

METHOD AND DEVICE FOR DETERMINING THE SMOKE POINT OF HYDROCARBONS

The present invention relates to an improved method for
5 determining the smoke point of hydrocarbons, comprising
in particular the acquisition and analysis of digital
images, and a device for implementing such a method.

The smoke point of a hydrocarbon is a characteristic
10 that is routinely determined in the laboratories of
refineries, mainly on kerosenes, aviation fuels and
even lamp oils.

This characteristic is an important parameter since it
15 is directly linked to the hydrocarbon composition of
the fuels under test. In practice, the greater the C/H
ratio, and therefore the lower the aromatic compound
content, the higher the smoke point becomes and the
better the fuel behaves on its combustion. In other
20 words, the smoke point is quantitatively linked to the
potential transfer of radiative heat and, in as much as
this heat transfer exerts a strong influence on the
temperature of the metallic parts, the smoke point
therefore becomes a predictive indicator of the
25 longevity of said metallic parts.

The smoke point does, unfortunately, however have the
drawback of being fairly difficult to measure.
Normally, for this, an analysis method is used that is
30 the subject of a standardization (such as the method
described in the ASTM D 1322 standard and its
equivalents, such as ISO 3014, IP 57 and NF M 07-028)
to enable the detection, then the measurement of the
maximum height of a flame (normally expressed in mm and
35 accurate to the nearest tenth of an mm) which can be
obtained from the hydrocarbon under test without the
formation of smoke.

In such a measurement, the sample is burned in a wick

lamp, also described in the ASTM D 1322 standard, and the operator varies the position of the burner so as to gradually modify the height and the appearance of the flame, which changes slowly from a relatively elongated and jumpy state with a top end giving off a light smoke, to a state in which the flame height is shorter, with a top end that is perfectly rounded. Between these two flame states, the operator must also distinguish two other intermediate shapes, namely that having an elongated point, the edges of which appear concave in the top part and the one in which the pointed end has just disappeared and which forms a flame that is slightly rounded without smoke. It is when the flame has this last appearance that the operator records the height of the flame, on a scale graduated in mm positioned inside and at the back of the lamp. The final value of the smoke point retained for the sample under analysis is the average of three successive measurements, calculated to the nearest 0.1 mm.

The method of measuring the smoke point, as defined in the ASTM D 1322 standard, like all the analysis methods of this type, does, however, have limitations in terms of accuracy, mainly due to the assessment difficulties of the operator, in particular when taking the decision to judge the correct appearance of the flame, according to the standard, and also at the moment when the height of this flame is measured visually on the graduated scale. In practice, the good quality of the measurement of this height requires particular procedural precautions, the application of which depends entirely on the operator. Thus, the repeatability and reproducibility of the standardized test are respectively 2 mm and 3 mm.

The Applicant has proposed to remedy this difficulty by replacing the eye and the brain of the operator with a technical system of acquiring digital images mainly comprising a digital camera, or an equivalent of such a

digital camera, and an associated computer system for analyzing and processing the stored digital images. However, the distinguishing of the different characteristic shapes of the flame, between the
5 relatively elongated and jumpy state with a top end giving off a light smoke and the state presenting a shorter flame height with a top end that is perfectly rounded, is subjective and is ill-suited to the normal use of a computer program for analyzing and processing
10 digital images. The Applicant, after much research work, has found that, surprisingly, the use of such a system associated with a choice of appropriate parameters and a correct calibration, would make it possible to spectacularly increase the accuracy of the
15 method and obtain the smoke point of the hydrocarbon under test but without the problem of the subjectivity of the operator.

The subject of the invention is, consequently, a method
20 for determining the smoke point of a hydrocarbon, comprising among the different steps defined in the ASTM D 1322 standard or its equivalents, the identification, among different appearances of the flame according to the position of the burner in the
25 lamp, of a particular appearance of the flame and the reading of the height of this flame on a scale graduated in mm, characterized in that a series of digital images of the flame is taken and stored using a digital camera or an equivalent of such a camera, at
30 intervals that are sufficiently close to enable, by analyzing these digital images, the detection of a sudden change in the shape of the flame, and that the height of said flame is measured at the moment of this sudden change in its shape, said height being
35 considered as the smoke point of the hydrocarbon under test.

Preferably, the digital camera will be equipped with a photodetecting charge-coupled device (CCD). The image-

taking intervals will preferably be between 0.1 and 2.0 seconds, and in particular between 0.5 and 1.0 second. The number of digital images of each series of images is preferably at least equal to 10.

