COMMONWEALTH OF AUSTRALIA

Patents Act 1952

Form 1 **603919** Regulation 9

APPLICATION FOR A STANDARD PATENT OR A STANDARD PATENT OF ADDITION

~~I~~/We , BP OIL INTERNATIONAL LIMITED, a Company incorporated in accordance with the laws of England, of Britannic House, Moor Lane, London, EC2Y 9BU, England,

hereby apply for the grant of a Standard Patent ~~Patent of Addition~~ for an invention entitled: "METHOD FOR THE DIRECT DETERMINATION OF PHYSICAL PROPERTIES OF HYDROCARBON PRODUCTS"

which is described in the accompanying ~~provisional~~ complete specification.

~~I/We request that the Patent may be granted as a Patent of Addition to the Patent applied for on Application No. filed~~

~~I/We request that the term of the Patent of Addition be the same as that of the patent for the main invention or so much of the term of the patent for the main invention as is unexpired.~~

This application is a Convention application and is based on an application/s numbered 8711678; 8711680; 8711681; 8711682; 8711683; 8711684; 8711685; 8711686, all filed 18th August 1987, and 8809492 filed 12th July 1988, for a patent or similar protection made in France.

~~My~~/Our address for service is care of E. F. WELLINGTON & CO., Patent Attorney/s, 457 St. Kilda Road, Melbourne, in the State of Victoria, Commonwealth of Australia.

APPLICATION ACCEPTED AND AMENDMENTS ALLOWED 4-9-90

1002004 16/08/88

DATED this 15th day of August, A.D. 19 88

For and on behalf of
BP OIL INTERNATIONAL LIMITED,
By its Patent Attorneys,
E. F. WELLINGTON & CO.
By:

CASE NO. BPOI 6838 cog

COMMONWEALTH OF AUSTRALIA
Patents Act 1952-1969
Regulations 12(1) and 12(2)

Form 7,8

DECLARATION IN SUPPORT OF AN APPLICATION FOR A PATENT

In support of the Convention application made by BP Oil International Limited for a patent for an invention entitled:

"Method for the direct determination of physical properties of hydrocarbon products".

I, Keith Warwick DENBIGH of the Applicant's address, do solemnly and sincerely declare as follows:

1. I am authorised by BP Oil International Limited, the applicant for the patent to make this Declaration on its behalf.

2. The basic applications as defined by Section 141 of the Act were made in France on the 18th day of August, 1987 and 12th July, 1988 by BP France Societe Anonyme.

3. Alain ESPINOSA, Didier Charles LAMBERT, Andre MARTENS and Gilbert VENTRON residing at 18 Avenue de Courchelettes, 13117 Lavera, France; 3 Rue de Bretagne, 13117 Lavera, France; Les Jardinets, Rue Jules Ferry, F-13220 Chateauneuf les Martigues, France and 12 Rue de Bretagne, 13117 Lavera, France, respectively, are the actual inventors of the invention and the facts upon which BP Oil International Limited is entitled to make the application are as follows:

   The said BP France Societe Anonyme lodged the basic applications referred to in paragraph 2 as assignee of the actual inventors, and BP Oil International Limited is assignee of the said BP France Societe Anonyme.

4. The basic applications referred to in paragraph 2 were the first applications made in a Convention country in respect of the invention the subject of the application.

Declared at Britannic House, Moor Lane, London, EC2Y 9BU, England, this 30th day of August, 1988.

K. W. DENBIGH
By Power of Attorney

To: The Commissioner of Patents

(12) PATENT ABRIDGMENT (11) Document No. AU-B-20953/88
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 603919

(54) Title
METHOD FOR THE DIRECT DETERMINATION OF PHYSICAL PROPERTIES OF HYDROCARBON PRODUCTS

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(56) Prior Art Documents
AU 28675/84
AU 20954/88
AU 12009/88

(57) Claim

1. A method for the determination of the properties of the product and/or the yield of the product in a hydrocarbon conversion or separation process, said method characterised in comprising the following steps:

(a) determining with an IR spectrometer the absorbance of the feedstock at a plurality of frequencies in the spectral range 16667 to 3840 cm^{-1}, starting from a defined base line, and

(b) determining the property and/or the yield by applying a correlation between the property and yield of the product and the absorbance values of the feedstock, the correlation being

.../2

determined experimentally by multivariate regression, the correlation depending on the type of spectrometer used, the property required, and the frequencies used.

12. A method according to any one of the preceding claims wherein the hydrocarbon conversion process is a catalytic reforming process, characterised in that the method comprises the further steps of:

(c) carrying out differential spectrum measurements in the same range and at the same frequencies on the feedstock and the product,

(d) applying a correlation between the differential spectrum and the octane number gain, the correlation being determined experimentally by multivariate regression analysis,

(e) comparing the sum of the two values thus determined (octane number and octane number gain) with a desired objective, and

(f) modifying the operating conditions of the catalytic reformer until the determined octane number gain is in agreement with the desired octane number gain.

COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952-1969

603919 FORM 10

COMPLETE SPECIFICATION
(Original)

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This document contains the amendments made under Section 49 and is correct for printing

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Complete Specification for the invention entitled:

"METHOD FOR THE DIRECT DETERMINATION OF PHYSICAL PROPERTIES OF HYDROCARBON PRODUCTS"

The following statement is a full description of this invention including the best method of performing it known to me/us:

This invention relates to a method for the determination of the properties of the feedstock, the properties of the product and/or the yield of the product in a hydrocarbon conversion or separation process by carrying out NIR (near infra red) spectroscopic analyses of the feedstock and correlating these with the desired properties. The method is suitable for use on-line and in real time and for incorporation in process control.

Callis et al, Analytical Chemistry, Vol 59, No. 9, pp 624A - 636A (May 1987) mentions the possibility of determining the octane rating of an unleaded gasoline by NIR spectroscopy and point out in this particular case, the existence of a connection between other properties of the products and the NIR spectra of the products.

However, when a product is produced by converting or separating various components, which are often themselves mixtures, major problems are encountered in predicting the properties of the final products. This arises from the fact that several properties do not follow linear laws.

For certain processes, models exist which enable the prediction of products and yields. To use these, however, it is usually necessary to know the properties of the feed in detail, the chemical composition, the molecular weights of the components etc. This information is normally obtained by laboratory analyses which involves a delay in obtaining the results.

