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(54) **METHOD AND SYSTEM FOR ESTIMATING PROPERTIES OF A VACUUM RESIDUE AND HOMOLOGATING A CRUDE OIL**

(57) The invention relates to a method and a system for estimating properties of a vacuum residue issued from crude oils that can be used for homologating this crude oil, in particular for paving bitumen application. In particular, the method of the invention allows estimation of properties useful for characterization of paving bitumen from properties which are usually not used for characterizing paving bitumen.

The method and the system of the invention can be integrated respectively in a method and a system for homologating a crude oil, in particular for paving bitumen application.

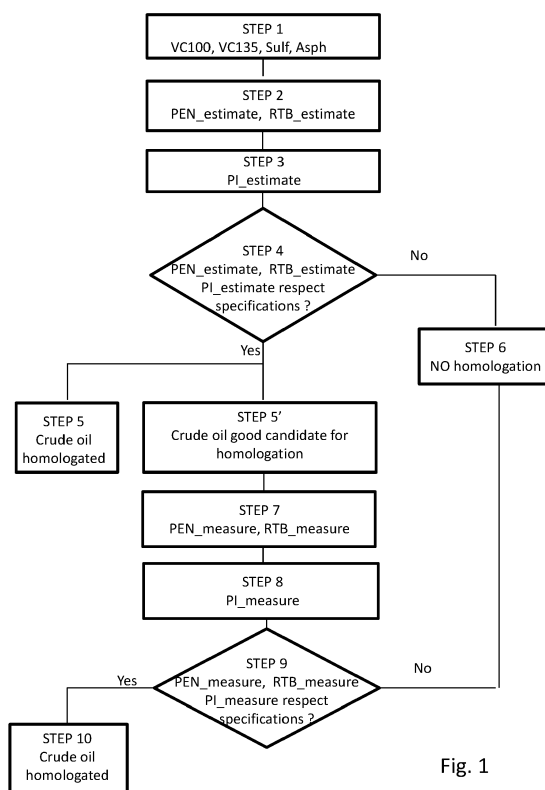


Fig. 1

Description**Technical field**

5 **[0001]** The present invention relates to bitumen and more specifically to homologation methods used to homologate crude oils for producing bitumen, in particular bitumen able to be used in the field of road construction or civil engineering (paving grade bitumen).

Background

10 **[0002]** Bitumen is the main hydrocarbon constituent used in the field of road construction or civil engineering. Bitumen can be defined as being a mixture of several "bitumen bases". Two or more bitumen bases can be mixed to form a composition formed of bitumen bases. A composition formed of bitumen bases can form bitumen. Two or several compositions formed of bitumen bases can also be mixed in order to obtain bitumen.

15 **[0003]** A bitumen base is regarded as being the product resulting from a refining process. Normally, a bitumen base can be produced by refining a crude oil, in particular a crude oil homologated to produce bitumen. This crude oil is heated to 300°C, partially vaporized in an oven and transferred into an atmospheric distillation column in which the separation of the different fractions is carried out. The lightest fraction vaporizes while the heaviest fraction (atmospheric residue) remains at the column bottom and passes into a second heat exchanger system before treatment in a vacuum distillation column. Finally, the bitumen base is recovered at the bottom of this vacuum distillation column. The bitumen base corresponds, for example, to the 560°C+ cut of the vacuum distillation.

20 **[0004]** In order to produce "bitumen bases", crude oils homologated to produce bitumen are normally selected as a function of their ability to produce the said bases. Thus, among all the crude oils referenced, only less than 10% make it possible to produce "bitumen bases". The said bases are generally obtained from residues resulting from vacuum distillation of crude oil. The main criteria for choosing the crude oils homologated to produce bitumen are:

- the technical characteristics of the bitumen bases resulting from these crude oils: penetrability, viscosity, softening point, and the like,
- 30 • adequacy with refinery plants, in particular the yields with respect to the cut temperatures of the vacuum distillation units.

[0005] The penetrability, more specifically the needle penetrability, is usually measured according to Standard NF EN 1426 (January 2018). The needle penetrability is the depth, expressed in tenths of a millimetre, to which a standardized needle with a diameter of 1 mm, under a load of 100 g, applied for 5s to a bitumen sample maintained at 25°C or at 15°C, drives into the sample.

35 **[0006]** The softening point, more specifically the ring and ball (RBT) softening point, usually measured according to Standard NF EN 1427 (January 2018), is a second fundamental characteristic of a bitumen : a small steel ball weighing 3.5 g and with a diameter of 9.5 mm is placed on a bitumen disk cast beforehand in a ring with an internal diameter of 19.8 mm, itself placed on a standardized support. The combined assembly is introduced in a water bath, the initial and stabilized temperature of which is 5°C. The lower face of the bitumen ring occurs at 25.4 mm from the upper surface of the plate of the bottom of the support, which corresponds to the distance which the ball falls during the test. The bath is heated at a constant rate of 5°C/min, with stirring, and the ring and ball softening point (often denoted RBT) is the temperature at which the bitumen pocket, formed during the fall of the ball, touches the reference plate placed (as has been said) at 25.4 mm under the bitumen ring. In this test, the higher the softening point, the harder the bitumen.