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The digital image-taking operations and the analysis and processing of these images to identify the image corresponding to the first sudden change in the shape of the flame are preferably automated using a dedicated software package.

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The various steps of the known method of measuring the smoke point discussed above and referred to in the preamble to the independent method claim, carried out in the procedural conditions defined in the ASTM D 1322 standard, or its equivalents, are as follows:

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- (a) preparing and calibrating a lamp for determining the smoke point as indicated in the standard, points 9 and 10,
- 20 (b) fitting a wick measuring at least 125 mm, soaked with sample, in the wick-holder of the lamp's burner,
- (c) introducing 20 ml of sample into the burner's tank,
- (d) screwing the wick-holder with the wick onto the burner's tank,
- 25 (e) cutting the top end of the wick so that it extends above the wick-holder by 6 mm and inserting the burner into the lamp,
- (f) lighting the wick and adjusting the wick until the flame has a height of approximately 1 cm and, after a combustion period of approximately 5 minutes,
- 30 (g) raising the burner until smoke appears, then,
- (h) slowly lowering the burner until the flame no longer gives off smoke and presents a top end that is perfectly rounded.
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The steps preliminary to identifying the sudden change in the shape of the flame are strictly in accordance with the ASTM D 1322 standard (in particular the

ASTM D 1322-97 standard), or one of its technical equivalents, so there is no need to describe in detail here all the procedural conditions and safety measures making it possible to correctly carry out the method of
5 determining the smoke point.

The step consisting in manually gradually lowering the wick of the lamp's burner to obtain a flame no longer giving off smoke and presenting a top end that is
10 slightly rounded, of the method of the present invention, differs, however from the corresponding step (h) of the method of the ASTM D 1322 standard by the fact that a series of digital images of the flame is acquired and stored, and the stored digital images are
15 analyzed using an appropriate image analysis system, so as to be able to deduce from it the maximum height of the flame that does not give off smoke, in other words the smoke point of the fuel under test.

20 To understand the invention better, the Applicant first describes the visual assessment of the maximum height of the flame that does not give off smoke as provided by the ASTM D 1322 standard, by here providing a partial transcription of point 11.4 of this standard:

25 11.4 Light the burner and adjust the wick so that the flame has a height of approximately 10 mm and leave the lamp burning for approximately 5 minutes. Raise the burner until smoke appears, then lower the burner
30 slowly passing through the following flame appearance steps:

11.4.1 a long point, smoke weakly visible, flame erratic and jumpy,
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11.4.2 an elongated point with lateral edges that are concave towards the top (flame A),

11.4.3 pointed end just disappears, leaving a flame

that is very slightly "blunted" (flame B),

11.4.4 a well rounded point (flame C). Determine the height of the flame B to the nearest 0.5 mm. Make a
5 note of the observed height.

11.4.4.1 To eliminate parallax errors, the eye of the observer must be slightly to one side of the central line so that the reflected image of the flame is
10 visible on the graduated scale to one side of the central vertical white line and the flame itself is visible against the other side of the graduated scale. The results of the two readings should be identical.

15 In the method of the present invention, the technical system for storing and analyzing images used to replace the eye of the observer serves both to determine the moment when the visible flame corresponds to the "flame B" of the standard, and to measure as accurately as
20 possible the height of this flame B.

Analysis of the digital images stored while the burner is being lowered that is accompanied by the reduction in the size of the flame, has shown that the sudden
25 change in the shape of the flame, that is, the appearance of the "flame B", corresponded to a sudden change, that is easy to detect, in the speed of reduction of the Feret diameter of the image of the flame. The Feret diameter of an object is equal to the
30 distance between two tangents to the object, parallel to each other, and defining an angle α relative to the horizontal, this angle being sometimes called "direction of measurement of the Feret diameter". When the object is not a circle, this distance depends on
35 the angle α defined by the tangents relative to the horizontal.

It will easily be understood that, in the case of the flame whose dimensions are to be measured, the distance

between two tangents to the flame, parallel to each other and to the horizontal ($\alpha = 0^\circ$) is equal to the height of the flame. This is why, in the description below, the height of the flame is sometimes called
5 Feret diameter with angle $\alpha = 0^\circ$.

The analysis of the images as such, whether analyzing the shape of the flame or its height, requires an additional operation, called "thresholding" or
10 "binarization", conventionally used in analyzing and processing digital images. This thresholding operation consists in setting to zero all the pixels having a gray level less than a certain value, called "threshold", and to 1 all the pixels having a value
15 above the threshold.