According to the present invention there is provided a method for the determination of the properties of the product and/or the yield of the product in a hydrocarbon conversion or separation process wherein the method comprises the following steps:

(a) determining with an IR spectrometer the absorbance of the feedstock at a certain number of frequencies in the spectral range 16667 to 3840 cm^{-1} (0.6 to 2.6 microns), preferably 12500 to 3840 cm^{-1} (0.8 to 2.6 micron), most preferably 4760 to 4000 cm^{-1} (2.1 to 2.5 micron) starting from a defined base line, and

(b) determining the property by applying a correlation between the property and the absorbance values, the correlation being determined experimentally by multivariate regression, depending on the type of spectrometer used, the property required, and the frequencies used.

The frequencies used may be selected from the following:

4670 cm^{-1}
4640
4615
4585
4485
4405
4390
4385
4332
4305
4260
4210
4170
4135
4100
4070
4060
4040

The corresponding frequencies expressed in statutory units (Hz) are obtained by multiplying these values by 3 x 10^{10}, the velocity of light in cm/s.

This choice is neither exhaustive nor exclusive. The choice of other frequencies will not alter the method but will require the use of other coefficients in the models enabling the calculation of the desired properties sought from the spectra.

The base line (regarded as corresponding to zero absorbance) is preferably 4780 cm^{-1}.

The processing equipment may be computer controlled by a feed-back control system for altering process conditions in response to variations in the property and/or yield of the product from the desired value, from the determination of the NIR spectrum of the feedstock.

The spectrometer may be linked to a signal processing device to permit numerical treatment of the spectrum, preferably by Fourier transformation.

The spectrometer suitably has a resolution power of 4 cm^{-1}.

The method may be used on-line and in real time.

The correlation may contain linear terms, quadratic terms and homographic (ratio) terms.

According to a specific embodiment, the hydrocarbon conversion process may be a catalytic reforming process and the properties may be selected from the following: yields of gasoline (C_5^+), hydrogen and gas, octane numbers (clear or leaded), vapour pressure and density.

The correlation in this case may be either linear for a feed of relatively constant composition (for example the KUOP characteristic varying between 11.8 and 12.2 (terms p of equation (1) below, or can involve quadratic terms (terms q of equation (1) or homographic (ratio) terms (terms r of equation (1) for a feed of varying composition, the KUOP characteristic varying between 10.8 and 12.2.

The KUOP characteristic or Watson factor is expressed by the formula:

$$\sqrt{\frac{\text{boiling point (Rankine scale)}}{\text{density 60/60}}}$$

Rankine scale = absolute Fahrenheit scale

Density 60/60 = density of the product at 60°F compared to water at 60°F.

$$X = C + \sum p_i . D_i + \sum q_{ij} D_i D_j + \sum r_{ij} D_i/D_j \qquad (1)$$

D_i ... D_j are absorbances at the wavelengths in question.

The constant C and the various coefficients p, q and r are obtained from multivariate digital analysis techniques applied to a set of feedstocks used for preliminary calibration.

In the computer-assisted operation of a reformer, the NIR spectrum of the feed as it enters the unit is taken in real time and is treated as an information vector which continuously monitors the potential of the feed.

The abundance of the NIR spectrum and the experimental accuracy obtained, resulting from the accumulation of spectral data by fast Fourier transformation, means that this information is both accurate and relevant to the chemical operations involved in the reforming process. The NIR spectrum is therefore a numerical indicator of the suitability of the feed for reforming.

Furthermore, it has been observed that the differential spectrum, when measured in real time, constitutes a digital signal enabling computer assisted operation of the unit to be improved. Catalytic reforming results in an increase in octane number which is related to the severity of the operating conditions for a given state of activity of the catalyst.

It has been noted that the differential spectrum can be directly correlated with the gain in octane number (motor and/or research) of the clear or leaded product at different levels of tetraethyl or tetramethyl lead and is related to the severity of the process.

It is therefore possible to have a sensitive and accurate method of computer assisted control for a catalytic reformer by continuously monitoring the differential spectrum, enabling parameters to be adjusted to produce gasoline of the spectified octane number, regardless of variation in the quality of the feed.

With this new method of operating the unit, variations in the differential spectrum in the NIR sensitively and rapidly optimise processing conditions using a computer programme.

Thus, according to a modification of the above embodiment, the hydrocarbon conversion process may be a catalytic reforming process wherein the method comprises the further steps of:

(c) carrying out differential spectrum measurements in the same range and at the same frequencies on the feedstock and the product,

(d) applying a correlation between the differential spectrum and the octane number gain, the correlation being determined experimentally by multivariate regression analysis,

(e) comparing the sum of the two values thus determined (octane number and octane number gain) with a desired objective, and

(f) modifying the operating conditions of the catalytic reformer until the determined octane number gain is in agreement with the desired octane number gain.

In catalytic reforming the frequencies used may be selected from the following:

4670 cm^{-1}
4485
4332
4305
4210
4100
4060
4040

The correlation between the differential spectrum and the octane number gain may be a linear correlation, e.g.

$$\text{Octane gain} = \alpha_0 + \sum_{i=i}^{7} \alpha_i \; DD_i \qquad (2)$$

where α_0 and α_i are constants and DDi is the input/output differential spectrum for wavelength i.

According to another embodiment, the hydrocarbon conversion process may be a selective hydrogenation process and the property may be the maleic anhydride number of the product.

The feedstock may be a C_5-200°C fraction derived from steam cracking called steam cracked gasoline.

The process is not restricted to this type of product, however, similar gasolines being derived from other types of cracking reaction.

It is possible to determine on-line and in real time the level of diolefins in steam cracked gasoline selectively hydrogenated, starting with the NIR spectrum of the steam cracked gasoline as it enters the unit. This is done under given operating conditions and for a catalyst of defined composition and activity.

The process based on the invention is not restricted to this particular property, however, it is possible to predict other properties of the product, for example:

- the content of olefins
- the clear octane numbers and those for leaded gasolines, different levels of tetraethyl lead or tetramethyl lead

Analysis by spectrometer in the NIR range makes it possible to calculate by differing numerical methods several properties of a hydrocarbon fraction from its spectrum.

