

Determination of Asphaltenes in Petroleum Crude Oils by Fourier Transform Infrared Spectroscopy

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An analytical method was developed to quantitatively determine the asphaltene content in petroleum crude oils by Fourier transform infrared spectroscopy (FT-IR). Asphaltenes are a solubility class of compounds found in crude oils. They are black to dark brown solids defined by their insolubility in *n*-alkane solvents. The structure of asphaltene molecules is polynuclear aromatic rings with alkyl side chains and heteroatoms such as nitrogen, oxygen, and sulfur attached. Asphaltenes are known to cause oil well plugging and irreversible catalyst deactivation in refineries. The asphaltene content of 50 crude oils from a wide array of geochemical conditions was determined by the standard *n*-pentane insolubles method. FT-IR spectra of the 50 crude oils were collected using an attenuated total reflectance cell. A partial least squares model was generated to predict the amount of asphaltenes from 42 of the crude oils. The model was shown to have an R^2 value of 0.95 and a standard error of estimate of 0.92 wt %. An independent prediction set of eight crude oils was used to test the validity of the model. The prediction set was shown to have an R^2 value of 0.96 and a standard error of prediction of 0.99 wt %. The FT-IR method compares favorably with the current laboratory method in terms of results, is faster, and uses no solvents.

Introduction

The petroleum industry is faced with having to refine much heavier crudes in the coming decades. Production of lighter crudes is peaking in non Middle East areas.¹ Dealing with the asphaltene fraction in heavier crude oils has continually posed a problem to petroleum producers and refiners. During production, unwanted asphaltene precipitation has been responsible for the plugging of wells—stopping the flow of oil.² Dickkian and Seay have shown asphaltene precipitation and its carbonization to be the major cause of refinery heat-exchanger fouling.³ This unwanted carbonization costs the refiner money by lowering the heat transfer coefficients of the exchangers affecting output and efficiency. Also, asphaltenes react with fluid catalytic-cracking catalysts causing a large deposit of coke on the surface of the catalyst.⁴ This deposition can cause a major monetary loss for the refiner by forcing the replacement of inefficient catalysts. The metals found in the asphaltene fraction can also cause irreversible deactivation

of expensive catalysts by binding with active sites.⁵ In today's world of environmental consciousness, the sulfur found in the crude oil asphaltene residual is unwanted in highly valued products such as gasoline and diesel fuel.² Since the formation of petroleum crude oil is dependent on a number of factors, the amount and structure of the asphaltenes can differ among crudes of different sources. Several characteristics are common among all petroleum asphaltenes, with the prevailing structures being polynuclear aromatic rings with alkyl side chains. Heteroatoms, such as sulfur, nitrogen, and oxygen, are also included in the asphaltene fraction.⁴ Several analytical techniques have been employed to characterize the asphaltene structure, including thermogravimetric analysis,⁶ gel permeation chromatography,^{7,8} NMR,^{9–12} electron–nuclear double resonance spectroscopy (ENDOR),¹³ UV spectroscopy,¹⁴ X-ray analy-

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Table 1. Crude Oil Descriptions and Sample Codes

sample code	crude description	sample code	crude description
ABL	Arabian Light	LMS	Louisiana-Mississippi Sweet
B22	B. C. F. 21.9	LSB	Light Sour Blend
BNM	Bonny Medium	MCS	Mid-Contentient
BOS	Bonito Sour	MED	Meditanos
BOW	Bow River	MER	Merey
BRN	Brent	MES	Mesa 30
CRB	Caripito Blend	MSO	Mixed Blend Sour
CRP	Caripito Pedernales	MSW	Mixed Blend Sweet
CTX	Cetex Stream	MYA	Maya
CUS	Cusiana	NDL	North Dakota Light
EIC	Eugene Island	NLS	North Louisiana Sweet
FLT	Flotta	ODU	Odudu
FOT	Forties – North Sea	OKW	Okwori South #1: Green Sand
GUA	Guafita	OLM	Olmeca
HND	Hainesville-Neuhoff Dome	PGE	Penn Grade (Eureka)
KUW	Kuwait Export Blend	PGP	Penn Grade (Peake Energy)
L24	Leona 24	STF	Statfjord
LLB	Lloyd Blend	TRL	Troll (North Sea)
LLD	Lloydminster	WKI	Weeks Island Sour
LLK	Lloydminster (Kerrobort)	WTS	West Texas Sour
LLS	Light Louisiana Sweet	WYF	Wytch Farm

sis,⁹ and Fourier transform infrared spectroscopy (FT-IR).^{6,7,10,14,15} Depending on the method of characterization, molecular weights have been reported from 2,000 to 300,000 amu. In a recent study conducted using analytical data and molecular modeling software, the molecular weight of a Venezuelan asphaltene was determined to be approximately 9,500, with a molecular formula of $C_{637}H_{745}O_{10}N_{12}S_{20}$.¹⁶ Recently, the structure of three asphaltenes obtained from bitumen processed at different Indian refineries was determined using X-ray diffractometry and NMR analysis.¹⁷