In practice, a flame is a physical phenomenon characterized by a local increase in heat and light intensity. Although the human eye believes it can
20 distinguish a fairly clear and precise flame outline, the digital image in terms of gray levels of a flame shows that the increase in light intensity is continuous and that there is a gradual change to increasingly higher brightnesses. This brightness
25 continuity appears clearly in figure 2 which represents the value of the gray levels of a line of pixels passing mid-height through the flame, perpendicular to the vertical axis of symmetry of said flame.

30 The sudden change in the speed of reduction of the Feret diameter, corresponding to the moment at which the "flame B" appears, is preferably determined by different measurements of the Feret diameter at an angle α that is other than zero. This angle α
35 nevertheless has a relatively low value, preferably less than 45° . Figure 1 shows, for four different angles α (0° , 18° , 36° and 90°), the trend of the Feret diameter (in 1/10 mm) according to the lowering of the position of the burner during the step (h) of the

method. On the three curves respectively corresponding to an angle $\alpha = 0^\circ$, 18° and 36° , a break point is observed, indicated by an arrow, where the slope of the curve (reflecting the speed of reduction of the height of the flame) changes suddenly. This break point or "fold" corresponds to the moment when the flame is a flame B in the sense of the ASTM D 1322 standard, for a precise position of the burner. It will be noted that, for an angle $\alpha = 90^\circ$ (Ferret diameter corresponding to the width of the flame), this characteristic break point is not observed.

After determining the position of the burner corresponding to the flame of maximum height not giving off smoke (flame B), secondly, the absolute height of this flame is determined which, after the appropriate corrections, gives the smoke point.

The absolute value of the height of the flame B is determined by comparing it with a graduated scale which is part of the lamp described in the ASTM D 1322 standard. In the known method, the operator directly reads the height of the flame B on the graduated scale, placed behind said flame, taking the precautions indicated in point 11.4.4.1. In the inventive method, the graduated scale is not visible on the digital image of the flame stored by the CCD digital camera. The height of the flame must consequently be determined by superimposing the digital image of the flame on the digital image of the graduated scale (calibration image), taken independently by the CCD apparatus from an identical position, but in the absence of flame, and with appropriate lighting.

As for the detection of the shape of the flame corresponding to the flame B, in the sense of the ASTM D 1322 standard, determining the height of the flame also entails a digital image thresholding operation.

In practice, to determine the height of a flame, it is necessary first to decide the light intensity from which the flame begins. This decision amounts to
5 setting a threshold (gray level value) above which it is considered that there is a flame, and below which it is considered that there is no flame. In figure 2, the chosen threshold corresponds to the dashed horizontal line. The arrows A and B mark the limits of the flame
10 for this threshold.

The difficulty lies in choosing the threshold to be used. In practice, the higher the chosen threshold is, the lower the absolute value of the height of the flame
15 that will be obtained becomes. The appropriate threshold, that is, the threshold that gives the absolute height of the flame leading to the correct smoke point of the fuel under test can be determined using one or more standard fuel mixtures
20 (toluene/2,2,4-trimethylpentane), for which the ASTM D 1322 standard indicates the smoke point. For this, a correctly installed apparatus is used to produce a series of digital images of a combustion flame of a standard fuel mixture, and, after an
25 appropriate thresholding operation as indicated above, the digital image corresponding to the sudden change in the speed of reduction of the Feret diameter is selected, then this image is subjected to a series of thresholding operations with different thresholds and
30 the threshold that gives a flame height equal to the smoke point indicated by the ASTM D 1322 standard for the standard fuel mixture used is retained. The duly determined threshold must be retained throughout the series of measurements.

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To complement the automation of the measurement of the smoke point of a hydrocarbon, according to the present invention, an ancillary continuous servo-control device between the position of the burner in the lamp and the

triggering of image-taking by the digital camera can be installed, the burner being in this case positioned at different levels in the lamp by means of an electric motor.

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Another subject of the present invention is a device for determining the smoke point of a hydrocarbon fuel, comprising:

- 10 (A) an apparatus for determining the smoke point conforming to the specifications of the ASTM D 1322 standard,
- (B) a digital camera, preferably a CCD digital camera, and, linked to said camera,
- 15 (C) a computer system designed and programmed to enable digital images taken by the digital camera to be stored, analyzed and processed.

