

Compositional Analysis of Bitumen and Bitumen-Derived Products

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Abstract

The American Society for Testing Materials (ASTM) procedures D2887 and D5307 are used for boiling range characterization of petroleum fractions up to 538°C (1000°F). The bitumens from the Uinta Basin (Utah) contain approximately 60.0 wt% material boiling above 538°C (1000°F); hence, the ASTM techniques leave major portions of the bitumens uncharacterized. A high-temperature chromatographic technique is developed to include a portion of the high-boiling-point compounds (greater than 538°C or 1000°F) in bitumen-derived heavy oils. A short 5-m glass capillary column is used to elute compounds with boiling points as high as 700°C (1292°F) (equivalent to C₉₀). Analyses of Uinta Basin (Utah) bitumens and bitumen-derived extract phases and residues are reported using the modified procedure. The characterization of the samples is extended by 30–35 wt% using the proposed simulated distillation method.

Introduction

Bitumens are ultraheavy oils with API gravity values less than 10°API and viscosity values greater than 10,000 cp at reservoir conditions. Whether the bitumens are produced by surface recovery (1,2) or by in-situ (3) processes, their characterization is important for the development of recovery process models and of kinetic and thermodynamic models of the processes employed to convert them to refinery feedstocks. Some of the processes used for upgrading bitumens and bitumen-derived heavy oils include coking (4,5), hydrotreatment (6), and supercritical fluid extraction (SFE) (7,8). Ryu (9) has extensively reviewed the literature on bitumen upgrading processes.

Boduszinski (10–13) reported a method for heavy oil characterization using both chromatographic and spectroscopic techniques and has suggested the use of the sequential elution fractionation (SEF) tech-

nique for extending the atmospheric equivalent boiling point (AEBP) beyond 704°C (1300°F) to as high as 1648°C (2998°F). Correlations (13) are available to estimate the mid-AEBP using the H:C atomic ratio or specific gravity of fractions obtained from SEF of hydrocarbons boiling above 704°C (1300°F). This permits complete characterization of petroleum crude oils and residual fractions. Bukka and co-workers (14,15) reported solubility class fractions of selected bitumens from the Uinta Basin of Utah. The fractions were separated using a sequence of solvents of increasing polarity. The separated solubility fractions were classified as saturates, aromatics I, aromatics II, resins I, resins II, and asphaltenes. The analyses of the fractions obtained from the Whiterocks, Asphalt Ridge, and Sunnyside bitumens led to the recommendation that the Whiterocks deposit be subjected to subsequent development studies based on the lower asphaltene content.

The boiling point distributions of oils can be determined using the ASTM D2892 (16) procedure. Distillation is carried out at a 5:1 reflux ratio on a column that contains 15 theoretical plates. Gas chromatographic simulated distillation (SIMDIS) techniques such as ASTM D2892 were developed to reduce the time required for boiling range analysis. The ASTM D2887 (17) simulated distillation procedure provided the means to determine boiling point distributions of oils containing components boiling below 538°C (1000°F). ASTM D5307-92 (18) was subsequently developed to account for the uneluted portion of the oil, based on the mathematical procedure originally proposed by

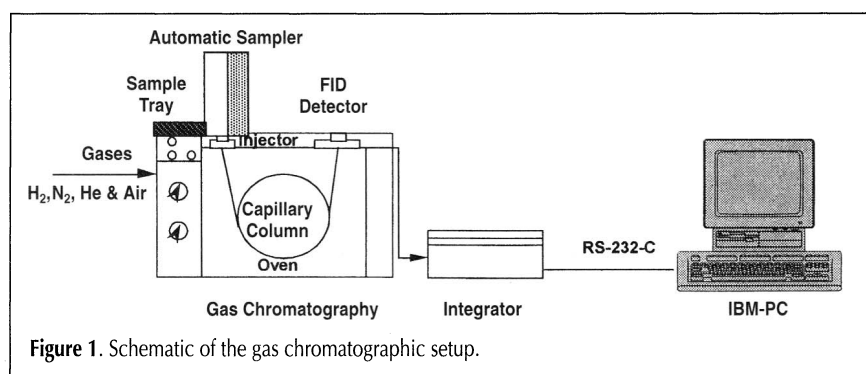


Figure 1. Schematic of the gas chromatographic setup.

