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(54) **Title**: APPARATUS FOR ONLINE ANALYSIS OF VISCOUS FLUIDS USING SPECTROSCOPY

(57) **Abstract**: An apparatus is provided for online IR analysis of viscous fluids for determining their several physico-chemical properties, the said apparatus comprising a sample dilution accessory, wherein the viscous fluid sample is diluted with IR transparent diluent in homogenous manner to obtain a diluted sample, the said accessory fitted with an IR Spectrometer for evaluating the absorbance of the said sample for analysis. The invention uses an apparatus wherein sample cells of less than 0.2mm can be utilized and IR absorption is evaluated for full IR spectrum from 800 cm^{-1} to 400 cm^{-1} for predicting the properties of viscous fluids.

APPARATUS FOR ONLINE ANALYSIS OF VISCOUS FLUIDS USING SPECTROSCOPY

FIELD OF THE INVENTION:

The present invention relates to an apparatus for online analysis of viscous fluids.
In particular, the invention relates to a sample dilution accessory fitted with the IR spectrometer, the said apparatus being capable of online IR analysis of diluted viscous samples in batch mode.

BACKGROUND OF THE INVENTION:

Online analysis of viscous fluids by NIR or IR spectroscopic technique has tremendous potential for determining several physico-chemical properties of such fluids in real time that are crucial for smooth operation and profit maximization.
A major problem in evaluating viscous fluids through online analysis apparatus of the state of art is their high viscosity because of which it becomes difficult to make them flow through the online sample cell for accurate analysis. Another major difficulty, particularly in the mid IR analysis, is the complete absorption of IR radiation in the region 2800 to 3100 cm^{-1} and 1100 to 1400 cm^{-1}. Due to complete absorption no useful information can be retrieved from these regions (**Figure 1**).

Existing Knowledge:

US6662116 B2 teaches to determine the properties of unknown fluids by IR analysis. However, this patent does not teach how to analyze the crude oil online.
US3996785 describes measurement of boiling point properties of crude by help of IR analysis, kinematic viscosity measurement and S analysis. This patent uses data from multiple sources to predict the boiling point properties of crude oil.

US5656810 describes composition evaluation of oil samples by comparison of spectral differences in luminescence, excitation, light scattering and absorption spectra in the near UV, visible and near IR regions.

US6490027 B1 teaches how to practice the automatic analysis of crude oil using near IR spectroscopy.

US4045671 discloses a method and an apparatus for continuously monitoring the presence of oil in water

WO2008135411A1 describes IR analysis method in batch mode using ATR technique. However, it does not indicate any dilution of crude oil and any corresponding online system for that.

US6087662 discloses an online process for determining asphaltene concentration in an asphaltene-containing hydrocarbon mixture using infrared spectroscopy and a multiple variable regression analysis. The Asphaltene content is predicted by Mid IR analysis using NaCl plates and Tunnel ATR cell in batch manner. Employing this method to an online and continuous system is seemingly non-feasible.

JP58037543A discloses a method for performing quantitative analysis by an infrared spectrophotometry to measure the amount of a curing agent in an epoxy resin using 0.05mm KBr cell and diluting the sample with chloroform (CHCl3).

None of the aforesaid patent or any other state of art discloses an apparatus employing mid IR spectroscopy as an online tool for accurately predicting various physico-chemical properties of viscous fluids.

Infact, it could not be practiced due to several practical difficulties such as viscosity of the fluid sample due to which the fluid needs IR sample cell with significant widths through which it could flow. But if the path length is increased, the sample becomes opaque to IR radiation. Previous patent data shows that some of the regions are completely opaque to IR radiation therefore no useful information could be retrieved from these regions. Another important difficulty is to clean the sample cell such that the results are representative to the actual sample passing through the cell at that particular instance.

In view of foregoing, there is a need for an improved apparatus to overcome these aforesaid problems in order to perform an accurate online IR analysis of viscous fluids.

OBJECTS OF THE INVENTION:

Accordingly, the objects of the present invention are as follows:

An object of present invention is to provide an apparatus comprising sample dilution accessory attached to an IR spectrometer for performing accurate online IR analysis of various viscous fluids in batch mode for predicting their physico-chemical properties.