Numerous Fourier transform infrared spectroscopy studies have been conducted to study the composition of asphaltenes. Yen has summarized the mid-IR work conducted during the 1960s to determine the functional groups in asphaltenes.¹⁸ This work centered on the methyl ratio present, the paraffinic and naphthenic carbons present, and the alkyl side chain length. FT-IR was also used to determine the type and relative amounts of functional groups present in asphaltenes of different origins.¹⁰ The technique was utilized in conjunction with several other analytical methods by Michels et al. to study the artificial maturation of asphaltenes.¹⁴ Mahlhotra and Buckmaster have studied chromatographically separated Boscan asphaltene fractions and determined the functional groups present by analyzing the absorption bands in the FT-IR spectra. In a recent study, FT-IR spectroscopy and partial least-squares analysis were used to quantitatively determine the physical and chemical properties of base oils.¹⁹

Other types of spectroscopy have been used in the determination of asphaltenes in petroleum. Zerlia and Pinelli utilized ultraviolet spectroscopy to predict the amount of asphaltenes in heavy petroleum products employing the partial least-squares (PLS) algorithm.²⁰ The results were comparable to the standard solvent determination method. To determine the flocculation threshold of petroleum asphaltenes in solvents, Lambert used near-IR analysis.²¹ The flocculation threshold was defined as the minimum quantity of an *n*-alkane that must be added to a specific quantity of petroleum to cause the asphaltene to precipitate. Lambert also described a technique in the near-IR to determine the asphaltene content in crude oil using absorbances from a series of wavelengths in the combination bands.²²

The spectroscopic method presented here allows determination of the asphaltene content of a crude oil more quickly than the current solvent methods. The current methods can take several hours to perform and use large quantities of solvents. The method described in this study could be applied to other refinery streams containing asphaltenes including residuals and crude unit vacuum bottoms.

Experimental Section

Sample Set. Fifty raw crude oil samples were collected for use in the asphaltene calibration and prediction sets. The oils had origins from a wide array of geochemical conditions from around the world. The samples were stored in 1 L glass bottles. Descriptions of crude oil types and the sample codes can be found in Table 1.

Vacuum distillations had previously been performed to produce different boiling-range fractions of each crude. The heavy-end boiling fractions from the crudes with boiling points >900 °F (~482 °C) were saved in small round metal cups and covered with aluminum foil. The heavy fractions from three oils were used in an experiment to determine the infrared absorbance bands for the asphaltenes.

The asphaltene content of the 50 crude oils and their heavy-boiling fractions were determined using a modified ASTM

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method D-4055. The standard method was followed except 200 mL of *n*-pentane was added to 5 g of sample, and the oil/pentane mixture was sonicated for 30 min and then filtered through a 1.5 μm filter. The deasphalted oil fractions from five selected crudes were recovered using a Buchi model 110 Rotovap.

FT-IR Instrumentation. Spectra of the 50 crude oils were collected on an Analect Diamond-20 spectrometer with a spectral range from 7000 to 400 cm^{-1} . Only the mid-IR spectral range from 4000 to 650 cm^{-1} was used in this study. Spectra were an accumulation of 128 scans with a spectral resolution of 4 cm^{-1} . The time to acquire all 128 scans was approximately 150 s. The infrared radiation was measured using a deuterated triglycine sulfate (DTGS) detector. The data system employed to control the instrument consisted of a Dell 486DX CPU with an arithmetic coprocessor and the instrument manufacturer's data collection software.

Two types of infrared sample cells were used in the experiment including NaCl plates (International Crystal Laboratories) and an ATR Tunnel cell from Axiom Analytical. The Tunnel cell contained a zinc selenide element with a diameter of 3.2 mm and a length of 40 mm. The number of effective internal reflections through the prism was 10 with a total flow volume of 40 μL .