According to a further embodiment, the hydrocarbon conversion process may be a catalytic hydrocracking process and the properties may be selected from the following: total conversion of the feedstock to product and conversion to gasoline.

In this case, the frequencies used are preferably selected from the following:

4585 cm^{-1}
4390
4332
4260
4210
4170
4100
4070

The sum of the absorbances for the eight wavelengths considered is reduced to 100 and thereafter the relative percentage of absorption for a given band will be used. Hence, if D_i is the absorbance for the wavelength i, then:

$$\sum_{i=1}^{8} D_i = S \qquad (3)$$

and the percentage of relative absorption for the frequency i : p_i will be equal to:

$$p_i = \frac{D_i}{S} \times 100 \qquad (4)$$

The feedstock may be vacuum distillates, atmospheric residues, extracts derived from lube oil processing, hydrogenated residues, deasphalted residues, cracked distillates and cat cracker recycle oils.

According to yet another embodiment, the hydrocarbon conversion process may be a catalytic cracking process and the properties are selected from the following:

(i) the conversion of the feed to gasoline and the yield of residue in the product,

(ii) the density, research octane number (RON) and motor octane number (MON) of clear and leaded gasolines from the cracking process.

For catalytic cracking, the frequencies may be selected from the following:

4670 cm^{-1}
4615
4485
4135
4100
4060

The feed may be a distillate, for example, vacuum distillate gas oils, an atmospheric residue (deasphalted or not), a lube oil extract, a hydrogenated or deasphalted residue, a cracked distillate or a cat cracker recycle oil.

Some of the properties which may be determined include:

- the research octane number (RON) and the motor octane number (MON) of the gasoline produced, whether clear or leaded, and at different levels of lead,
- the cetane number of gas oil,
- the densities of various fractions produced (gasoline, gas oil, residue),
- the viscosity of the residue, etc.

By combination with computer control of a fluidised bed catalytic cracker, the NIR spectrum allows complete and accurate characterisation of the feed which, together with the processing conditions of the unit (for example, inlet and outlet temperature of the heater, C/O (ratio of the catalyst flow rate to the feed flow rate), space velocity, nature and activity of the catalyst, etc) leads to an efficient, computer model.

It is thus possible to control the unit with great precision.

According to another embodiment, the hydrocarbon conversion process may be a visbreaking process and the properties may be selected from the following: the yield of the gas oil product, the viscosity, specific gravity and xylene equivalent of the residue, and the conversion limit, i.e, the maximum conversion before an unstable residue is produced.

The feedstock may be atmospheric or vacuum distillate residues. They may be used in a blend and may have various petroleum fractions added to them: e.g. lube oil extracts, hydrogenated or deasphalted residues, cracked distillate units, catalytic cracker recycled oils, etc.

In addition to the hydrocarbon conversion processes exemplified above, the method is also applicable to the control of separation processes such as distillation.

Thus according to another embodiment, the hydrocarbon separation process may be a distillation process and the properties may be selected from the following:

(i) the quantitative composition of a mixture of crude oils constituting the feedstock when the distilland is known to be a blend of crude oils,

(ii) the yields of products obtained,

(iii) the densities and viscosities of the various products,

(iv) the cetane number, aniline point, cloud point and the KUOP characterisation factor of the gas oil,

(v) the n-paraffinic, isoparaffinic, cycloparaffinic and aromatic content of the gasoline and the research octane number (RON) and motor octane number (MON) of clear and leaded gasoline with different contents of tetraethyl or tetramethyl lead.

In computer assisted operation of a processing unit, the NIR spectrum of the feedstock entering the unit is thus taken in real time and treated as an information vector which characterises the processing properties of the feedstock in the process on a continuous basis. The detail in the NIR spectrum and the experimental precision which may be obtained from the accumulation of spectral data ensures that this information is reliable and relevant to processing operations.

The analysis and data processing time is less than 1 minute.

According to another aspect of the present invention there is provided apparatus for carrying out a method for the determination of the properties of the product and/or the yield of the product in a hydrocarbon conversion or separation process as hereinbefore described, the apparatus comprising an infra red spectrometer linked to a computer programmed in such manner that the property may be determined continuously and in real time.

The apparatus may comprise a feedback control system for computer control of the processing equipment in response to variations of the property and/or yield of the product from the desired value, from the determination of the NIR spectrun of the feedstock.

On-line analysis may use fibre optics as a means fortransmitting light signals or may involve physical transferance of a sample to the cell of the spectrometer.

The invention is illustrated but not limited with reference to the following examples:

Example 1 - Catalytic Reforming

Operating conditions of the reformer were as follows:

reactor temperature : 510°C

LHSV : 1.85

catalyst : R_{12} UOP

pressure : 39 bar

The NIR spectrum of the feed at the entry to the reformer was determined at four frequencies, F_1, F_2, F_3 and F_4.

The product yield and RON of the product were then predicted using the following equations (5) and (6):

$$\text{RON clear} = 2151.36\ D_5 + 131.22\ D_{14} + 280.67\ D_9 - 243.157 \qquad (5)$$

$$\text{Yield } C_5^+ = -\ 114.84\ D_{14} + 227.18\ D_{16} + 338.81\ D_9 - 249.3\ \%\text{vol} \qquad (6)$$

D_i represents the absorbance of the NIR spectrum at frequency F_i, the zero base being taken as 4780 cm^{-1}.

The predictions were then compared with the actual results obtained by conventional means.

The feedstock was a highly paraffinic naphtha having a KUOP factor of 12.2, a 90% ASTM distillation point of 135°C and a density d^{15} of 0.686.

NIR spectrum:

Frequency		Absorbance	
F_1	4485 cm^{-1}	D_1	0.0347
F_2	4100	D_2	0.55594
F_3	4060	D_3	0.61466
F_4	4305	D_4	0.65502

	Yield %vol	RON Clear
Predicted properties from equations (5) and (6)	81.2	88.3
Actual properties by experiment	80.4	88.0

Example 2 - Catalytic Reforming

Example 1 was repeated using a diferent feedstock.

This was a moderately paraffinic naphtha having a KUOP factor of 11.8, a 90% ASTM distillation point of 148°C, and a density d^{15} of 0.7421.