Another object of the invention is to provide an apparatus for online IR analysis of viscous fluids, wherein an accessory is provided for diluting viscous fluid with an inert diluent.

Another object of the invention is to provide an apparatus for online IR analysis of viscous fluids, wherein the sample dilution accessory comprises flow control devices for controlling the flow of viscous sample and diluent into the mixing vessel.

Another object of the present invention is to reduce the viscosity of the sample in order to allow its better flow through the online IR sample cell of IR spectrometer.

Another object of the invention is to perform IR analysis of various viscous fluids using smaller IR sample cell than a typical IR sample cell, preferably the sample cell having a path length of 0.05mm.

Still another object of the invention is to utilize full IR spectrum from 4000 cm^{-1} to 400 cm^{-1} for predicting the properties of viscous fluid sample under examination by adding an inert diluent, preferably Carbon tetrachloride.

Still another object of the invention is to perform contamination free analysis by making the IR sample cell free of any sample residue with little cleaning effort.

Yet another object of the invention is to provide such apparatus which utilizes an inert diluent, halogenated C_1 to C_3 hydrocarbons e.g. Carbon tetrachloride, 1,2-dibromo-1,1,2,2-tetrachloroethane ($C_2Br_2Cl_4$) and Hexachloroethane (C_2Cl_6) , as cleaning solution for IR sample cell in order to quickly clean the sample cells to prepare them for next sample batch.

SUMMARY OF THE INVENTION:

In accordance with the present invention, there is provided an apparatus comprising sample dilution accessory attached to an IR spectrometer for performing accurate online IR analysis of various viscous fluids in batch mode for predicting their physico-chemical properties.

The said apparatus comprising an accessory for diluting the fluid sample, said accessory adapted to be fitted to an infrared spectrometer for analyzing the sample, wherein said accessory includes means to withdraw a sample from a stream at a controlled rates; means to dilute the withdrawn sample with a diluent; means to mix the diluent with the withdrawn sample in a homogenous manner, and means to introduce the diluted sample to IR absorption cells of the infrared spectrometer for analysis.

The viscosity of said diluted sample, obtained by diluting the said viscous sample with said diluent, is significantly reduced to make it easily flow through the online sample cell of significantly reduced path length. Further, the infrared absorbency of said sample is significantly reduced at a predetermined infrared wavelength(s)

The apparatus in accordance with the present invention is a sample dilution accessory fitted with an IR spectrometer, which is capable of online IR analysis of diluted sample in batch mode.

After the online analysis the sample cell can be flushed with pure halogenated C_1 to C_3 hydrocarbons e.g. Carbon tetrachloride, 1,2-dibromo-1,1,2,2-tetrachloroethane ($C_2Br_2Cl_4$) and Hexachloroethane (C_2Cl_6) so that the next diluted sample can be analyzed for contamination free analysis.

The apparatus in accordance with the invention is capable of providing accurate analysis of variety of viscous fluids. The diluent is used to dilute the viscous sample in accordance with the present invention in order to reduce the viscosity of said sample, allowing it to easily pass through the sample cell of the online IR analyzer. In accordance with the invention, the diluent is selected from group of inert IR transparent diluents, halogenated C_1 to C_3 hydrocarbons e.g. Carbon tetrachloride, 1,2-dibromo-1,1,2,2-tetrachloroethane ($C_2Br_2Cl_4$) and Hexachloroethane (C_2Cl_6)

The intensity of strong absorption bands of 2800–3200 cm^{-1} and 1100–1500cm^{-1} is significantly reduced due to dilution of the viscous fluid therefore these peaks can also be used in the chemometric analysis which was not possible for pure samples without any dilution.

In accordance with the invention, the viscosity of homogenous solution obtained after diluting the viscous sample with diluent in the sample dilution apparatus of the invention, is significantly lower than pure viscous fluid. This reduced viscosity, therefore makes the flow of sample through the online sample cell much easier as compared to the pure fluid. Another advantage with the apparatus of the invention is that the cleaning of the cell is better therefore the analysis is more accurate and contamination of online IR cell with previously analyzed sample is minimized.

BRIEF DESCRIPTION OF DRAWINGS:

The invention will now be described with reference to accompanying drawing.