Asphaltene Absorption Band Determinations. Five crudes having differing amounts of asphaltenes were selected to determine infrared absorption bands for the asphaltenes that were common to all samples. Crude oils selected included a Eugene Island, a Maya, a Statfjord, a Mesa 30, and a Cetex. Once the asphaltene content for each was determined, the asphaltene fraction and the deasphalted oil fraction for each crude were retained for FT-IR analysis. Heavy-end boiling fractions from three crudes (Eugene Island, Statfjord, and Cetex) were deasphalted to obtain enough asphaltene to analyze by FT-IR.

A small amount of the asphaltene was transferred from the filter paper to a 15 mL Pyrex beaker and dissolved with approximately 5 mL of 99.9% spectrophotometric-grade carbon tetrachloride (CCl_4) (Aldrich Chemical). Two clean 25 mm \times 2 mm NaCl salt plates (International Crystal Laboratories) were assembled in a holder and placed in the Analect Diamond-20 to obtain a background spectrum. After completion, the NaCl plates were disassembled and placed face up on a hot plate on low heat. A polyethylene transfer pipet was used to drop the asphaltene/ CCl_4 mixture on to the center of the heated NaCl plates, evaporating the CCl_4 and leaving the asphaltene. After a sufficient amount of asphaltene was deposited on the plates, they were put in a vacuum oven at 160 $^\circ\text{C}$ for 15 min. After cooling, the plates were reassembled and the FT-IR spectra acquired. Spectra of the deasphalted oil fraction were obtained in the same manner, except that heating the NaCl plates on the hot plate was not necessary because the oil was placed directly on the plates and no CCl_4 needed to be evaporated.

Spectra were also acquired on a 50% asphaltene/50% deasphalted oil mixture of each of the five selected crudes to help in verifying the asphaltene absorption bands. The samples were prepared by weighing 0.1 g of the asphaltene into a 15 mL beaker and then adding 0.1 g of the deasphalted oil. Five milliliters of CCl_4 was added to the beaker to fully dissolve the asphaltene. The sample was mixed thoroughly with a glass stirring rod and then deposited using a transfer pipet on heated salt plates to evaporate the CCl_4 . The plates were placed in the vacuum oven and then allowed to cool before FT-IR spectra were obtained.

FT-IR Crude Oil Analysis. Samples of the 50 raw crude oils were shaken vigorously and poured into 15 mL beakers. The crude oil was introduced into the ATR Tunnel cell installed on the Analect Diamond-20 instrument using 20 mL disposable syringes. Approximately 10 cm of poly(tetrafluoroethylene) tubing was used to connect the syringe port to the inlet of the

Tunnel cell, thus the sample compartment did not have to be opened to inject the crude oil sample. Poly(tetrafluoroethylene) tubing was also used to connect the outlet of the cell to a 1000 mL Erlenmeyer flask used as a waste collection reservoir. The cell was cleaned between oil samples with 50 mL of toluene followed by 50 mL of *n*-pentane. The pentane was evaporated from the cell by purging for 5 min with air. The technique was repeated for all 50 crude oil samples.

All collected spectra were imported into Galactics' Grams 386 version 3.03 software located on a Compaq Deskpro computer for multivariate regression analysis.

Results

The FT-IR asphaltene absorption bands were determined by analyzing the asphaltene fraction, the deasphalted oil (DAO), and a 50% asphaltene/50% DAO sample for each of 5 crude oils (MES, MYA, EIC, CTX, STF) used in the calibration set. The asphaltene content for the 5 oil samples ranged from 0.50 to 14.93 wt %. Each crude in this set was deasphalted 3 times to test the repeatability of the test method. The average asphaltene contents and the corresponding standard deviations for each crude were determined to be as follows: MES 3.34 ± 0.13 wt %, MYA 14.93 ± 0.42 wt %, EIC 1.61 ± 0.15 wt %, CTX 8.49 ± 0.21 wt %, and STF 0.50 ± 0.08 wt %. The repeatability limit of the ASTM method at 95% confidence limits is dependent upon the amount of asphaltene present in the oil and is defined by the following equation²³

$$\text{repeatability} = 0.177 \left(\frac{2}{3}x \right)$$

where x is the average asphaltene value. All samples except the STF crude are well within the repeatability limits. The repeatability of the STF crude is slightly greater than the repeatability limit of 0.06 wt %.