Absorbance values at the same frequencies as Example 1 were:

D_1	0.0364
D_2	0.52127
D_3	0.60595
D_4	0.66828

	Yield %vol	RON Clear
Predicted properties from equations (5) and (6)	88.3	91.1
Actual properties by experiment	87.6	91.4

Example 3 - Catalytic Reforming

(1) The octane number of the naphtha feedstock to the reformer was obtained from its NIR spectrum at the following frequencies:

Frequency		Absorbance	
F_1	4670 cm^{-1}	D_1	0.01223
F_2	4485	D_2	0.03878
F_3	4332	D_3	0.9339
F_4	4100	D_4	0.6167
F_5	4040	D_5	0.6083
F_6	4305	D_6	0.6794
F_7	4210	D_7	0.4800

The research octane number values were obtained after adding 0.4 g/l tetraethyl lead.

Operating conditions of the reformer were as follows:-

reactor temperature	510°C
LHSV	1.85
catalyst	R_{12} UOP
pressure	39 bar

As before, the octane number ON of the product from the feed is predicted by equation (1) in the form:

$$ON = C + \sum p_i D_i + \sum q_{ij} D_i D_j + \sum r_{ij} D_i/d_j$$

The constant C and the various coefficients p,q and r are obtained from multivariate digital analysis techniques applied to a set of feedstocks used for preliminary calibration.

Equation (1) becomes:

$$(RON - 0.4) = - 65.52 - 3.178\ D_4.D_6 + 180.3\ D_2 - 6.32\ D_3 - 1814\ D_5.D_6 + 1232.D_5 + 1165.D_6 - 109\ D_7$$

The RON calculated by means of Equation (1) is 83.2 compared with an experimental value of 83.5.

A similar procedure could be followed for other clear or leaded octane numbers.

(2) The octane gain is estimated by Equation (2):

$$\text{Octane gain} = \alpha 0 + \sum_{i=i}^{7} \alpha i\ D.D_i$$

where $\alpha 0$ and αi are constants and D D_i is the input/output differential spectrum for the frequency i.

The values of the constants are as follows:

$\alpha 0 = 25.32$

$\alpha 1 = 136.48$

$\alpha 2 = -268.14$

$\alpha 3 = 51.15$

$\alpha 4 = 138.756$

$\alpha 5 = -68.40$

$\alpha 6 = 61.85$

$\alpha 7 = -288.12$

The absorbance of the NIR spectrum of the feedstock and product were as follows for the frequencies investigated:

Under these conditions Equation (2) gives an octane increase of 13.5.

(3) In view of the quality of the feedstock and the product specification an octane increase of 15 is required.

Consequently the reforming temperature must be progressively increased until the required gain is achieved. This operation is carried out by increasing the temperature by 4°C.

In this operation the differential spectrum serves as a guide in increasing the temperature. This type of adjustment based on the differential spectrum remains valid even if the nature of the feedstock changes during adjustment of the temperature.

<u>Example 4 - Selective Hydrogenation</u>

The operating conditions of a gasoline selective hydrogenation unit were as follows:

- temperature entering the reactor : 70°C
- temperature emerging from the reactor : 170°C
- through-put of hydrogen : 45 Nm^3/m^3 of batch
- V.V.h = 4
- total pressure : 15 bars
- palladium catalyst : Procatalyse LD 265.

The unit was fed with a steam cracked gasoline having a maleic anhydride number of 40.2. The hydrogenated gasoline has a maleic anhydride number of 7.

Frequencies cm^{-1}		Spectrum of Feedstock Absorbance	Spectrum at Outlet from reactor Absorbance
4670	D_1	0.18186	0.18476
4615	D_2	0.14555	0.14888
4585	D_3	0.16501	0.17428
4485	D_4	0.11747	0.07242
4332	D_5	0.56587	0.58186
4260	D_6	0.46701	0.4934
4170	D_7	0.29995	0.30184
4100	D_8	0.35576	0.34264
4060	D_9	0.95571	0.98812
4040	D_{10}	0.50597	0.50567
4640	D_{11}	0.14633	0.15406
4385	D_{12}	0.41282	0.40466
4305	D_{13}	0.48511	0.50243
4210	D_{14}	0.29911	0.29836
4135	D_{15}	0.28855	0.28183

Using the spectrum of the feedstock, the value of the maleic anhydride number of the hydrogenated gasoline emerging from the reactor was determined by means of the following relation:

Maleic anhydride number =

$$- 325.05 - 1744.6.D_1 + 649.D_2 - 565.5.D_3 + 813.1.D_4 + 349.3.D_5 - 356.9.D_6 + 845.8.D_7 + 528.9.D_8 + 285.7.D_9 + 116.1.D_{10} + 124.3.6.D_{11} + 445.6.D_{12} - 333.9.D_{13} + 182.2.D_{14} - 1773.4.D_{15}$$

The calculated value is 5.6 as against 7 by measurement.

Example 5 - Selective Hydrogenation

A similar exercise was carried out on the feedstock to the selective hydrogenation unit of Example 4.

The maleic anhydride number values were measured on the feedstock, using the feedstock spectrum and on the hydrogenated gasoline product using the feedstock spectrum according to the following relationship:maleic anhydride number = - 283.05 - 1704.2.D_1 + 633.9.D_2 - 552.4.D_3 + 794.2.D_4 + 341.2.D_5 - 348.6.D_6 + 826.2.D_7 + 516.7.D_8 + 279.1.D_9 + 113.4.D_{10} + 1214.8.D_{11} + 435.25.D_{12} - 326.1.D_{13} + 178.1.D_{14} - 1732.3.D_{15}

The following results are obtained:

	Calculated	Measured
Maleic anhydride number on entry	40	40.2
Maleic anhydride number at exit	8	7.8

This example shows that it is also possible to determine the value of the maleic anhydride number directly, using the product spectrum.

Example 6 - Hydrocracking

The feedstock was a distillate derived from crude petroleum from the Cameroons, KOLE.

The relative absorbance spectrum as a percentage of the total absorbance is as follows:

D_4	18.43 %
D_5	13.06 %
D_6	13.98 %
D_7	12.12 %
D_8	11.87 %

The KUOP factor of the feedstock was obtained from the following equation:

KUOP = 0.618 D_4 + 0.36 D_5 + 1.07 D_6 - 0.248 D_7 + 1.032 D_8 - 28.5

The KUOP factor as measured was 12.31 compared with a calculated value of 12.2.