Figure 1: shows that the region 2800 to 3200cm^{-1} (C–H stretching vibrations) and 1100 to 1500 cm^{-1} is completely opaque to IR radiation and no information can be retrieved from these regions for as such crude samples (US6662116 B2).

Figure 2: shows that Carbon tetra chloride (CCl_4) is almost transparent to IR radiation except 750 to 800 cm^{-1} region.

Figure 3: shows sample dilution accessory fitted with the IR spectrometer.

DETAILED DESCRIPTION OF THE INVENTION:

The present invention provides an improved apparatus comprising sample dilution accessory attached to an IR spectrometer for performing accurate online IR analysis of various viscous fluids in batch mode for predicting their physico-chemical properties.

The said apparatus comprising in combination:

a means (m-1) for directing a portion of viscous sample from the viscous fluid stream to a flow control device (F-1) and/or directing the said portion of viscous sample back to the said stream.

a storage vessel for storing the diluent

a Flow control device (F-1) for withdrawing a viscous sample from the means (m-1) at a controlled rate and transferring the said viscous sample at a controlled rate to heating and mixing vessel

a Flow control device (F-2) for directing a diluent from a storage vessel at a controlled rate and transferring the diluent at a controlled rate to heating and mixing vessel

a heating and mixing vessel for diluting a predetermined portion of viscous sample by mixing the said sample with a predetermined proportion of a diluent to form a homogenous solution, comprising a means for combining streams of said diluent and said sample in said predetermined proportions, and means for heating and mixing the said combined streams to form a homogenous solution without producing aggregates and/or precipitates of diluent insoluble components

an infrared spectrometer for analyzing the viscous fluids

a means (m-2) for allowing the said homogenous solution or a portion thereof to the past IR absorption cells of an infrared spectrometer

a collection vessel for collecting the diluted sample after analysis

a means (m-3) for flushing the online IR sample cells with pure diluent for preparing the sample cells for next contamination free analysis

wherein the viscosity of said homogenous solution, obtained by diluting the said viscous sample with said diluent, is significantly reduced to make it easily flow through the online sample cell of significantly reduced path length

wherein the infrared absorbency of said homogenous solution is significantly reduced at a predetermined infrared wavelength(s)

The apparatus in accordance with the present invention is a sample dilution accessory fitted with the IR spectrometer, which is capable of online IR analysis of diluted viscous samples in batch mode.

The intensity of strong absorption bands of 2800–3200 cm^{-1} and 1100–1500cm^{-1} is significantly reduced due to dilution of the sample therefore these peaks can also be used in the chemometric analysis which was not possible for pure samples of such fluids.

Typically IR sample cells have a path length in the range of 0.2mm - 0.5 mm. Even at the lower limit of path length i.e. 0.2mm, two regions i.e. 2800-3100 cm^{-1} and 1100-1400 cm^{-1}) show 100% absorption of IR radiation (US patent 6662116 B2), due to which, no information can be retrieved from these regions for pure fluid samples. There is no state of art apparatus available, wherein, an online sample cell of 0.05 mm path length is used.

Further, IR sample cells with smaller path length (<0.2 mm) could not be used due to following two reasons:

(a) There is a difficulty in passing the viscous fluid through the IR sample cell.

(b) Efficient cleaning of the sample cell for accurate analysis becomes a problem.

IR cells with larger path length, although could solve the problems associated with flow of viscous fluid and cleaning of IR sample cell, but the problem of 100% absorption of IR radiation still persists.

This difficulty is addressed in accordance with this invention by dissolving viscous fluid in an inert IR transparent diluent which reduces its viscosity. Viscous fluids have

a good solubility in Carbon tetra chloride (particularly crude oil has 10 wt% solubility).

The IR spectrum of Carbon tetra chloride (CCl_4) as presented in **Figure 2** having wave number on X-axis and % Transmittance on Y-axis, clearly shows that the carbon tetrachloride is almost transparent to IR radiation except at 750 cm^{-1} to 800 cm^{-1} region.

Other than Carbon tetrachloride, solvents selected from the group consisting of halogenated C1 to C3 hydrocarbons including 1,2-dibromo-1,1,2,2-tetrachloroethane ($C_2Br_2Cl_4$) and Hexachloroethane (C_2Cl_6) can also be used. Similar to carbon tetrachloride solution, these two solvents 1,2-dibromo-1,1,2,2-tetrachloroethane and Hexachloroethane are also transparent to the region of interest of IR radiation except at 600 cm^{-1} to 800 cm^{-1}.