45 **[0007]** The penetration index (PI), according to Standard NF EN 12591 (december 2009), makes it possible to determine the thermal susceptibility of a bitumen. The PI is calculated by means of a formula from the value of the penetrability at 25°C and from the RBT softening point value of a given bitumen. The result is expressed without dimensions.

[0008] Homologation of crude oils usually requires a long time, about 3 to 7 months, as the crude oils has to be submitted to extensive laboratory tests. In general, the crude oils are submitted to an atmospheric distillation and a vacuum distillation to recover a vacuum residue as disclosed above, which is then further characterised to check its suitability for use as bitumen base. This characterisation can be performed according to standard NF EN12591 (December 2009), with the measure of the softening point, the penetrability at 25°C, the viscosity, etc. With such homologation proceeding there is no risk for the refineries to buy a crude oil, the vacuum residue of which would finally not be suitable as bitumen base. However, such a long time of homologation may result in a loss of bitumen valorisation for some crude oils that have not been bought as not homologated but which are suitable for bitumen use.

55 **[0009]** There is therefore a need for a rapid homologation method of crude oils that allows the refiners to take rapidly decisions on whether a crude oil can be bought or not to produce bitumen.

Summary

[0010] The present invention relates to a method and a system for estimating properties of a vacuum residue issued from crude oils that can be used for homologating this crude oil, in particular for paving bitumen application. In particular, the method of the invention allows estimation of properties useful for characterization of paving bitumen from properties which are usually not used for characterizing paving bitumen.

[0011] The method and the system of the invention can be integrated respectively in a method and a system for homologating a crude oil, in particular for paving bitumen application.

Definitions

[0012] A vacuum residue of a crude oil is defined as the residue of vacuum distillation of an atmospheric residue resulting from the atmospheric distillation of a crude oil. A residue is defined as the bottom of the distillation unit resulting from the distillation of a product. A vacuum residue usually corresponds to the 560+ cut, but may correspond to 520+ cut or 570+ cut or have still another cut point value depending on unit performance. The man skilled in the art is able to determine a cut point appropriate for a vacuum residue, in particular as a function of the performance of the unit considered.

[0013] "Crude oil" (or "crude") means oil from a natural reservoir that is extracted in liquid form at atmospheric pressure. It is therefore a natural product before refining, but which has already lost some of its initial composition, as the fraction of light hydrocarbons leaves the liquid phase at the point of extraction.

[0014] Asphaltenes are defined as the heavy products contained in crude oil which are insoluble in n-heptane and soluble in toluene. In general, asphaltenes contain carbon, hydrogen, nitrogen, oxygen, sulfur, vanadium and nickel.

Detailed description

[0015] According to a first aspect, a method for estimating properties of a vacuum residue issued from a crude oil is proposed. This method comprises :

(a) receiving a set of property data values of a vacuum residue of a crude oil, said set of property data comprising the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content, and optionally the asphaltene content,

(b) calculating an estimate of a penetrability value and an estimate of a softening point value of said vacuum residue using an estimating model arranged to calculate an estimate of the penetrability property data value and an estimate of the softening point property data value of a vacuum residue from the set of property data values received in step (a), said estimating model including :

a first correlation correlating a penetrability property data value to said set of property data values,

a second correlation correlating a softening point property data value to said set of property data values.

[0016] By allowing to estimate the penetrability value and the softening point value of a vacuum residue from other property values which are usually easily available, the method according to the invention allows to characterize properties of the vacuum residue which are used to determine if such vacuum residue is usable for a particular application, in particular as paving bitumen, for example as defined in NF EN12591 (dec 2009).

[0017] In particular, in the present invention, the penetrability value estimated is an estimate of the penetrability value at 25°C, expressed in 1/10mm, as defined in NF EN 1426 (January 2018) and the estimated softening point value is an estimate of the RBT softening point, expressed in °C, as defined in NF EN 1427 (January 2018).

[0018] The set of property data values received in step (a) can be measured values, estimated values or both, preferably measured values. When they are measured, the kinematic viscosity values (at 100°C and 135°C) may be measured according to NF EN 12595 (December 2014). The kinematic viscosity values are expressed in mm²/s. The sulphur content value may be measured according to ASTM D2622-16. The asphaltene content value may be measured according to NF T60-115 (January 2020). The sulphur and asphaltene contents are expressed in weight %.

[0019] The first and second correlations may correlate the estimated value with the same set of properties or with different set of properties.

[0020] Preferably, these correlations may be linear relations, in particular of the form :

$$y = a_1x_1 + a_2x_2 + \dots + a_jx_j + a_{p-1}x_{p-1} + a_0,$$

where $p-1$ is the number of property data the values of which are received in step (a), x_j is the value of the j^{th} property data, y is the value of the estimated property data and a_j are regression coefficients.

[0021] The invention is not limited to such correlations and any other appropriate correlation may be used.

[0022] Advantageously, step (b) may comprise a step of constructing the estimating model, comprising :

(i) receiving a first set of property data values for a number N of vacuum residues, said first set of data property values comprising values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and optionally the asphaltene content for each of the N vacuum residues,

(ii) receiving a second set of property data values for the same N vacuum residues, said second set of data property values comprising penetrability values and softening point values,

(iii) generating using statistical methods :

the first correlation correlating the penetrability value of said second said of property data values to said first set of property data values for each of the N vacuum residues,

the second correlation correlating the softening point value of said second said of property data values to said first set of property data values for each of the N vacuum residues.