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Worman and Green (19). Neer and Deo (20) established the mathematical equivalence between this procedure and the more intuitive lever arm rule to quantitate the uneluted fraction.

ASTM D2887 and D5307 make use of packed columns to determine the boiling point distributions of oils up to 538°C (1000°F). It should be possible to elute heavier fractions of oils and to obtain boiling point (or equivalent carbon number) distributions up to about 700°C (1292°F) using capillary columns with high phase ratios (approximately 500) or equivalent packed columns. The development of this technique would be particularly useful for analysis of heavy oils and bitumens that typically contain greater than 50 wt% material boiling above 538°C (1000°F).

Table I. Temperature Program for Simulated Distillation

| | Initial temp. (°C) | Initial time (min) | Ramp (°C/min) | Final temp. (°C) | Final time (min) |
|----------|--------------------|--------------------|---------------|------------------|------------------|
| Oven | 35 | 4.5 | 12.0 | 380 | 8.75 |
| Injector | 200 | 2.0 | 10.0 | 400 | 20.0 |

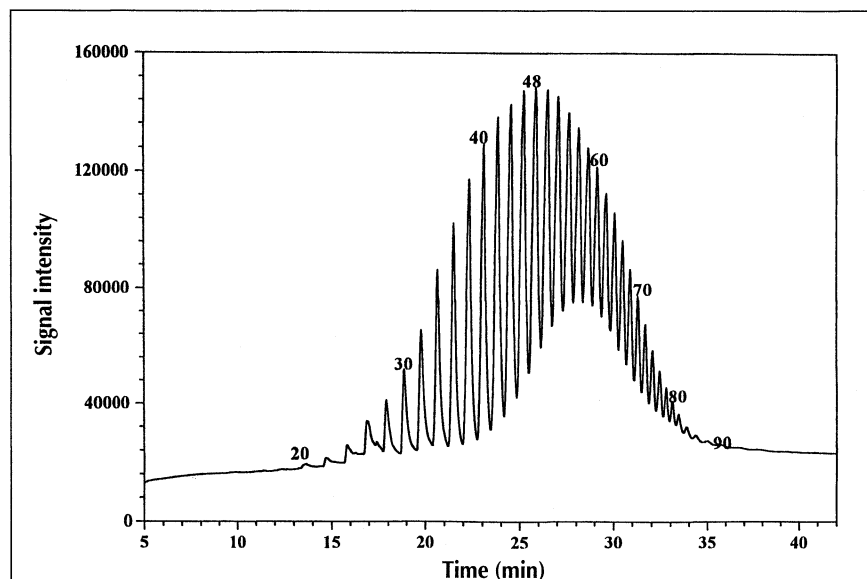


Figure 2. Chromatogram of calibration mixture Polywax 655 on a Petrocol EX 2887 column (Supelco). The carrier gas was helium at a flow rate of 20 cc/min. The initial temperature was 35°C. It was held for 4.5 min, increased at 12°C/min to 380°C, and held 8.75 min.