Viscous fluids diluted in halogenated C_1 to C_3 hydrocarbons e.g. Carbon tetrachloride, 1,2-dibromo-1,1,2,2-tetrachloroethane ($C_2Br_2Cl_4$) and Hexachloroethane (C_2Cl_6) have considerably lower viscosity and it can smoothly flow through the online IR sample cell.

Furthermore, the absorption intensity is significantly lower for the diluted samples so more information can be retrieved from the 2800 to 3100 cm^{-1} (CH stretching) and 1100 to 1400 cm^{-1} (CH bending) region, which was never hitherto possible for pure samples of viscous fluids .

The present invention therefore provides a solution to this longstanding problem of analyzing viscous fluids by providing the following improvements:

In accordance with an aspect of the invention, the viscosity of diluted homogenous solution is significantly reduced therefore the flow of sample through the online cell is much easier as compared to the pure sample. As a result, sample cells of less than 0.2mm (e.g. 0.05mm) can be used.

In accordance with another aspect of the invention, the absorbance intensity of sample in regions 2800 cm^{-1} –3100 cm^{-1} and 1100 cm^{-1} –1400cm^{-1} is reduced considerably therefore full IR spectrum from 4000 cm^{-1} to 400 cm^{-1} is used for predicting the properties of sample under examination.

The reduction in absorbance with dilution using the apparatus in accordance with this invention is tabulated in Table No. 1 and Table No. 2.

Table 1:

	0.05 KBr Cell	% Absorbance			
	% Dilution	~2950cm^{-1}	% Reduction in absorbance	~1450cm^{-1}	% Reduction in absorbance
Neat crude	0.0	100	-	97	-
1mL Crude + 1mL CCl4	50.0	94	6	52	46
1mL Crude + 5mL CCl4	83.3	73	27	26	73
1mL Crude + 10mL CCl4	90.9	46	54	13	87

Table 2:

	ATR Cell	% Absorbance			
	% Dilution	~2950cm^{-1}	% Reduction in absorbance	~1450cm^{-1}	% Reduction in absorbance
Neat crude	0.0	58	-	36	-
1mL Crude + 1mL CCl4	50.0	51	12	28	22
1mL Crude + 5mL CCl4	83.3	27	53	13	64
1mL Crude + 10mL CCl4	90.9	14	76	6	83

In accordance with another aspect of the invention, the cleaning of the cell is better therefore the analysis is more accurate and contamination of online IR cell with previously analyzed sample is minimized.

The further figure accompanying the invention is described below:

Figure 3 shows sample dilution accessory fitted with the IR spectrometer.

As shown in FIG. 3, the apparatus comprises a side stream (m-1) comprising a two way valve V-1 which can direct the viscous fluid to the Flow control device (F-1) and /or back to the viscous fluid line, a flow control device (F-1) for withdrawing a viscous fluid from m-1 at a controlled rate and transferring the said viscous fluid at a controlled rate to heating and mixing vessel, a storage vessel for storing the diluent, a Flow control device (F-2) having a valve (V-2) for directing a diluent from a storage vessel at a controlled rate and transferring the diluent at a controlled rate to heating and mixing vessel, heating and mixing vessel for mixing viscous fluid with diluent and heating to form homogenous solution, an IR spectrometer for analyzing the viscous fluid and a waste collection vessel for collecting the homogenous solution after being passed through online sample cells of infrared spectrometer.

Example 1: Analysis of Crude Oil (Viscous Fluid):

To operate the apparatus (FIG. 3), first, a crude oil in the crude line is withdrawn by the side stream and fed to the Flow control device (F-1). Simultaneously, the diluent, carbon tetrachloride is withdrawn from the storage vessel and fed to the Flow control device (F-2). Both crude oil sample and carbon tetrachloride are transferred at a controlled rate to the heating and mixing vessel where the crude oil sample is diluted in the range of 50-99% with carbon tetrachloride and heated to form a homogenous solution preventing the formation of any insoluble precipitates or aggregates. The homogenous solution formed or a portion thereof is allowed to flow through the IR absorption cells of an infrared spectrometer and absorption is evaluated, after which

the solution is collected in the waste collection vessel. Thereafter the sample cells are flushed with pure carbon tetrachloride for preparing the sample cells for next contamination free analysis and analysis of next batch of sample is conducted in the similar manner.