In Figure 1, a series of FT-IR spectra of the fractions from two crudes, CTX and EIC, are presented. There are absorbance bands in both asphaltene spectra (A) that are not present in the DAO spectra (C). These areas include a peak located at 1602 cm^{-1} , a shoulder centered near 1435 cm^{-1} , and peaks at 1306, 1032, 868, 812, 752, and 722 cm^{-1} . It is important to note the same peaks are observed for both crude oils even though the asphaltene concentrations of the two crudes were determined to be very different (1.6 and 8.5 wt %) and the oils were formed under different geological conditions. Spectra collected on the fractions of the remaining crudes were typical of the spectra presented in Figure 1. Spectra of all five asphaltenes are included in Supporting Information.

The functional groups present in the asphaltene and 50/50 mixture fractions were identified and assigned from the literature.^{6,7,10,14} The broad peak at 1602 cm^{-1} corresponds to the aromatic C=C stretching vibrations.⁷ The absorbances at 1459 and 1435 cm^{-1} were due mainly to CH_2 bending modes with some contribution from CH_3 bending modes.⁷ Methyl bending vibrations are responsible for the peak at 1377 cm^{-1} .⁷ The sharp peak located at 1306 cm^{-1} is due to the presence of ethers or esters.¹⁴ The peak near 1032 cm^{-1} can be assigned to ester linkages present in the asphaltene

(23) Ashland Petroleum Research, Applications, and Development Laboratory Methodology.

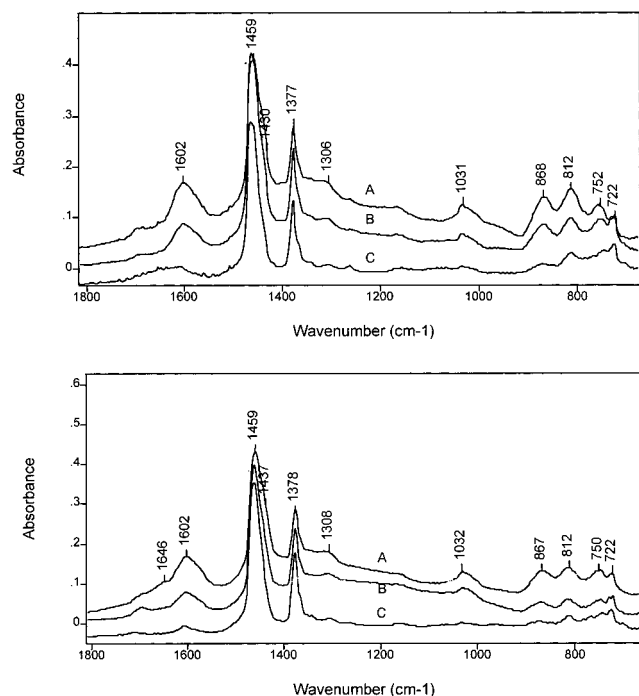


Figure 1. FT-IR spectra of the fractions of two crude oils from 1800 to 680 cm^{-1} with peak positions identified. Upper figure shows (A) 100% asphaltene, (B) 50% ASP/50% DAO, and (C) 100% DAO for the CTX crude. Lower figure shows the same fractions for the EIC crude.

molecule.⁷ The four absorbances observed between 870 and 722 cm^{-1} can be assigned to the aromatic CH out-of-plane bending vibrations.^{6,10} The number of hydrogen atoms adjacent to the CH bond causes four peaks to be observed.¹⁰

The spectra of five of the 50 whole crude oils analyzed using the Tunnel cell are presented in Figure 2. The five oils selected included the CRP, MYA, ABL, KUW, and CUS crude oils. The crude oils had a range of asphaltene concentration from 0.8 to 14.55 wt %. As can be seen from the spectra, some of the crudes contained large amounts of water, characterized by the large broad band at 3450 cm^{-1} . Absorbances found in the region from 1800 to 700 cm^{-1} of the crude oil samples show many of the same characteristics of the spectra of the fractions shown in Figure 1. The peaks include a shoulder at 1602 cm^{-1} and the CH_2 and CH_3 bending vibrations between 1500 and 1350 cm^{-1} . A peak is observed at 1306 cm^{-1} in the area of the bending vibrations of ethers or esters. The aromatic bending vibrations are also observed between 900 and 700 cm^{-1} .