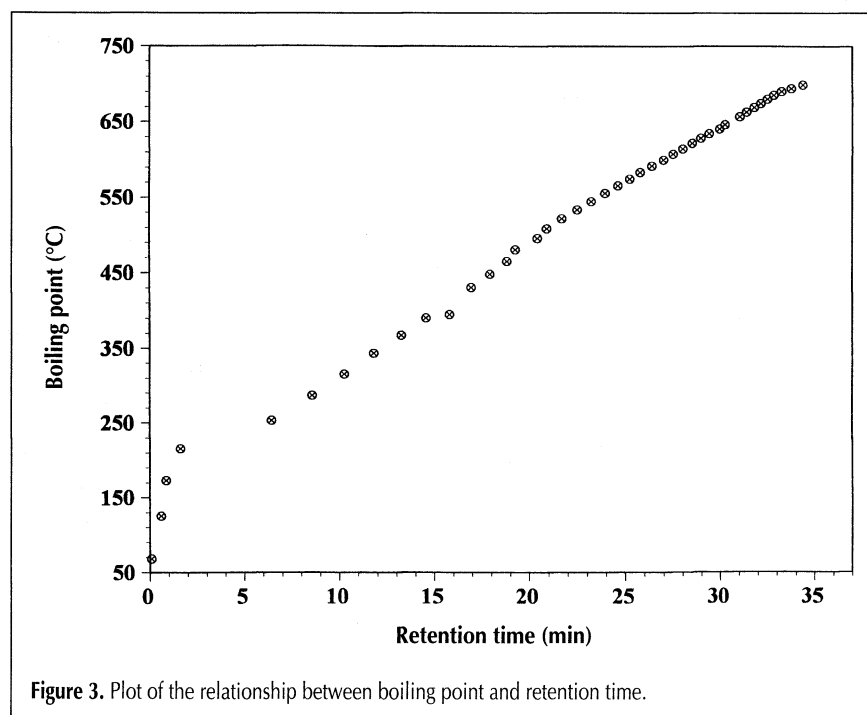


Figure 3. Plot of the relationship between boiling point and retention time.

The objective of this paper is to demonstrate the viability of using short, high-phase-ratio capillary columns for the characterization of ultraheavy oils and bitumens. The ASTM D5307 method has been extended to higher boiling point components and thus, to higher carbon numbers. The modified technique has been used to analyze bitumens from the Whiterocks, Asphalt Ridge, PR Spring, and Sunnyside oil sands deposits of the Uinta Basin (Utah). Four extract phases and four residues generated during SFE of the bitumens with propane (7,8) have been analyzed, and the boiling range distributions have been reported.

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Experimental

Gas chromatography

A Hewlett-Packard Model 5890 Series II gas chromatograph (GC) (Palo Alto, CA) was used to analyze the samples in this study. The chromatograph was equipped with on-column injection and a flame-ionization detector (FID). A schematic of the GC setup is presented in Figure 1. Helium was used as the carrier gas. Air, hydrogen, and nitrogen were used to sustain the FID flame. Injections were performed using the Model A 7673 automatic sampler from Hewlett-Packard. The signals from the detector were sent to an IBM-PC through a Hewlett-Packard 3396 Series II integrator using a Hewlett-Packard file server program. The signals were integrated using a slicing program at the integrator and were stored as report files for further computations. A Microsoft Windows based SIMDIS program was developed in-house to read the sliced and calibration data and to obtain the boiling point distributions for totally and partially eluted samples.

A Petrocol EX-2887 fused-silica capillary column (5 m × 0.53-mm i.d.; 0.1- μ m film thickness; phase ratio = 1325) from Supelco

(Bellefonte, PA) was used to characterize Whiterocks and PR Spring bitumens and the four extracts and residues. A Model Petrocol 2887 column from Supelco with a 0.5- μm film thickness (phase ratio = 265) was used to analyze the Asphalt Ridge and Sunnyside bitumens to achieve improved resolution because this column has a lower phase ratio. The recommended maximum operating temperature of the columns was 380°C (716°F).

The initial oven temperature was maintained at 35°C (95°F) for 4.5 min, increased at a constant rate of 12°C/min to a final oven temperature of 380°C (716°F), and held there for 8.75 min. The detector was maintained at 400°C (752°F) during the entire analysis. The temperature of the injector was initially

maintained at 200°C (392°F) for 2.0 min, increased at 10°C/min to a final temperature of 400°C (752°F), and held at 400°C (752°F) until the end of the oven program. The oven and injector temperature programs are reported in Table I. The carrier gas flow rates were maintained at 20 and 13 cc/min for the Petrocol EX-2887 and 2887 columns, respectively.