Example 7 - Hydrocracking

A heavy aromatic distillate of KUOP factor 10.9 was used.

The relative absorbance spectrum of this feedstock was as follows:

D_4	16.93 %
D_5	12.76 %
D_6	12.77 %
D_7	10.94 %
D_8	12.50 %

The value for Conradson carbon residue may be determined by the following equation:

$$C\%\ \text{weight} = 36.84 - 3.77.D_4 - 2.42.D_5 + 8.444\ D_6 - 6.473.D_7 + 2.023.D_8$$

The value determined in this way is 4.5% by weight as against 5.5% by weight by experiment. This agreement is satisfactory, taking into account experimental accuracy.

Other correlations can be obtained for the nitrogen content and density of the feed.

Example 8 - Hydrocracking

The operating conditions used in this example were as follows:

Reactor - entry temperature	400°C
Reactor - exit temperature	430°C
Total pressure	155 bars
Hydrogen flow rate	1300 N m^3/m^3
Average temperature	423°C

Catalyst : molybdenum cobalt on aluminium silicate.

The following correlations ccan be established for these operating conditions:

$$\text{Conversion \% by weight} = A + B \log_{10} \left(\frac{D_8}{D_4}\right) \qquad (7)$$

$$\text{Conversion into gasoline \% by weight} = C \frac{D_8}{D_4} - D \qquad (8)$$

A, B, C and D are constants linked to the operating conditions of the reactor.

For the conditions described above, the values of these constants are:

A = 123.12   B = 277.14   C = 35.893   D = 8.55

The unit is fed with a distillate, the characteristics of which are as follows:

Density : 0.952
KUOP factor : 11.5
nitrogen content : 1735 ppm
Conradson carbon residue : 0.42% by weight
Saturated hydrocarbons : 37.20% by weight
Aromatic hydrocarbons : 61.30% by weight

The absorption spectrum of the feedstock gives the following relative absorbance factors:

$D_8 = 12.2$

$D_4 = 17.75$

The application of equations (3) and (4) gives the following results by comparison with experimental figures:

	Calculated	Measured
Conversion into gasoline % by weight	16.1	15
Total conversion	78	79.5

The reactor operating in the same conditions is fed with a different batch whose characteristics are as follows:

Density	0.920
KUOP factor	11.7
Nitrogen content	1300 ppm
Carbon residue	0.26% by weight

The relative absorbance factors are as follows:

$D_8 = 12.19$

$D_4 = 18.05$

The equations (7) and (8) give the following results:

	Calculated	Measured
Conversion into gasoline % by weight	15.7	16.5
Total conversion	75.9	75

Example 9 - Catalytic Cracking

In this and the following Examples 10, 11 and 12 operating conditions were as follows:

Temperature at entry to riser : 250°C
Temperature at exit from riser : 525°C
Mass flow rate : 78 kg/h per kg
C/O : 6.6
Catalyst MAT activity ASTM-D 3907 : 65

The feedstock had a KUOP factor of 11.95 and had the following absorption spectrum:

$F_1 = 4615 \text{ cm}^{-1}$ $D_1 = 0.02395$
$F_2 = 4135$ $D_2 = 0.55726$
$F_3 = 4100$ $D_3 = 0.5193$
$F_4 = 4485$ $D_4 = 0.04015$

The gasoline yield had the following characteristics: ASTM distillation : initial boiling point 38°C, 90% distilled at 190°C, being 47.7% by weight.

The yield of residue following the ASTM distillation provided 10% distilled at 385°C, being 9% by weight.

The yields can be obtained from the feedstock spectrum by the following relationships, for the control conditions for the unit as given below:

$$\text{Yield of gasoline \% by weight} = 59.3 - 280 \frac{D_1}{D_2}$$

$$\text{Yield of residue \% by weight} = \frac{496.7}{\left(\frac{D_3}{D_4}\right)^{1.55}}$$

The results obtained are 47.27% for the yield of gasoline and 9.39% for the yield of residue, using these proportions.

Example 10 - Catalytic Cracking

A feedstock having a KUOP factor of 11.6 was used with the following spectrum measured under the same conditions as in Example 9:

$F_1 = 4615 \text{ cm}^{-1}$ $D_1 = 0.3357$

$F_2 = 4135$ $D_2 = 0.52092$
$F_3 = 4100$ $D_3 = 0.48834$
$F_4 = 4485$ $D_4 = 0.04989$

The observed and calculated yields using the proportions as in Example 9 are:

	Observed	Calculated
Yield of gasoline % by weight	40.6	41.25
Yield of residue % by weight	15	14.47

Example 11 - Catalytic Cracking

A feed of KUOP factor = 11.95 was used. This had the following absorption spectrum:

$F_1 = 4670\ \mathrm{cm}^{-1}$ $D_1 = 0.00977$
$F_2 = 4485$ $D_2 = 0.04015$
$F_3 = 4100$ $D_3 = 0.5193$
$F_4 = 4060$ $D_4 = 0.52665$

The following feed properties are obtained by the following relationships:

Density of the feed $= 0.96497 + 4.1D_1 - 0.1935D_3$

Conradson carbon residue of the feed as % by weight $= -7.744 + 16.07D_3 + 48.175D_1$

Aniline point of feed in °C $= 70 - 500\ (D_4/D_3) - 1.066$

The experimentally determined values and those calculated with the aid of the above correlations are as follows:

	Calculated	Found Experimentally
Density at 15°C	0.905	0.908
% weight carbon residue	1.07	1.10
Aniline point °C	96	94.4

Example 12 - Catalytic Cracking

A feed of KUOP factor = 11.6 was used. The gasoline obtained by cracking had the following characteristics:

Distillation : initial boiling point 38°C, 90% distilled at 190°C

Density at 15°C : 0.749

Research octane number after addition of lead tetraethyl at 0.4 g/l : 92.4

The absorption spectrum for the feed is measured under the following conditions:

Frequencies	Absorbance
F_1 = 4670 cm^{-1}	D_1 = 0.0155
F_2 = 4485	D_2 = 0.05079
F_3 = 4100	D_3 = 0.48692
F_4 = 4060	D_4 = 0.51066

The characteristics of the gasoline produced are obtained from the spectrum of the feed from the following relationships:

Density of the gasoline = $0.7553 - 0.0355 \log_{10} (D_3/6.2D_2)$

Research octane number after addition of lead tetraethyl at 0.4 g/l (RON 0.4) = $93.6 - 4.82 \log_{10} (D_3/6.2D_2)$

The calculated values are d = 0.7486 and octane number 92.7 for experimentally determined values of d = 0.749 and octane number 92.4.