The actual online IR sample cells have a typical path length of 0.2 to 0.5 mm therefore the absorbance of these two bands (2800 – 3100 cm^{-1} & 1100 - 1500 cm^{-1}) will be always 100% for pure crude. Whereas for diluted crude, there is a significant decrease in the absorption intensity for the two bands 2800 – 3100 cm^{-1} & 1100 - 1500 cm^{-1} in 0.05 mm KBr cell. The diluted crude solution prepared in accordance with the invention was examined under optical microscope to check if there is sedimentation or precipitation of CCl_4 insoluble components. The optical microscope images showed no aggregates and/or precipitates with 50 to 91% dilution of crude oil by CCl_4. Therefore the homogeneity of crude oil is maintained after dilution with CCl_4.

Technical advancement:

The apparatus in accordance with the invention has several advantages such as:

(a) Accurate analysis of various viscous fluids can be done for determining their several physico-chemical properties in real time.

(b) Viscosity of the viscous fluids is significantly reduced in order to allow better flow through the IR sample cell.

(c) IR analysis of viscous fluids can be performed using smaller IR sample cell than a typical IR sample cell.

(d) By adding an inert diluent, particularly Carbon tetrachloride, full IR spectrum can be utilized from 4000 to 400 cm^{-1} for predicting the properties of viscous fluid sample under examination.

(e) Contamination free analysis can be performed by making the IR sample cell free of any residual sample with little cleaning effort.

(f) Halogenated C_1 to C_3 hydrocarbons e.g. Carbon tetrachloride, 1,2-dibromo-1,1,2,2-tetrachloroethane ($C_2Br_2Cl_4$) and Hexachloroethane (C_2Cl_6) can be used as a cleaning solution for IR sample cell in order to quickly clean the cell.

While considerable emphasis has been placed herein on the various components of the preferred embodiment, it will be appreciated that many alterations can be made and that many modifications can be made in the preferred embodiment without departing from the principles of the invention. These and other changes in the preferred embodiment as well as other embodiments of the invention will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the invention and not as a limitation.

Claims:

1. An apparatus for online analysis of a viscous fluid sample comprising an accessory for diluting the sample, said accessory adapted to be fitted to an infrared spectrometer for analyzing the sample.
2. An apparatus as claimed in claim 1 wherein said accessory includes means to withdraw a sample from a stream at a controlled rate; means to mix a diluent with the withdrawn sample in a homogenous manner to form a dilute sample, and means to introduce the diluted sample to IR absorption cells of the infrared spectrometer for analysis.
3. An apparatus as claimed in claim 1 wherein the means to withdraw a sample from a stream includes a flow control device for withdrawing said sample from said stream and transferring said withdrawn sample to a mixing vessel.
4. An apparatus as claimed in claim 3 which includes a storage vessel for storing the diluent.
5. An apparatus as claimed in claim 4 wherein the storage vessel includes means to feed diluent stored therein at a controlled rate to the mixing vessel.
6. An apparatus as claimed in claims 2 to 5 wherein the mixing vessel includes a heating device to heat the diluent and the withdrawn sample during the process of mixing.
7. An apparatus as claimed in claim 2 which includes a collection vessel for collecting the diluted sample after analysis.
8. An apparatus as claimed in claim 1 which includes a flushing means to flush out any remnant in the IR sample cells for carrying out the analysis of subsequent samples.
9. An apparatus according to claim 2 where the IR absorption cells are KBr cells
10. An apparatus as claimed in claim 2 wherein the IR spectrometer is adapted to measure absorption over the full IR spectrum of 4000 cm^{-1} to 400 cm^{-1}

11. An apparatus according to claim 2 wherein the path length of the IR absorption cells is in the range of 0.05 mm to 0.75 mm.