[0023] The number N of vacuum residues used in steps (i) and (ii) will be chosen so as to be superior to the number of property data in the correlation and preferably so as to be statistically representative of the existing vacuum residues for which an estimate is needed. By way of example, N may be equal to 10 or more, preferably equal to 50 or more, more preferably of more than 90 or 100. The higher the number N of vacuum residues, the better the model obtained.

By "statistically representative", we mean a number N sufficient for the property data values to be representative.

[0024] The first and second sets of property data values received in steps (i) and (ii) can be measured values, estimated values or both, preferably measured values.

[0025] In particular, in steps (i) and (ii), each of the vacuum residues may be issued from a single crude oil. Several crude oils may however be used to obtain each of the vacuum residues.

[0026] In step (a) and/or in steps (i), (ii), the vacuum residues may present any cut point value (i.e. any initial boiling point) allowing obtaining a vacuum residue.

[0027] The statistical methods used for generating the correlation include multivariate statistical analysis techniques such as Partial Least Squares, Multiple Linear Regression, Reduced Rank Regression, Principal Component Analysis and the like, or neural networks, preferably multiple linear regressions, in particular a forward stepwise regression. These statistical analysis techniques or neural networks are well known to those skilled in the art and will therefore not be described in detail. In particular, the property data of the first set of data property values may preferably be uncorrelated property data. Uncorrelated property data may be identified by calculating correlation matrix or by using any other appropriate method such as Ridge regression, partial least squares, principal component regression.

[0028] The property data values used to construct the model may be submitted to a mathematical treatment allowing linearization of the problem to solve for the construction of the estimating model.

[0029] In a general way, the estimating model is constructed on the basis of a set of values which are between minima and maxima values. Advantageously, the vacuum residues for which an estimate of the penetrability and of the softening point is needed may preferably be chosen among vacuum residues which present values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content, and optionally of the asphaltene content, within the minima and maxima of the values used to construct the model. It should be noted that the set of values used to construct the estimating model and the values of the vacuum residue to estimate using the model should preferably be measured using the same standards. These standards may be the standards mentioned in the specification or other versions of these standards, or any other existing standards, provided the same standards are used for the values used for constructing the model and the values used for estimation purpose.

[0030] Advantageously, the step of constructing the model may include a step (iv) of validation in which for a number M of vacuum residues, which are different from the N vacuum residues used to generate the correlations, penetrability and softening point values are estimated by implementing the correlations obtained in step (iii) from the set of property data values of the M vacuum residues, and compared to measured values of penetrability and softening point for these M vacuum residues. This number M of residues may be of 5 or more, preferably of 10 or more.

[0031] Advantageously, in step (b), the first correlation of the estimating model may correlate the penetrability property data value to the values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C and the sulphur content.

[0032] Advantageously, in step (b), the second correlation of the estimating model may correlate the softening point property data value to the values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content

and the asphaltene content.

[0033] An estimation can then be obtained with a reduced number of properties, those properties being generally easily obtained.

[0034] According to another aspect, a method for homologating a crude oil is provided, which comprises :

(A) receiving an estimate of the penetrability value and an estimate of the softening point value of a vacuum residue issued from said crude oil, these estimated values being determined by implementing the estimating method according to the invention,

(B) optionally calculating an estimate of a penetration index value of said vacuum residue using the estimate of the penetrability value and the estimate of the softening point value received in step (A),

(C) comparing the estimated values of said vacuum residue received in step (A), and optionally calculated in step (B), with threshold values required for using a vacuum residue for a particular application, in particular for a paving bitumen application,

(D) deciding of the homologation of the crude oil from which is issued said vacuum residue intended for said particular application.

[0035] These threshold values of step (B) may consist of a lower limit, an upper limit, or both. These threshold values may for example correspond to the values required for classification of a bitumen or a bitumen base in one of the grades defined in the standard NF EN 12591 (December 2009), or to any other specification required by a national requirement, a standard or a use.

[0036] The decision step (D), may comprise, or consist of, a step (D1) comprising deciding a crude oil is homologated to produce a vacuum residue for said particular application when the estimated values comply with said threshold values, otherwise deciding the crude oil is not homologated.

[0037] It should be noted that the above homologating method is not limited to homologation for paving bitumen but could be used for homologating a crude oil for any other application for which the knowing of the penetrability value and of the softening point value is of importance.

[0038] Of course, the invention is not limited to a particular standard, and the threshold values may be chosen by the man skilled in the art according to other standards or national requirements, or to values required for particular applications.

[0039] Instead of deciding in step (D1) that the crude oil is homologated, step (D) may comprise further steps to take such decision.

[0040] The deciding step (D) may then comprise, or consist of:

a step (D'1) comprising :

- when the estimated values comply with said thresholds values :

measuring at least the penetrability at 25°C and softening point of the vacuum residue, and optionally calculating the penetration index from the measured values of penetrability at 25°C and softening point,

comparing the measured values of penetrability at 25°C and softening point, and optionally the calculated penetration index with said thresholds values,

deciding the crude oil is homologated if the measured values are within said threshold values, otherwise deciding the crude oil is not homologated.

- when the estimated values do not comply with the thresholds values, deciding the crude oil is not homologated.