The process used therefore accurately predicts the experimental results obtained from the unit operated under the conditions established above.

Example 13 - Visbreaking

A feed was used consisting of a vacuum distillation residue obtained from a crude of Kuwait origin distilling above 565°C.

The product temperature on leaving the visbreaker was 481.4°C.

The following were determined from the NIR spectrum of the unit feed:

- the visbreaking conversion,
- the xylene equivalent of the visbreaking residue,
- the gasoline yield.

The absorbances were determined at the following frequencies from the feed spectrum:

Frequency (cm^{-1})	Absorbance	
4670	D_1	0.00964
4640	D_2	0.02301
4615	D_3	0.02961
4585	D_4	0.03566
4485	D_5	0.05314
4385	D_6	0.3485

4332	D_7	0.6971
4305	D_8	0.4710
4260	D_9	0.5692
4210	D_{10}	0.4019
4170	D_{11}	0.3974
4135	D_{12}	0.3546
4100	D_{13}	0.3185
4060	D_{14}	0.3381
Frequency (cm^{-1})	Absorbance	
4040	D_{15}	0.2695
4405	D_{16}	0.255

The xylene equivalent of the visbroken residue was determined by the following equation which links the absorbances and the reaction temperature T:

$$\text{Xylene equivalent} = -1553.9 + 1.849\,T - 162\,D_1 + 6824.8\,(D_3) + 1027.9\,(D_5) + 2480.9\,(D_6) + 598.3\,(D_7) - 1717.2\,(D_{10}) + 7368.3\,(D_{11}) - 8313\,(D_{14}) - 573.4\,(D_{15}) - 262.6\,(D_{16})$$

The experimentally determined xylene equivalent had a value of 79 as against a calculated value of 78.9.

The gasoline yield (C_5 cut - 185°C) was obtained by the following equation:

$$\text{Gasoline yield} = 2.73 + 1166.2\,(D_5) + 46.59\,(D_8) - 253.03\,(D_{13})$$

The residue yield (cut point above 350°C) was obtained by the following equation:

$$\text{Residue yield} = 20.46 + 2934.4\,(D_1) - 3905.6\,(D_2) + 173.39\,(D_7)$$

The gasoline and residue yields determined by the equations above are respectively 6.07 and 79.75% weight.

A comparison with the values noted is shown below:

	Calculated	Actual
Xylene equivalent	79	78.9
Gasoline yield % weight	6.07	7.7
Residue yield % weight	79.75	78

Example 14 - Visbreaking

The reaction temperature which will give an xylene equivalent of 70 and obtain the corresponding gas oil yield was determined.

The temperature may be obtained by the following equation which links the xylene equivalent and the reaction temperature RT in °C.

$$RT = 840.4 + 0.54 \text{ xylene equivalent} + 87.6 (D_1) - 3691.1 (D_3) - 555.9 (D_5) - 1341.75 (D_6) - 323.6 (D_7) + 928.7 (D_{10}) - 3985 (D_{11}) + 4495.9 (D_{14}) + 310.1 (D_{15}) + 142 (D_{16})$$

The unit used operates under the same conditions as Example 13 and was fed with a vacuum distillation residue of Brent origin, the NIR spectrum of which has the following characteristics:

Frequency (cm^{-1})	Absorbance	
4670	D_1	0.01898
4640	D_2	0.03318
4615	D_3	0.03889
4585	D_4	0.04543
4485	D_5	0.06489
4385	D_6	0.3789
4332	D_7	0.7612
4305	D_8	0.5216
4260	D_9	0.6501
4210	D_{10}	0.4613
4170	D_{11}	0.4646
4135	D_{12}	0.4265
4100	D_{13}	0.389
4060	D_{14}	0.4041
4040	D_{15}	0.332
4405	D_{16}	0.2688

The temperature determined by means of the above equation and the spectrum above was 480.4°C.

The gas oil yield was obtained by the following equation, linking the reaction temperature in °C and the absorbances:

$$\text{Gas oil yield, \% weight} = - 290.45 + 0.34 (RT) + 626.5 (D_6) - 234.34 (D_{14})$$

The gas oil yield obtained for the calculated temperature of 480.4°C was 15.6% weight.

The actual and predicted results are given below:

	Predicted	Actual
Xylene equivalent	70	60
RT °C	480.4	480
Gas oil yield, % weight	15.6	14.8

Example 15 - Visbreaking

The conversion limits of the Kuwait and Brent feedstocks of Examples 13 and 14 were determined from the spectra given in Examples 13 and 14 under the operating conditions of Example 13.

The conversion limit value was determined by the following equation:

$$CL = -53.05 + 917.83\ (D_5) + 1903.94\ (D_{12}) - 2090.1\ (D_{13}) + 55.86\ (D_{16})$$

The actual and calculated values were as follows:

	Calculated	Actual
Brent	20.5	21.1
Kuwait	19.4	19.9

Example 16 - Distillation

In this Example and the following Examples 17 - 23, the experimental distillation procedure was carried out on a distillation column having an efficiency of 14 theoretical plates.

The NIR spectra of three crude oils was determined. The values of the absorbances for these three crude oils are as follows at the frequencies used, the base line being taken as 4870 cm^{-1}:

Crude Oil	Absorbances D_1			
	Iranian Heavy	Zakum	Djeno	Mixture 50-35-15
Frequencies F_i cm^{-1}				
F_3 : 4615	0.02736	0.0248	0.01758	0.0251
F_4 : 4585	0.02671	0.02562	0.01895	0.0255
F_5 : 4485	0.03811	0.03981	0.02172	0.0379
F_7 : 4332	0.93621	0.96899	0.94434	0.9494
F_9 : 4260	0.77561	0.79598	0.80701	0.7876
F_{11} : 4170	0.55478	0.56599	0.57862	0.5622
F_{13} : 4100	0.49855	0.50971	0.51222	0.5050
F_{14} : 4060	0.53475	0.54304	0.52624	0.5361
F_{15} : 4040	0.41833	0.42156	0.41788	0.4196

A homogeneous mixture of the three crude oils was produced in the following proportions: Iranian Heavy : 50% by weight - Zakum : 35% by weight - Djeno : 15% by weight.