Once the crude oil spectra were collected, the spectra were imported into the multivariate analysis software to regress against the asphaltene content determined by the *n*-pentane solvent method. The data set of crude oils was divided into a calibration set of 42 samples and a prediction set of eight crude oils. The prediction set was used as a set of unknowns to test the validity of the model generated. The crude oil spectra used in the calibration were mean centered in the PLS Plus regression package before the regression was carried out. "Leave one out cross validation" was used to test the model. In this validation, each spectrum in the calibration set was left out and then predicted from a model using all the other samples to test for concentration

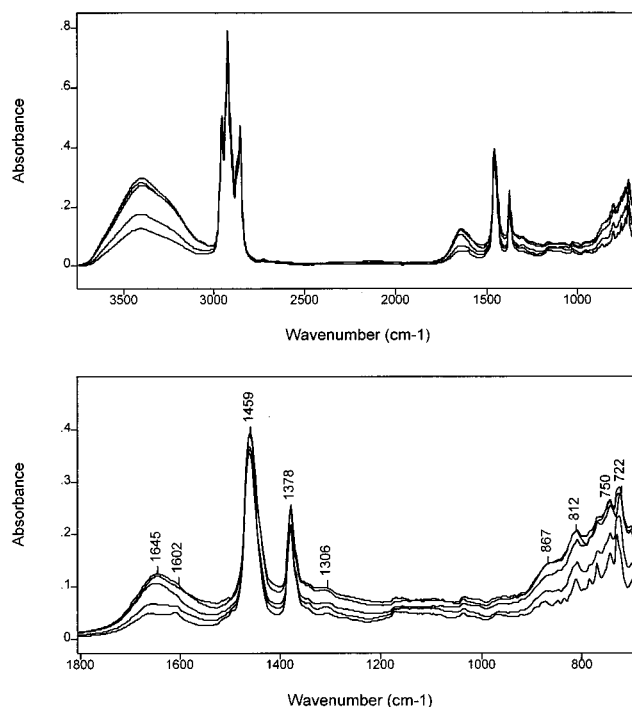


Figure 2. FT-IR spectra of five selected crude oils. Upper figure shows spectra from 3700 cm^{-1} to 680 cm^{-1} . Lower diagram includes the fingerprint region from 1400 to 680 cm^{-1} . Crudes represented include CRP, MYA, KUW, ABL, and CUS.

Table 2. PLS Parameters and Statistics for Determination of Asphaltenes in Crude Oil

model no.	wavelength regions used (cm^{-1})	no. of samples	factors used	R^2	SEE (wt % Asp.)
1	1615–1300, 1100–1000, 975–700	42	9	0.9495	0.92
2	4000–700	42	7	0.9036	1.29
3	900–700	42	6	0.9224	1.15

outliers. Several PLS models were developed using different spectral regions to determine if the absorbance bands identified for asphaltenes gave the best results. In Table 2, the results of the PLS modeling are summarized.

Table 2 shows model 1 is constructed from absorbance bands previously assigned to the asphaltene present in the crude oil. This model has a better R^2 and Standard error of estimate (SEE) than the other two models. Figure 3 is a plot of the FT-IR-predicted asphaltene value using model 1 versus the *n*-pentane solvent method. There is a linear relationship between the two results. From the residual plot it is apparent that the absolute error is constant across the entire range of asphaltene concentration. Model 2 was built using all wavelengths in the mid-IR region. This model is shown to have the worst SEE of the three. Model 3 was generated using only those bands assigned to the aromatic out-of-plane bending vibrations. It produced better results than model 2, but it was still inferior to model 1. This is proof that the oxygen functional groups are important in determining the amount of asphaltene present in the crude oil. The error is determined to be approximately ± 1 wt % asphaltene.

A second set of eight crude oils was used as a prediction set to test the validity of the calibration. The

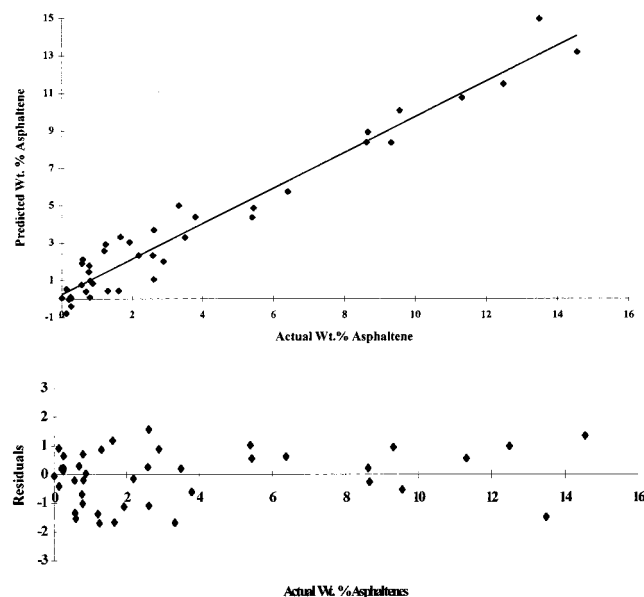


Figure 3. Predicted asphaltene vs actual asphaltene content for crude oil samples used in the calibration set. Lower diagram shows the absolute residuals for the sample set.