[0041] The measures made when the estimated values are within the thresholds ranges can include measurements of further properties. For example, for a vacuum residue intended to be used as a paving grade, all the properties mentioned in the standard NF EN 12591 can be measured.

[0042] According to another aspect, a system for estimating properties of a vacuum residue issued from a crude oil is proposed. Advantageously, this estimating system is arranged to implement the steps of the method for estimating properties according to the invention.

[0043] This estimating system comprises :

(a) receiving means arranged for receiving a set of property data values of a vacuum residue of a crude oil, said

set of property data comprising the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and optionally the asphaltene content,

(b) treatment means arranged for calculating an estimate of a penetrability value and an estimate of a softening point value of said vacuum residue using an estimating model arranged to calculate an estimate of the penetrability property data value and an estimate of the softening point property data value of a vacuum residue from the set of property data values of said vacuum residue received in step (a), said estimating model including :

a first correlation correlating a penetrability property data value to said set of property data values,

a second correlation correlating a softening point property data value to said set of property data values.

[0044] The receiving means may be input or input/output interfaces. They can be wireless communication interfaces (Bluetooth, WIFI or other) or connectors (network port, USB port, serial port, Firewire® port, SCSI port or other).

[0045] The treatment means may be one or several processors, for example microprocessors or microcontrollers. The processor(s) may have storage means which may be random access memory (RAM), Electrically-Erasable Programmable Read-Only Memory (EEPROM), flash memory, external memory, or other. These storage devices can store, among other things, received data, estimated data and estimating model and computer program(s).

[0046] Advantageously, the estimating system may be arranged to perform a step of constructing the estimating model as disclosed above in respect of the estimating method of the invention. The estimating system may then comprise :

(i) receiving means for receiving a first set of property data values for a number N of vacuum residues, said first set of data property values comprising values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and optionally the asphaltene content for each of the N vacuum residues,

(ii) receiving means for receiving a second set of property data values for the same N vacuum residues, said second set of data property values comprising penetrability values and softening point values,

(iii) treatment means for generating using statistical methods :

the first correlation correlating the penetrability value of said second said of property data values to said first set of property data values for each of the N vacuum residues,

the second correlation correlating the softening point value of said second said of property data values to said first set of property data values for each of the N vacuum residues.

[0047] The treatment means generating the model may be the same or different than the treatment means calculating the estimates. The receiving means may be the same or different than the previously mentioned receiving means. These treatment means and receiving means may be as previously defined.

[0048] In particular, the estimating model may be as previously disclosed with respect to the estimating method and the treatment means arranged for generating first and second correlations as previously disclosed.

[0049] According to another aspect, a system for homologating a crude oil is proposed. Advantageously, this homologating system may be arranged to implement the homologation method according to the invention. This homologating system comprises :

- receiving means for receiving an estimate of the penetrability value and an estimate of the softening point value of a vacuum residue issued from said crude oil, these estimated values being received from the estimating system according to the invention,

- treatment means arranged to :

optionally calculate an estimate of the penetration index value of said vacuum residue using the estimate of the penetrability value and the estimate of the softening point value received by the receiving means,

compare the estimated values of said vacuum residue received by the receiving means, and optionally calculated, with threshold values required for using a vacuum residue for a particular application, in particular a paving bitumen application,

decide of the homologation of the crude oil from which is issued said vacuum residue intended for said particular application.

[0050] The receiving means and treatment means may be defined as the previously mentioned receiving means and treatment means.

[0051] In particular, the receiving means may be arranged to receive estimated data from said estimating system, for example by appropriate communication means such as wiring elements or wireless communication elements.

[0052] Advantageously, the treatment means are arranged to decide the homologation of the crude oil as explained above for the homologating method.

[0053] Advantageously, such homologating system may further include the estimating system of the invention and the receiving means may be arranged to receive estimated data from said estimating system. The receiving means and treatment means of the homologating system may include the receiving means and treatment means of the estimating system.

[0054] A computer program product is also provided comprising the instructions for carrying out the steps of the estimating method of the invention or of the homologating method of the invention, when said instructions are executed by one or more processors. The above methods may indeed be implemented in a treatment device such as a processor, for example a microprocessor, a microcontroller or else.

Description of the drawings

[0055] The invention will be better understood with reference to the figures, which show exemplary embodiments of the invention.

[0056] Figure 1 represents a flowchart of a method for homologating a crude oil including a method for estimating properties according to one embodiment.

[0057] In STEP 1, property data values of a vacuum residue issued from crude oil to homologate are provided, these property data values are : VC135 and VC100 as previously defined, Sulf for the wt% content of sulfur, Asph for the wt% content of asphaltenes.

[0058] In STEP 2, penetrability and softening point values, respectively noted PEN_estimate and RTB_estimate are estimated using an estimating model generated as previously explained.

[0059] STEP 1 and STEP 2 can be implemented by the estimating system of the invention.

[0060] In STEP 3, the penetrability index, noted PI_estimate, is calculated from PEN_estimate and RTB_estimate, as defined in EN 12591.

[0061] In STEP 4, PEN_estimate, RTB_estimate and PI_estimate are compared with threshold from specifications, here from specifications of EN12591.