The last column in the above table indicates the absorbances obtained by analysis of the mixture.

By a least square method the nine equations are solved in relation to the three unknowns (% by weight of each of the crude oils in the mixture).

The values thus calculated are as follows, in % by weight:

Iranian Heavy : 51.4% - Zakum : 34.2% - Djeno : 14.4%

The accuracy, in relation to the control mixture, is therefore satisfactory, the differences observed being acceptable and due to the repeatability of the absorbance measurements.

This method enables the composition of a mixture of known crude oils to be obtained in real time.

It is therefore possible, if the distillation unit is supplied from tanks containing different crude oils, to know, in real time, the composition of the mixture of crude oils which is supplied to the distillation unit.

This method may be applied to measuring the supplies of mixed crude oils. Example 17 - Distillation

A Djeno crude oil was used and the absorbances D_i were measured for 12 frequencies F_i. The results are summarised below:

F_2 : 4640 cm^{-1} D_2 = 0.01364
F_3 : 4615 D_3 = 0.01758
F_4 : 4584 D_4 = 0.01895
F_5 : 4485 D_5 = 0.03172
F_6 : 4385 D_6 = 0.45629
F_7 : 4332 D_7 = 0.94434
F_8 : 4305 D_8 = 0.63842
F_9 : 4260 D_9 = 0.80701
F_{11} : 4170 D_{11} = 0.57862
F_{13} : 4100 D_{13} = 0.51222
F_{14} : 4060 D_{14} = 0.52624
F_{15} : 4040 D_{15} = 0.41788

The yields of the defined fractions previously obtained by multivariate regression are expressed as a function of the absorbances D_i of the different frequencies by the following expressions:

Residue output = $359.88 - 1385.51\ D_2 - 441.15\ D_6 - 291.84\ D_7 + 225.1\ D_8 + 128.33\ D_{15}$

Light distillate output = $-\ 113.6 + 541.26\ D_2 - 108.75\ D_6 + 122.71\ D_7 - 55.49\ D_8 - 31.64\ D_{15}$

Gas oil output = $-\ 54.03 + 373.77\ D_2 + 199.13\ D_6 + 67.77\ D_7 - 101.61\ D_8 - 57.92\ D_{15}$

The calculation carried out on the basis of the above formulae and of the absorbances gives the following results, compared with the experimental values observed for the Djeno crude oil examined here:

	Calculated	Observed
Yield of residue, % by weight	61.4	63

Light distillate	10.6	9.1
Gas oil	16.8	16.2

If this type of regression method is extended to the entire distillation range, it is then possible to calculate the distillation curve and the mean distillation temperature of the feed in real time.

This method is also applicable to mixtures and feedstocks of variable compositions.

In a computer-assisted process in a distillation unit, the NIR spectrum of the feedstock entering the unit is thus taken in real time and treated as an information vector which characterises the distillation properties of the feedstock in the distillation process on a continuous basis. The amount of detail in the near infra red spectrum and the experimental accuracy which may be obtained as required from the accumulation of spectral data, using fast Fourier transformation, ensures that this information is reliable and relevant to distillation.

In the following examples it is demonstrated that for a fixed set of conditions in the unit, the variations of the quantity of the products formed may be correlated, by means of a numerical treatment, with variations in the NIR spectrum of the feedstock.

Example 18 - Distillation

The Conradson carbon residue according to Standard T60-116) of a crude petroleum of Mexican ISTHMUS origin was 4.1% by weight.

The absorbances of a test sample of this crude petroleum were measured for 3 frequencies, and had the following values:

F_2 : 4640 cm^{-1} $D_2 = 0.0186$
F_7 : 4332 $D_7 = 0.9435$
F_8 : 4305 $D_8 = 0.6374$

The value for the carbon residue was calculated from the following equation:

$$C = 84.306 - 64.1794\ D_7 - 252.437\ D_2 - 22.432\ D_8/D_7$$

In the case of the crude petroleum, the figure of 3.9% by weight was obtained, which was satisfactory, taking into account the accuracy of the test.

Example 19 - Distillation

The asphaltene content of a crude petroleum of Suez Blend origin, measured by the AFNOR T60 - 115 method was 1.5% by weight.

The NIR spectrum of this crude was determined at the following frequencies and gave the following absorbances:

F_2 : 4640 cm^{-1} D_2 = 0.01868
F_7 : 4332 D_7 = 0.93972
F_8 : 4305 D_8 = 0.6358
F_2 : 4640 cm^{-1} D_2 = 0.0210
F_7 : 4332 D_7 = 0.9362
F_8 : 4305 D_8 = 0.6427

The asphaltene content was obtained by the following equation correlating the absorbances:

$$\text{Asphaltenes (\% by weight)} = 22.31\ (3.758 - 2.861\ D_7 - 11.253\ D_2 - D_8/D_7)^{1.5} - 0.465$$

The calculated value was 1.3% by weight which was satisfactory.

Example 20 - Distillation

A gas oil fraction was used which was taken from a Iranian Heavy crude petroleum. The cetane number of this fraction was 49.6 (Standard MD7 - 035).

The NIR spectrum was obtained for the crude petroleum. Absorbances D_i were measured at the following frequencies:

The absorbance values were used in the following equation in order to calculate the cetane number of the gas oil from the NIR spectrum of the crude:

$$\text{Cetane number} = 155.529 - 12.053\ D_7 - 47.408\ D_2 - 135.217\ D_8/D_7$$

The value obtained was 50.4.

Thus, the above equation enables the cetane number of a gas oil to be determined as a function of the NIR spectrum of the feedstock.

Example 21 - Distillation

The aniline point of a gas oil fraction from a light crude petroleum of Lokele, Cameroons origin, was determined (Standard ASTM-D611). This was 52°C.

The values for the absorbances measured at the following 4 frequencies of the NIR spectrum of this crude petroleum were:

F_6 : 4385 cm^{-1} $D_6 = 0.5181$
F_7 : 4332 $D_7 = 0.8268$
F_8 : 4305 $D_8 = 0.6786$
F_{15} : 4040 $D_{15} = 0.4293$

The following equation enabled the aniline point to be calculated from the absorbances of the crude:

$$\text{Aniline point of the gas oil produced} = 27.1 + 53.57\ D_7 + 113.8\ D_{15} + 199.6\ D_8 - 391.2\ D_6$$

The value thus calculated was 53°C.

