the smoke point determined for the second reference fuel blend
- F** = correction factor

A quality control (QC) sample and control chart should be utilized for verification that the instrument is performing satisfactorily each day testing is completed. Ensure that the QC sample is comparable with fuel(s) routinely tested by the laboratory to confirm the instrument is in statistical control following guidelines given in ASTM Practice D6299, "Standard Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance." The instrument must be recalibrated if a QC data point exceeds the control limits.

Smoke Point Determination

1. Measure 20 ml of sample into a 25-ml graduated cylinder.
 - As little as 10 mL of sample may be used if the amount of available sample is limited.
2. Pour the sample from the graduated cylinder into the base of the candle, with the overflow port on the top side of the candle.
3. Utilizing a wick trimmer assembly, carefully pull an extracted and dried wick, at least 125 mm in length, through the wick tube with the wick puller without twisting.
 - It is advisable to re-soak the burning end of the wick in the sample after the wick is inserted in the wick tube.
4. Cut off the excess wick from the wick spacer with a razor blade. After removing the wick spacer, cut off any frayed portions of the wick as they can affect the smoke point reading.
5. The wick tube is inserted into the wick candle and screwed together until finger tight.
6. Allow the wick to draw up the sample for at least 10 minutes.
7. Once the wick is visibly soaked in the sample, place the candle on the elevator platform in the circular divot provided.
8. Select the "smoke point" icon on the main screen. Enter in all of the sample details and current barometric pressure (in hPa). Begin the analysis by pressing the "Start" icon.
 - The instrument accepts pressure entries in hectopascals (hPa), rather than the more common kilopascals (kPa), see Equation 1.
9. The wick candle is raised into the lamp and ignited.
10. The candle is adjusted to automatically provide a flame height of 10 mm and burns for 5 min.
11. Once the 5-minute stabilization time is complete, the flame height is increased until a smoky tail is present and is then decreased. When the flame shape corresponds to position B, as described in ASTM Method D1322, "Standard Test Method for Smoke Point of Kerosine and Aviation Turbine Fuel," Figure 2, the image is captured in digital format.
12. Three consecutive measurements, as described in Step 11, are recorded. The results are averaged, multiplied by the appropriate correction factor, and reported to the nearest 0.1 mm.
 - The instrument will warn the operator if the values diverge over a range greater than 1.0 mm. The test should be repeated with a fresh sample and extracted wick.
 - The instrument will warn the operator if an acceptable reference blend isn't stored in the calibration database used to calculate the result. The sample should be retested once the instrument is calibrated at the correct barometric pressure.

13. Once the three measurements are complete, the elevator lowers the candle, automatically extinguishes the flame, and stops at rest position.
14. Remove the candle from the elevator and rinse with toluene and acetone. Purge the candle with air to dry and prepare for re-use.

Calculations

All calculations are performed by the instrument. For additional information, refer to ASTM Method D1322.

Precision

Precision statements were determined using UOP Method 999, "Precision Statements in UOP Methods."

Repeatability and Site Precision

A nested design was carried out for determining smoke point on three samples. The samples were analyzed by two analysts, with each analyst performing two analyses on two separate days for a total of 24 analyses. Using a stepwise analysis of variance procedure, the within-day and within-lab estimated standard deviations (esd) were calculated at the concentration means listed in Table 2. Two analyses performed in one laboratory by the same analyst on the same day should not differ by more than the repeatability allowable differences shown in Table 2 with 95% confidence. Two analyses performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable differences shown in Table 2 with 95% confidence.

Table 2
Repeatability and Site Precision, Smoke Point, mm

<u>Sample</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>	
		<u>Within-Day esd</u>	<u>Allowable Difference</u>	<u>Within-Lab esd</u>	<u>Allowable Difference</u>
A	19.0	0.15	0.5	0.12	0.6
B	23.9	0.22	0.8	0.22	0.9
C	41.6	0.25	1.0	0.36	1.4

Separately, 15 samples with smoke points in the range of 23 to 41 mm were analyzed in duplicate. The estimated standard deviations (esd) and allowable differences were calculated using UOP Method 666, "Precision Statements in UOP Methods." Two analyses performed in one laboratory should not differ by more than the allowable difference shown in Table 3 with 95% confidence.

Table 3
Repeatability from Pairs of Analyses, Smoke Point, mm

<u>Range</u>	<u>esd</u>	<u>Allowable Difference</u>
23 - 41	0.20	0.6

The data in Tables 2 and 3 represent short-term estimates of the repeatability of the method. When the test is run routinely, use of a control standard and a control chart is recommended to generate an estimate of long-term repeatability.

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

Time for Analysis

The elapsed time and labor requirements for one analysis, not including calibration, are identical, 0.3 hour.

Suggested Suppliers

AD systems, 20 rue François Arago, 14123 Ifs, France +33(0)2 50 28 14 20 www.adsystems-sa.com

Burdick & Jackson, 1953 S. Harvey St., Muskegan, MI 49442, USA (973-455-5268)
www.bandj.com

Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219-4785, USA (412-490-8300)
www.fishersci.com

VWR International, 1310 Goshen Parkway, West Chester, PA 19380, USA (610-431-1700)
www.vwr.com