[0062] If these values do not comply with the specifications, then the process goes to STEP 6 : the vacuum residue is considered as not homologated, more particularly not suitable for being homologated, the process is stopped.

[0063] If these values comply with the specifications, then the process goes to STEP 5.

[0064] STEP 5 : the vacuum residue and the crude oil from which it is issued is homologated to produce a bitumen, in particular a bitumen of a specific grade and the process is stopped. Alternatively, it can be considered in STEP 5', that the vacuum residue (and the crude oil from which it is issued) is a good candidate for such homologation and the process then goes to STEP 7.

[0065] STEP 7 : the crude oil is submitted to an atmospheric distillation and the atmospheric residue is submitted to a vacuum distillation to obtain a 560+cut. The 560+cut then undergoes at least penetrability and softening point measurements, and preferably a measure of all the other properties required by the specifications for homologating a vacuum residue. Measured values of penetrability (PEN_measure) and of the softening point (RTB_measure) are then obtained.

[0066] In STEP 8, the PI index is calculated from PEN_measure and RTB_measure, the value obtained is noted PI_measure.

[0067] In STEP 9, PEN_measure, RTB_measure and PI_measure are compared with threshold from specifications, here from specifications of EN12591.

[0068] If these values do not comply with the specifications, then the process goes to STEP 6 : the vacuum residue (and the crude oil from which it is issued) is considered as not homologated.

[0069] If these values comply with the specifications, then the process goes to STEP 10, where it is decided that the vacuum residue and the crude oil from which it is issued is homologated. It can then be decided that such vacuum residue can be incorporated, for example at less than 30wt%, to produce a bitumen of this specific grade.

[0070] Such decision of homologation can be further confirmed by further steps, with a more complete analysis of said crude oil. For example, with respect to EN12591, the crude oil can be distilled (by atmospheric and vacuum distillation) to isolate several vacuum residues covering the penetrability ranges of all of the grades of EN12591, the properties of each of the vacuum residues being then measured according to the specifications.

[0071] STEP 3 to STEP 10 can be implemented by the homologating system of the invention.

Examples

Example 1

[0072] Measured values VC135, VC100, Sulf, Asph, PEN (penetrability at 25°C) and RTB (softening point) have been collected from 69 vacuum residues issued from 50 crude oils.

[0073] The vacuum residues used for constructing the models have a penetrability at 25°C from 34 to 226 (1/10 mm). The values VC100, VC135 and Sulf of these vacuum residues are within the minima and maxima collected in table 1.

Table 1 : Minima and maxima of property data values of vacuum residues used for generating the model

	VC100	VC135	Sulf	Asph
	mm ² /s	mm ² /s	wt%	wt%
MIN	253	63.1	0.345	0.02
MAX	122494	5369	9.060	29.50

[0074] Two models have been generated from this set of values for each of the property to estimate :

Penetrability estimation (using : N = 58 vacuum residues)

- model A : $PEN = f_A(VC100, VC135, Sulf)$
- model B : $PEN = f_B(VC100, VC135, Sulf)$

Softening point estimation :

- model A : $RTB = f_A(VC100, VC135, Sulf, Asph)$
- model B : $RTB = f_B(VC100, VC135, Sulf)$

[0075] Where f_A , f_B , f_A , f_B are distinct functions correlating PEN and RTB to the properties.

[0076] The data of 11 vacuum residues have been collected to check the validity of each model. The values of this validation data are within the minima and maxima values of the data used for generating the models.

Table 2 : Minima and maxima of property data values of the 11 vacuum residues used for validating the models

	VC100	VC135	Sulf	Asph
	mm ² /s	mm ² /s	wt%	wt%
MIN	384	81.9	0.361	0.42
MAX	23486	1789	5.683	16.4

Table 3 : Data values used for generating models A and B of PEN from 58 vacuum residues

PEN (1/10 mm)	Measured values	Model A	Model B
Min	36	16	16
Max	214	194	184
Mean value	100	91	90
sum of the VA deviations in 1/10 mm		1452	1517
mean of the VA deviations in 1/10 mm		25	26
standard deviation of the deviations in 1/10 mm		24	24
MAD calibration		14	14

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(continued)

PEN (1/10 mm)	Measured values	Model A	Model B
RMSE		35	35
R ²		0.854	0.855
PEN prediction +/- 30%		79%	78%

Table 4 : Data values used for validating models A and B of PEN from 11 vacuum residues

PEN (1/10 mm)	Measured values	Model A	Model B
Min	46	35	34
Max	186	311	332
Mean value	97	115	117
sum of the VA deviations in 1/10 mm		462	512
mean of the VA deviations in 1/10 mm		42	47
standard deviation of the deviations in 1/10 mm		60	65
MAD validation		18	19
RMSE		71	78
PEN prediction +/- 30%		73%	73%

Table 5 : Data values used for generating models A and B of RTB from 58 vacuum residues

RTB (°C)	Measured values	Model A	Model B
Min	36.8	37.9	37.5
Max	59.2	62.3	61.4
Mean value	45.4	45.8	45.6
sum of the VA deviations in 1/10 mm		132.5	123.9
mean of the VA deviations in 1/10 mm		2.3	2.1
standard deviation of the deviations in 1/10 mm		2.3	2.1
MAD calibration		1.2	0.8
RMSE		3.2	3.0
repeatability		1.0	1.0
reproductibility		2.0	2.0
R ²		0.846	0.893
RTB prediction +/- 4°C		84%	88%