Example 22 - Distillation

The cloud point of a gas oil fraction taken from an Egyptian Belayim Crude was -10°C (Standard T60 - 105).

The values for the absorbances measured at the following four frequencies of the NIR spectrum of a sample of this crude petroleum were:

F_5 : 4485 cm^{-1} $D_5 = 0.0335$
F_6 : 4385 $D_6 = 0.45821$
F_{13} : 4100 $D_{13} = 0.49486$
F_{15} : 4040 $D_{15} = 0.41039$

Example 23

The viscosity at 60°C of the 369 - 509°C fraction of a Congolese Djeno crude petroleum was 27.2 cSt.

The values for the absorbances measured at four frequencies of the NIR spectrum of a sample of this crude petroleum were:

F_2 : 4640 cm^{-1} $D_2 = 0.01364$
F_7 : 4332 $D_7 = 0.94434$
F_8 : 4305 $D_8 = 0.63842$
F_{14} : 4060 $D_{14} = 0.52624$

The value calculated by introducing the measured absorbances in the following equation was:

$$\log_{10} \text{viscosity at } 60°C = 1.4468 - 5.662\ D_7 + 0.874\ D_{14} - 29.980\ D_2 + 8.310\ D_8 = 28.5 \text{ cSt}$$

The equation enabled the viscosity of the residue to be calculated from the NIR spectrum of the feedstock.

The matter contained in each of the following claims is to be read as part of the general description of the present invention.

The claims defining the invention are as follows:

1. A method for the determination of the properties of the product and/or the yield of the product in a hydrocarbon conversion or separation process, said method characterised in comprising the following steps:

(a) determining with an IR spectrometer the absorbance of the feedstock at a plurality of frequencies in the spectral range 16667 to 3840 cm^{-1}, starting from a defined base line, and

(b) determining the property and/or the yield by applying a correlation between the property and yield of the product and the absorbance values of the feedstock, the correlation being determined experimentally by multivariate regression, the correlation depending on the type of spectrometer used, the property required, and the frequencies used.

2. A method according to claim 1 wherein equipment used in processing is computer controlled by a feedback control system for altering process conditions in response to variations in the property and/or yield of the product from the desired value, from the determination of the NIR spectrum of the feedstock.

3. A method according to either of the preceding claims wherein the frequencies are in the spectral range 12500 to 3840 cm^{-1}.

4. A method according to claim 3 wherein the frequencies are in the spectral range 4760 to 4000 cm^{-1}.

5. A method according to any one of the preceding claims characterised in that the frequencies used are selected from the following:

4670 cm^{-1}
4640
4615
4585

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4485

4405

4390

4385

4332

4305

4260

4210

4170

4135

4100

4070

4060

4040 .

6. A method according to any one of the preceding claims characterised in that the base line is taken as 4780 cm^{-1}.

7. A method according to any one of the preceding claims wherein the spectrometer is linked to a signal processing device to effect numerical treatment of the spectrum.

8. A method according to claim 7 wherein the treatment is by Fourier transformation.

9. A method according to any one of the preceding claims wherein the method is on-line and in real time.

10. A method according to any one of the preceding claims wherein the correlation contains linear terms, quadratic terms and homographic terms.

11. A method according to any one of the preceding claims wherein the hydrocarbon conversion process is a catalytic reforming process and the properties are selected from the following: yields of gasoline (C_5+), hydrogen and gas, octane numbers (clear or leaded), vapour pressure and density.

12. A method according to any one of the preceding claims wherein the hydrocarbon conversion process is a catalytic reforming process, characterised in that the method comprises the further steps of:

(c) carrying out differential spectrum measurements in the same range and at the same frequencies on the feedstock and the product,

(d) applying a correlation between the differential spectrum and the octane number gain, the correlation being determined experimentally by multivariate regression analysis,

(e) comparing the sum of the two values thus determined (octane number and octane number gain) with a desired objective, and

(f) modifying the operating conditions of the catalytic reformer until the determined octane number gain is in agreement with the desired octane number gain.

13. A method according to either of claims 11 or 12 wherein the frequencies used are selected from the following:

4670 cm^{-1}
4485
4332
4305
4210
4100
4060
4040 .

14. A method according to any one of claims 1 to 10 wherein the hydrocarbon conversion process is a selective hydrogenation process and the property is the maleic anhydride number of the product.

15. A method according to any one of claims 1 to 10 wherein the hydrocarbon conversion process is a catalytic hydrocracking process and the properties are selected from the following: the total conversion of the feedstock to product and the conversion to gasoline.

16. A method according to claim 15 wherein the frequencies used are selected from the following:

4585 cm^{-1}
4390
4332
4260
4210
4170
4100
4070 .

17. A method according to any one of claims 1 to 10 wherein the hydrocarbon conversion process is a catalytic cracking process and the properties are selected from the following:

(i) the conversion of the feed to gasoline and the yield of residue in the product,

(ii) the density, research octane number (RON) and motor octane number (MON) of clear and leaded gasoline from the cracking process.

18. A method according to claim 17 wherein the frequencies are selected from the following:

4670 cm^{-1}
4615
4485
4135
4100
4060 .

19. A method according to any one of claims 1 to 10 wherein the hydrocarbon conversion process is a visbreaking process and the properties are selected from the following: yield of the gas oil product, the viscosity, specific gravity and xylene equivalent of the residue, and the conversion limit.

20. A method according to any one of claims 1 to 10 wherein the hydrocarbon separation process is a distillation process and the properties are selected from the following:

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(i) the quantitative composition of a mixture of crude oils constituting the feedstock when the distilland is known to be a blend of crude oils,

(ii) the yields of products obtained,

(iii) the densities and viscosities of the products,

(iv) the cetane number, aniline point, cloud point and KUOP characterisation factor of the gas oil product,

(v) the n-paraffinic, isoparaffinic, cycloparaffinic and aromatic content of the gasoline and the research octane number (RON) and motor octane number (MON) of clear and leaded gasoline.

DATED this 20th day of August, 1990

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