This device also preferably comprises an anti-infrared filter placed between the apparatus for determining the smoke point (A) and the digital camera (B), and which serves to intercept the infrared radiation emitted by the flame that would saturate the images and render them unusable.

25 The CCD device digital camera preferably covers wavelengths ranging from the ultraviolet to the infrared, but models covering only the visible spectrum or the visible-UV spectrum can, however, also be used. The use of an anti-infrared filter then becomes
30 superfluous.

The dynamic range of the digital camera used to acquire the digital images is not a determining factor. To obtain images offering a satisfactory resolution, at
35 least 10-bit CCD digital cameras should however be used, making it possible to store digital images with at least 512 gray levels. 16-bit or higher CCD digital cameras giving images with 32 768 gray levels, or more, are particularly preferred.

The CCD digital camera normally comprises a zoom and is placed at a distance of approximately 1 m to 1.5 m from the lamp of the apparatus for determining the smoke point. The zoom is then set so that the stored digital image contains the image of all the graduated scale of the apparatus for determining the smoke point. It is essential for the relative position of the CCD digital camera, relative to the lamp, not to vary between two images. The invariability of this position is, in effect, the essential condition that makes it possible to compare the different stored images and the smoke point values calculated from the latter. If, however, the position of the digital camera or the zoom setting were altered inadvertently, a new calibration image (image of the graduated scale in the absence of flame) can and must be acquired.

Given the very high sensitivity of the sensors of CCD digital cameras, a certain number of measures must be taken to ensure a good image quality. The different protocols for calibrating and eliminating background noise are familiar to those skilled in the art using CCD sensors and do not need to be described in detail here. As an example, measures ensuring a good image quality, cooling of the CCD sensors by a Peltier module, in order to eliminate the formation of so-called "thermal" charges, can be cited.

The present invention is illustrated by the following exemplary applications.

Example 1

Validation of the method according to the invention by determination of the smoke point of a fuel mixture with known smoke point - determination of an appropriate threshold

After having installed and calibrated the wick lamp and the device according to the present invention, two successive series, each of ten digital images, are taken of a combustion flame of a mixture of 15% by volume of toluene and 85% by volume of 2,2,4-trimethylpentane (reference smoke point: 25.8 mm), the height of which is gradually reduced in accordance with the protocol described in point 11.4 of the ASTM D 1322 standard. Based on the digital images obtained, previously subjected to a thresholding of a value equal to approximately 15% of the measurement range of the CCD sensor, i.e., for this test, a value of 2000, a curve of the value of the Feret diameter of the flame is plotted, for a measurement direction α of this same Feret diameter equal to 36° , according to the position of the burner, or more precisely, according to the image number. A sudden change in the speed of reduction of the height of the flame is observed for the image number 4. Various thresholding operations are then applied to this image and the threshold that gives a Feret diameter at $\alpha = 0^\circ$ that is the closest to the theoretical value of the standard (25.8 mm) is retained. The duly determined threshold of 4000 gives a Feret diameter ($\alpha = 0^\circ$) of 26.2 mm for the first series of images and 25.9 mm for the second series of images.

The software for analyzing and processing the two series of ten digital images, used in the present test, is supplied by Neosis, marketed by the name Visilog 6. The threshold values retained and the values of the angle α for the calculation of the corresponding Feret diameters were determined previously using different reference products according to the present invention in the procedural conditions conforming to the ASTM D 1322 standard.

Example 2

Repeatability of the flame height measurement

With a device according to the invention comprising:

- (a) an apparatus for measuring the smoke point according to the ASTM D 1322 standard,
- 5 (b) a CCD digital camera (16 bits) provided with a zoom,
- (c) linked to the digital camera, a computer with the Visilog 6 software published by Neosis installed, capable of storing, analyzing then processing the
- 10 digital images originating from the digital camera, and
- (d) an anti-infrared filter placed between (a) and (b), three successive series, each of four digital images, are acquired, respectively corresponding to four
- 15 different heights of a combustion flame of a hydrocarbon to be analyzed. The duly obtained digital images are subjected to a thresholding operation with a threshold equal to 4000, determined in the example 1.

20 The following flame height values are obtained (expressed in mm):

Series No.	Flame 1	Flame 2	Flame 3	Flame 4
1	15.9	11.4	9.4	5.6
2	15.9	11.2	9.4	5.6
3	16.1	11.2	9.2	5.7
Average	15.9	11.3	9.3	5.6
Standard deviation	0.1	0.1	0.1	0.1
Calculated repeatability	0.3	0.3	0.3	0.3
ASTM D 1322 repeatability	2	2	2	2

It can be seen that the standard deviations are of the order of 0.1 mm.