Table 6 : data values used for validating models A and B of RTB from 11 vacuum residues

PEN	Measured values	Model A	Model B
Min	38.2	37.1	38.5
Max	51.0	54.4	54.6
Mean value	46.6	44.6	44.9
sum of the VA deviations in 1/10 mm		40.7	36.1

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(continued)

PEN	Measured values	Model A	Model B
mean of the VA deviations in 1/10 mm		3.7	3.3
standard deviation of the deviations in 1/10 mm		3.7	3.1
MAD validation		1.0	1.1
RMSE		5.1	4.4
PEN prediction +/- 30%		73%	73%

Table 7 : penetrability index calculated from estimated values of models A and B on the 58 vacuum residues used for generating the models

penetrability index	Values calculated from measured values	Model A	Model B
Min	-1.9	-2.1	-2.1
Max	0.6	-0.2	-0.2
Mean value	-1.0	-1.3	-1.2
sum of the VA deviations in 1/10 mm		23.8	22.6
mean of the VA deviations in 1/10 mm		0.4	0.4
standard deviation of the deviations in 1/10 mm		0.4	0.4
MAD calibration		0.2	0.2
RMSE		0.6	0.6

Table 8 : penetrability index calculated from estimated values of models A and B on the 21 vacuum residues used for validating the models

penetrability index	Values calculated from measured values	Model A	Model B
Min	-1.5	-1.9	-1.5
Max	1.4	2.4	1.3
Mean value	-0.8	-0.8	-0.2
sum of the VA deviations in 1/10 mm		5.4	5.9
mean of the VA deviations in 1/10 mm		0.5	0.5
standard deviation of the deviations in 1/10 mm		0.5	0.6
MAD validation		0.2	0.1
RMSE		0.7	0.8

[0077] In the above tables 3 to 8, RMSE stands for Root mean square Error, MAD stands for Median of absolute values of deviations from data's median

[0078] The above tables show that both models can be used for estimating the penetrability values as well as the softening point values, and for calculating the penetrability index from these estimates.

Example 2

[0079] PEN and RTB values have been estimated for a 560+cut of a crude using the models A of example 1. The below table 9 gives the estimated values and measured values.

Table 9

	unit	Standard	Measured values	Measured values entered into the models	Estimated values	EN12591 (dec 2009) specification
VC100	mm ² /s	EN12595		6302		
VC135	mm ² /s	EN12595		770		370 or more
Asph	wt%			8,0		
Sulf	wt%	ASTM D6622		1,720		
PEN (at 25°C)	1/10mm	EN1426	33		38	35 to 50
RTB	°C	EN1427	54		54.3	50 to 58
PI (calculated)		EN1427	-1.1		-0.8	-1.5 to 0.7

[0080] These results show that estimated values are close to real measured values and allow to determine that the vacuum residue can be homologated as a paving grade of 35/50 according to specification EN12591.

Claims

1. Method for estimating properties of a vacuum residue issued from a crude oil comprising :

(a) receiving a set of property data values of a vacuum residue of a crude oil, said set of property data comprising the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content, and optionally the asphaltene content,

(b) calculating an estimate of a penetrability value and an estimate of a softening point value of said vacuum residue using an estimating model arranged to calculate an estimate of the penetrability property data value and an estimate of the softening point property data value of a vacuum residue from the set of property data values received in step (a), said estimating model including :

a first correlation correlating a penetrability property data value to said set of property data values,
a second correlation correlating a softening point property data value to said set of property data values.

2. Method according to claim 1, wherein step (b) comprises a step of constructing the estimating model, comprising :

(i) receiving a first set of property data values for a number N of vacuum residues, said first set of data property values comprising values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and optionally the asphaltene content for each of the N vacuum residues,

(ii) receiving a second set of property data values for the same N vacuum residues, said second set of data property values comprising penetrability values and softening point values,

(iii) generating using statistical methods :

the first correlation correlating the penetrability value of said second said of property data values to said first set of property data values for each of the N vacuum residues,
the second correlation correlating the softening point value of said second said of property data values to said first set of property data values for each of the N vacuum residues.

3. Method according to claim 2, wherein, in steps (i) and (ii), each of the vacuum residues is issued from a single crude oil.

4. Method according to any one of claims 1 to 3, wherein, in step (b) :

- the first correlation of the estimating model correlates the penetrability property data value to the values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C and the sulphur content,
- the second correlation of the estimating model correlates the softening point property data value to the values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and the asphaltene content.

5. Method for homologating a crude oil, comprising :

- (A) receiving an estimate of the penetrability value and an estimate of the softening point value of a vacuum residue issued from said crude oil, these estimated values being determined by implementing the estimating method of any of the preceding claims,
- (B) optionally calculating an estimate of the penetration index value of said vacuum residue using the estimate of the penetrability value and the estimate of the softening point value received in step (A),
- (C) comparing the estimated values of said vacuum residue received in step (A), and optionally calculated in step (B), with threshold values required for using a vacuum residue for a particular application,
- (D) deciding of the homologation of the crude oil from which is issued said vacuum residue intended for said particular application.