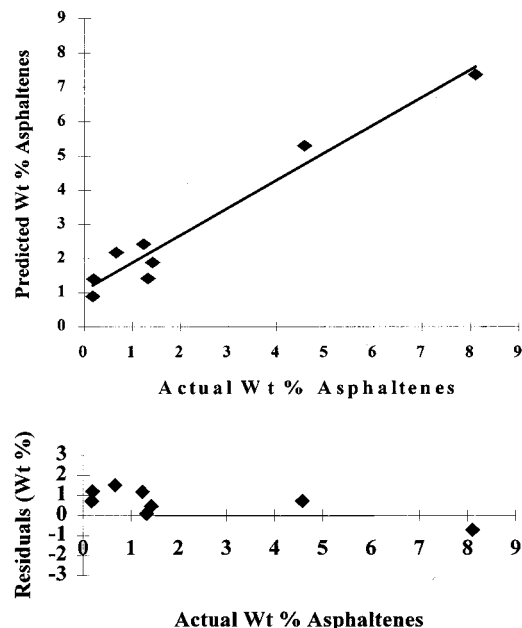


Figure 4. Predicted wt % asphaltene vs actual wt % asphaltene content for the prediction set of eight crude oils. Lower diagram shows the absolute residuals for each sample.

eight crudes (MSW, BOW, PGP, PGE, GUA, OLM, NDL, and NLS) were selected at random. The prediction plot of these eight crudes is presented in Figure 4. The R^2 value for the plot is 0.956 with a standard error of prediction (SEP) of 0.99 wt %. Although a linear relationship exists between the predicted and actual wt % asphaltene prediction, the residual plot shows a slight apparent bias in the predictions of the crudes containing lower amounts of asphaltenes. There could be several reasons for this bias. First, two of the samples containing lower amounts of asphaltenes (PGP, PGE) are

Pennsylvania-grade crude oils. Because this type of crude was not present in the calibration model, predicting this type of crude may lead to the observed bias. Second, the eight samples were analyzed at a later date, which could have led to slight changes in the instrument. The residual results are still comparable to the 1 wt % residuals observed for the calibration set residuals. Third, the validation set is small. The predicted values are still within the results from the calibration set for samples at lower asphaltene concentrations. With only eight samples it is possible that random selection of samples might lead to an apparent positive bias for lower asphaltene content. Fourth, errors could have occurred in the determination of the asphaltene content by the standard method, which resulted in a loss of precipitated asphaltene. These errors would be most pronounced in low asphaltic oils.

Conclusions

Asphaltenes are defined as a solubility class of compounds found in petroleum crude oils. Upon addition of an *n*-alkane solvent, the asphaltenes are precipitated from the crude oil. Asphaltenes are known to cause problems in both production and refining of crude oils by causing catalyst coking, catalyst poisoning, and plugging of oil wells. Production of heavier crude oils containing greater amounts of asphaltenes will increase in the coming decades. The current *n*-alkane solvent precipitation laboratory method takes up to 3 h to perform, uses large amounts of solvents, and has very poor reproducibility between laboratories. In this study, the asphaltene content of 50 crude oils was determined using an ASTM method and then analyzed on an FT-IR spectrophotometer equipped with an ATR cell. A PLS algorithm was employed to generate a calibration equation using 42 crude oil samples. The model had an R^2 value of 0.95 and an SEE of 0.92 wt %. A set of eight crudes was predicted as unknowns using the model, and an SEP of 0.99 wt % was observed. This study has shown it is possible to quantitatively determine the asphaltene content in crude oils by FT-IR spectroscopy using a PLS algorithm. The method can predict the amount of asphaltenes across a wide range of content and crude oil types. The method is much quicker with typical asphaltene determinations taking less than 10 min and uses no solvents. The FT-IR method could be applied to other laboratory samples containing asphaltenes, such as vacuum bottoms or heavy residuals.

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Supporting Information Available: Asphaltene spectra of crude oils and tables of statistical data for samples (13 pages). Ordering information is given on any current masthead page.

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