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The repeatabilities obtained with the method and the device according to the present invention are of the

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order of 6 to 7 times better than those stated in the ASTM D 1322 standard.

CLAIMS

1. A method for determining the smoke point of a hydrocarbon, comprising among the different steps
5 defined in the ASTM D 1322 standard or its equivalents, the identification, among different appearances of the flame according to the position of the burner in the lamp, of a particular appearance of the flame and the reading of the
10 height of this flame on a scale graduated in mm, characterized in that a series of digital images of the flame is taken and stored using a digital camera or an equivalent of such a camera, at intervals that are sufficiently close to enable,
15 by analyzing these digital images, the detection of a sudden change in the shape of the flame, and that the height of said flame is measured at the moment of this sudden change in its shape, said height being considered as the smoke point of the
20 hydrocarbon under test.
2. The method as claimed in claim 1, characterized in that the digital camera comprises a photodetecting charge-coupled device (CCD).
- 25 3. The method as claimed in claims 1 and 2, characterized in that the image-taking intervals are between 0.1 and 2.0 seconds, and preferably between 0.5 and 1.0 second.
- 30 4. The method as claimed in any one of the preceding claims, characterized in that the detection of the sudden change in the shape of the flame is achieved by measuring the sudden change in the
35 speed of reduction of the Feret diameter of the image of the flame.
5. The method as claimed in claim 4, characterized in that, to detect the sudden change in the speed of

reduction of the Feret diameter, this Feret diameter is measured at an angle α less than 45° .

- 5 6. The method as claimed in one of claims 4 and 5, characterized in that the height of the flame is equal to the Feret diameter for $\alpha = 0^\circ$ of the image of the flame.
- 10 7. The method as claimed in one of claims 4 to 6, characterized in that the digital image corresponding to the sudden change in the speed of reduction of the Feret diameter is subjected to a thresholding operation using a determined threshold with the help of one or more standard
15 fuel mixtures (toluene/2,2,4-trimethylpentane) with known smoke point (ASTM D 1322).
- 20 8. The method as claimed in one of the preceding claims, characterized in that the charge-coupled device (CCD) digital camera covers wavelengths ranging from the ultraviolet to the infrared.
- 25 9. The method as claimed in one of the preceding claims, characterized in that an anti-infrared filter is placed between the flame and the CCD digital camera.
- 30 10. The method as claimed in one of the preceding claims, characterized in that the digital camera with CCD can be used to store digital images with at least 512 gray levels (10 bits) and preferably at least 32 768 gray levels (16 bits).
- 35 11. The method as claimed in any one of the preceding claims, characterized in that a CCD digital camera with zoom is used and that this camera is placed at a distance of approximately 1 m to 1.5 m from the lamp.

12. The method as claimed in claim 11, characterized
in that the zoom is set so that the stored digital
image contains the image of all the graduated
scale of the device for determining the smoke
point.
13. The method as claimed in any one of the preceding
claims, characterized in that the number of
digital images of each series is at least equal to
10.
14. The method as claimed in the preceding claims,
characterized in that the digital image-taking
operations and the analysis and processing of
these images are automated using a dedicated
software package.
15. A device for determining the smoke point of a
hydrocarbon fuel, comprising:
- an apparatus for determining the smoke point
conforming to the specifications of the
ASTM D 1322 standard,
 - a digital camera and, linked to said camera,
 - a computer system designed and programmed to
enable digital images taken by the digital
camera to be stored, analyzed and processed.
16. The device as claimed in claim 15, characterized
in that it also comprises an anti-infrared filter
placed between the apparatus for determining the
smoke point and the digital camera.
17. The device as claimed in claim 15 or 16,
characterized in that the digital camera covers
wavelengths ranging from the ultraviolet to the
infrared.
18. The device as claimed in one of claims 15 to 17,
characterized in that the digital camera can be

used to store digital images with at least 512 gray levels (10 bits), preferably at least 32 768 gray levels (16 bits).

- 5 19. The device as claimed in one of claims 15 to 18,
characterized in that the digital camera comprises
a zoom and is placed at a distance of
approximately 1 m to 1.5 m from the lamp of the
apparatus for determining the smoke point.
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20. The device as claimed in claim 19, characterized
in that the zoom is set so that the stored digital
image contains the image of all the graduated
scale of the apparatus for determining the smoke
point.
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