6. Method for homologating a crude oil according to claim 5, in which step (D) comprises:

a step (D1) comprising deciding a crude oil is homologated to produce a vacuum residue for said particular application when the estimated values comply with said threshold values, otherwise deciding the crude oil is not homologated.

7. Method for homologating a crude oil according to claim 5, in which step (D) comprises:

a step (D'1) comprising:

- when the estimated values comply with said thresholds values :

measuring at least the penetrability at 25°C and softening point of the vacuum residue, and optionally calculating the penetration index from the measured values of penetrability at 25°C and softening point, comparing the measured values of penetrability at 25°C and softening point, and optionally the calculated penetration index with said thresholds values, deciding the crude oil is homologated if the measured values comply with said threshold values, otherwise deciding the crude oil is not homologated.

- when the estimated values do not comply with said thresholds values, deciding the crude oil is not homologated.

8. System for estimating properties of a vacuum residue issued from a crude oil comprising :

- (a) receiving means arranged for receiving a set of property data values of a vacuum residue of a crude oil, said set of property data comprising the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and optionally the asphaltene content,
- (b) treatment means arranged for calculating an estimate of a penetrability value and an estimate of a softening point value of said vacuum residue using an estimating model arranged to calculate an estimate of the penetrability property data value and an estimate of the softening point property data value of a vacuum residue from the set of property data values of said vacuum residue received in step (a), said estimating model including :

a first correlation correlating a penetrability property data value to said set of property data values,
a second correlation correlating a softening point property data value to said set of property data values.

9. System according to claim 8, wherein it further comprises:

- (i) receiving means for receiving a first set of property data values for a number N of vacuum residues, said first set of data property values comprising values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and optionally the asphaltene content for each of the N vacuum residues,
- (ii) receiving means for receiving a second set of property data values for the same N vacuum residues, said second set of data property values comprising penetrability values and softening point values,

(iii) treatment means for generating using statistical methods :

a first correlation correlating the penetrability property data value of said second said of property data values to said first set of property data values for each of the N vacuum residues,
a second correlation correlating the softening point property data value of said second said of property data values to said first set of property data values for each of the N vacuum residues.

10. System according to claim 8 or 9, wherein, the estimating model includes :

- the first correlation which correlates the penetrability property data value to the values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C and the sulphur content,
- the second correlation which correlates the softening point property data value to the values of the kinematic viscosity at 100°C, the kinematic viscosity at 135°C, the sulphur content and the asphaltene content.

11. System for homologating a crude oil, comprising :

- receiving means for receiving an estimate of the penetrability value and an estimate of the softening point value of a vacuum residue issued from said crude oil, these estimated values being received from the estimating system according to any of claims 8-10,
- treatment means arranged to :

optionally calculate an estimate of the penetration index value of said vacuum residue using the estimate of the penetrability value and the estimate of the softening point value received by the receiving means, compare the estimated values of said vacuum residue received by the receiving means, and optionally calculated, with threshold values required for using a vacuum residue for a particular application, decide of the homologation of the crude oil from which is issued said vacuum residue intended for said particular application.

12. System according to claim 11, wherein the treatment means are arranged to :

- decide a crude oil is homologated to produce a vacuum residue for said particular application when the estimated values comply with said threshold values, otherwise deciding the crude oil is not homologated,

13. System according to claim 11, wherein the treatment means are arranged to :

- when the estimated values comply with said thresholds values :

measure at least the penetrability at 25°C and softening point of the vacuum residue, and optionally calculating the penetration index from the measured values of penetrability at 25°C and softening point, compare the measured values of penetrability at 25°C and softening point, and optionally the calculated penetration index with said thresholds values, decide the crude oil is homologated if the measured values comply with said threshold values, otherwise decide the crude oil is not homologated.

- when the estimated values do not comply with said thresholds values, decide the crude oil is not homologated.

14. System according to any of claims 11 to 13, further including the estimating system of any of claims 8 to 10 and wherein the receiving means are arranged to receive estimated data from said estimating system.

15. A computer program product comprising the instructions for carrying out the steps of the method as claimed in any of claims 1 to 7, when said instructions are executed by one or more processors.