A 10- μL syringe from Hewlett-Packard and a nanoliter adapter kit were used in the automatic sampler to reduce the injection volume to 0.2 μL of sample. Polywax 655, a calibration mixture from Supelco, was used to calibrate the column. Polywax 655 is a blend of polyethylene oligomers with a carbon number range of C_{10} to C_{110} in even-number increments. The chromatogram of the Polywax 655 calibration standard for the

Petrocol EX-2887 column is presented in Figure 2. The peaks for carbon numbers C_{20} , C_{30} , C_{40} , C_{50} , C_{60} , C_{70} , C_{80} , and C_{90} were observed at approximately 13.5, 19.0, 23.5, 27.5, 29.5, 31.0, 33.0, and 35.5 min, respectively (Figure 2). The retention times for earlier peaks (with a carbon number less than 20) were obtained using different calibration mixtures from Supelco. Separate calibration runs using Polywax 655 were carried out with the Petrocol 2887 column used to analyze the Asphalt Ridge and Sunnyside bitumen samples.

The relationship between carbon number and retention time was obtained from the calibration mixtures. The boiling point of hydrocarbons (C_{10} to C_{90}) were obtained from TRC Thermodynamic Tables (21). The relationship between the boiling point and retention time is nearly linear and is presented in Figure 3. A Hewlett-Packard internal standard, which contained C_{14} , C_{15} , C_{16} , and C_{17} normal alkanes, was used in approximately a 1:10 weight ratio with the samples to permit calculation of the uneluted portion of the sample.

Table II. Typical Physical and Chemical Properties of Bitumen

| Properties | Source of bitumen | | | |
|--|-------------------|---------------|-----------|-----------|
| | Whiterocks | Asphalt Ridge | PR Spring | Sunnyside |
| Specific gravity (15°C/15°C) | 0.98 | 0.985 | 1.005 | 1.015 |
| API Gravity (°API) | 12.9 | 12.1 | 9.3 | 7.9 |
| Conradson carbon (wt%) | 9.5 | 13.9 | 14.17 | 15.0 |
| Pour point (°C) | 54 | 47 | 46 | 75 |
| Viscosity (Pa · s @ 70°C) | 4.8 | 5.0 | 47.0 | 173.0 |
| Asphaltenes* (wt%) | 2.9 | 6.8 | 19.3 | 23.6 |
| Saturates (wt%) | 35.7 | 39.2 | 33.4 | 20.0 |
| Aromatics (wt%) | 7.0 | 9.0 | 3.6 | 15.1 |
| Resins (wt%) | 54.5 | 44.1 | 43.8 | 36.8 |
| Molecular weight (g/gmol) | 653 | 426 | 670 | 593 |
| <i>Elemental analysis</i> [†] | | | | |
| C (wt%) | 87.0 | 86.9 | 87.0 | 86.8 |
| H (wt%) | 11.2 | 11.6 | 11.3 | 10.8 |
| N (wt%) | 1.4 | 1.7 | 1.3 | 1.1 |
| S (wt%) | 0.4 | 0.4 | 0.4 | 0.7 |
| H/C Atomic ratio | 1.56 | 1.60 | 1.56 | 1.49 |

* Pentane insolubles.
[†] C,H,N,S analyses normalized to 100%.

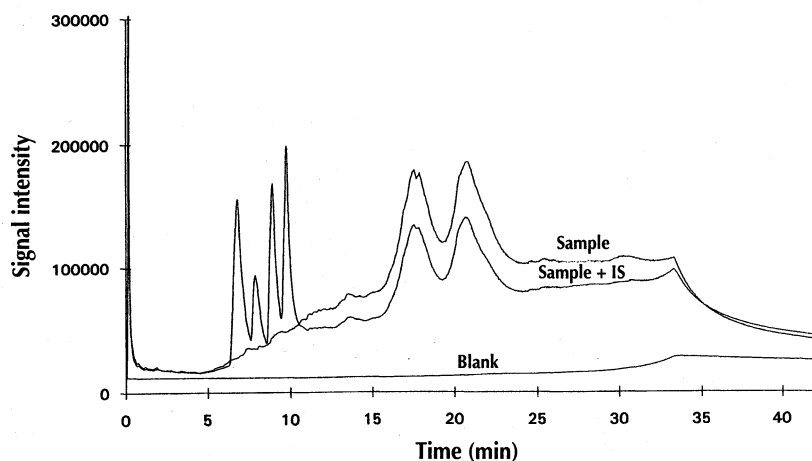


Figure 4. Chromatogram of Whiterocks bitumen using a Petrocol EX-2887 column (Supelco). The carrier gas was helium at 20 cc/min. The initial temperature was 35°C. It was held for 4.5 min then increased at 12°C/min to 380°C and held 8.75 min.