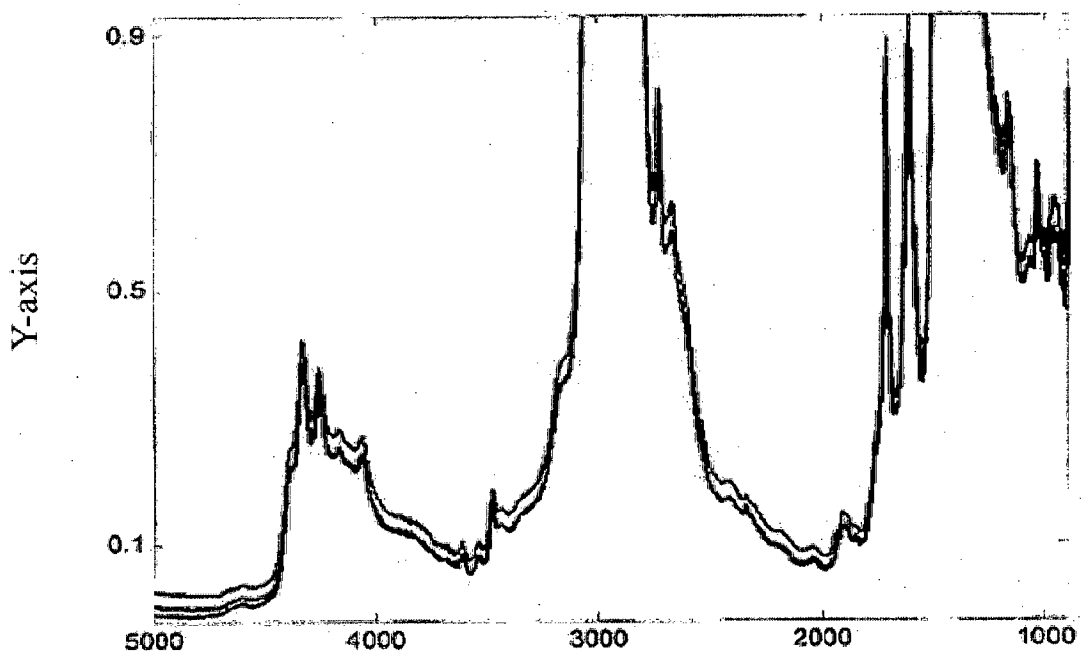


FIGURE 1

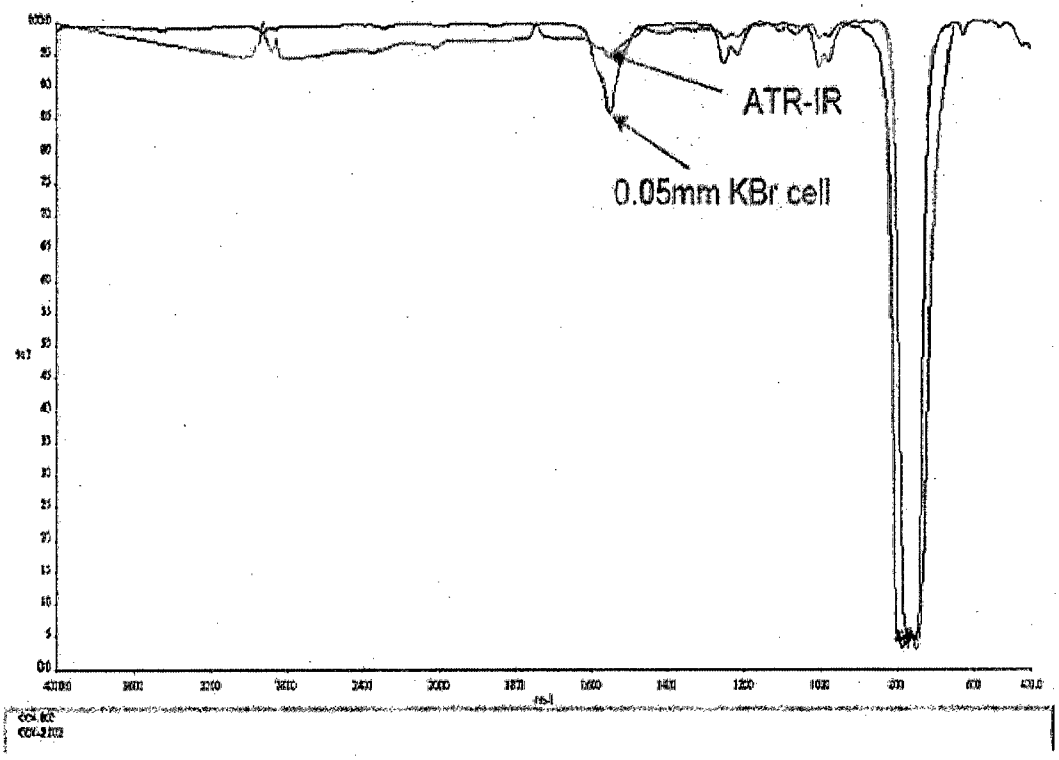


FIGURE 2

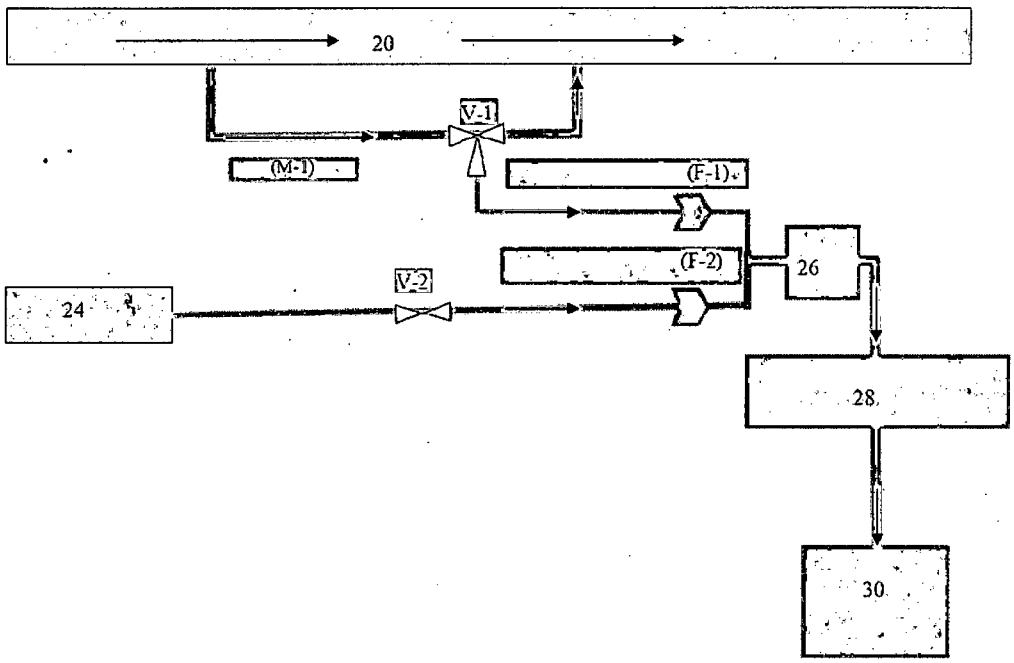


FIGURE 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IN2011/000224

A. CLASSIFICATION OF SUBJECT MATTER

G01N 21/35(2006.01)i

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: G01N 21/-

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI;EPODOC;CNPAT;CNKI;ISI web of Knowledge; CA: spectro+, online, analy+, crude, hydrocarbon, oil, dilute+

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CN101487818A (STATE GRID ELECTRIC POWER RES.), 22 Jul.2009(22.07.2009),claims 1-8	1-11
A	CN101846628A(SHANGHAI SK. PETROLEUM & CHEMICAL EQUIPMENT CORP. LTD.) 29 Sep.2010(29.09.2010),claims 1-8	1-11
A	US2005279131A1(CONOCO PHILLIPS CO. et al.), 22 Dec.2005(22.12.2005),claims 1-60	1-11

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
09 Oct.2011(09.10.2011)	**10 Nov. 2011 (10.11.2011)**
Name and mailing address of the ISA/CN The State Intellectual Property Office, the P.R.China 6 Xitucheng Rd., Jimen Bridge, Haidian District, Beijing, China 100088 Facsimile No. 86-10-62019451	Authorized officer YIN,Li Telephone No. (86-10)82245671

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
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Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
CN101487818A	22.07.2009	None	
CN101846628A	29.09.2010	CN101846628B	03.08.2011
US2005279131A1	22.12.2005	WO2006009621A2	26.01.2006
		US7213413B2	08.05.2007