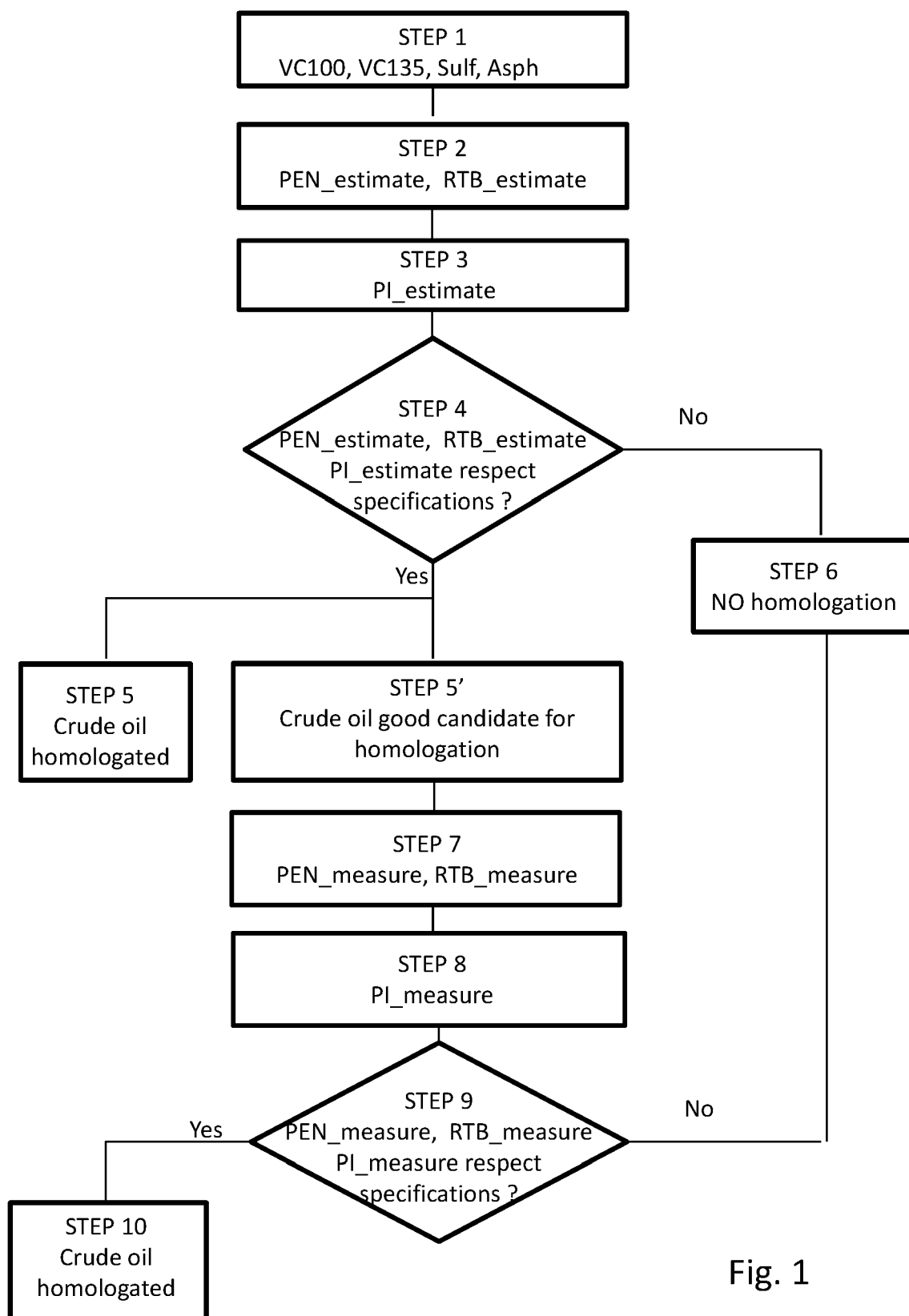


Fig. 1



EUROPEAN SEARCH REPORT

Application Number
EP 20 30 5976

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X A	US 6 011 095 A (PLANCHE JEAN-PASCAL [FR] ET AL) 4 January 2000 (2000-01-04) * example 9 *	7 1-6	INV. C10G7/06 G01N11/00
X A	WO 2015/173205 A1 (TOTAL MARKETING SERVICES [FR]) 19 November 2015 (2015-11-19) * page 11, lines 10-18 * * figure 1 *	7 1-6	
X A	US 2010/174494 A1 (DE PEINDER PETER [NL] ET AL) 8 July 2010 (2010-07-08) * paragraph [0018] *	7 1-6	
X	US 2014/156241 A1 (KUMAR RAJEEV [IN] ET AL) 5 June 2014 (2014-06-05) * paragraphs [0041] - [0052] * * figure 1 *	8-15	
			TECHNICAL FIELDS SEARCHED (IPC)
			C10G G01N
The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 17 February 2021	Examiner Pardo Torre, J
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 20 30 5976

5 This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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ORM P0459

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6011095 A	04-01-2000	AT 259861 T	15-03-2004
		AU 2902697 A	05-12-1997
		BR 9702211 A	20-07-1999
		CA 2224418 A1	20-11-1997
		CN 1195361 A	07-10-1998
		DE 69727638 T2	18-11-2004
		DK 0837910 T3	21-06-2004
		EP 0837910 A1	29-04-1998
		ES 2216149 T3	16-10-2004
		FR 2748488 A1	14-11-1997
		ID 19544 A	23-07-1998
		JP 4095117 B2	04-06-2008
		JP H11510553 A	14-09-1999
		KR 19990028874 A	15-04-1999
		MY 128318 A	31-01-2007
		NO 311526 B1	03-12-2001
		PT 837910 E	30-07-2004
		US 6011095 A	04-01-2000
		WO 9743342 A1	20-11-1997

WO 2015173205 A1	19-11-2015	FR 3021051 A1	20-11-2015
		RU 2014120190 A	27-11-2015
		WO 2015173205 A1	19-11-2015

US 2010174494 A1	08-07-2010	AT 482389 T	15-10-2010
		CN 101675332 A	17-03-2010
		EP 2142908 A1	13-01-2010
		US 2010174494 A1	08-07-2010
		WO 2008135411 A1	13-11-2008

US 2014156241 A1	05-06-2014	EP 2699900 A1	26-02-2014
		ES 2721908 T3	06-08-2019
		US 2014156241 A1	05-06-2014
		WO 2013102916 A1	11-07-2013