Samples

Four Uinta Basin bitumens from the Whiterocks, Asphalt Ridge, PR Spring, and Sunnyside deposits were analyzed in this study. The bitumens were extracted from the crushed oil sands ores in a conventional Dean-Stark extraction apparatus using toluene as the solvent. The bitumen-toluene solution was concentrated in a rotary evaporator, and the toluene was removed from the solution at 80°C (176°F). The physical and chemical properties of the bitumens are presented in Table II. The extract samples for all four bitumens were obtained by SFE using propane as the solvent at 5.6 MPa and 193°C (380°F). The residual samples were obtained by SFE of the four bitumens at 17.3 MPa and 193°C (380°F). The supercritical fluid ex-

traction procedure has been described in detail elsewhere (8). The objective of this work was to demonstrate the applicability of the chromatographic technique to materials lighter and heavier than bitumens. The extract samples were viscous light hydrocarbons, containing mainly saturates, aromatics, resins, and little or no asphaltenes. Asphaltenes were pentane insolubles obtained by a technique (22) prescribed by Syncrude Canada Ltd. Resins were measured using the technique described by Bukka and co-workers (14,15). The residual samples were black powders at ambient temperatures and consisted primarily of asphaltenes and resins and contained small amounts of saturates and aromatics. The residual samples were expected to contain 80 to 90 wt% of 538°C (1000°F) plus fractions. The bitumens and residual samples were diluted by adding 50–100 wt% of carbon disulfide to facilitate injection using the automatic sampler. The extent of dilution varied for

individual samples to permit easy flow into the syringe. The extract phase samples were injected without dilution. However, due to their viscous nature, the turret tray of the automatic sampler was maintained at 60°C (140°F) using a constant temperature water circulation bath. The automatic sampler viscosity parameter was maintained at 7 for syringe rinsing and injection.

Results and Discussion

Once a boiling point versus retention time calibration curve has been established for a given column at prescribed conditions, the following procedure permits calculation of the boiling point distribution of a given sample to the upper limit of the calibration curve. The curve used in this study (Figure 3) gave retention times of hydrocarbons boiling up to 700°C (1292°F). The uneluted portion of the sample, which in this case corresponds to species boiling above 700°C (1292°F), was calculated using chromatograms of the sample and chromatograms of the sample containing a known weight of the internal standard (typically 10 wt%). The baseline signal was subtracted from both the sample and sample containing the internal standard chromatograms before the uneluted fractions were calculated according to the following procedure (18).

The total area of the chromatogram was calculated from Equation 1:

$$A_T = [(I_A \times R) - I_B] \times \left[\frac{1 - W}{W} \right] \quad \text{Eq 1}$$

where A_T is the total area under the chromatogram, $R = (B - I_B) / (A - I_A)$; W is the weight of the internal standard divided by the sum of the weights of the sample and the internal standard (18); A is the area of the internal standard chromatogram up to 700°C (1292°F), B is the area of the sample chromatogram up to 700°C (1292°F); I_A is the area of the internal standard segment of the internal standard chromatogram; and I_B is the area of the internal standard segment of the sample chromatogram.

The weight fractions of different boiling point or carbon number segments were calculated using the total chromatogram area according to Equation 2:

$$W_n = \left[\frac{B_n - B_{n-1}}{A_T} \right] \quad \text{Eq 2}$$

where W_n is the weight fraction of the sample eluted between carbon numbers n and $n - 1$; and B is the area of the sample chromatogram up to a given carbon number.

The chromatogram of the Whiterocks bitumen is presented in Figure 4. Chromatograms of the sample plus the internal standard are also shown in Figure 4. The chromatograms indicate that even at high temperatures, negligible column bleed

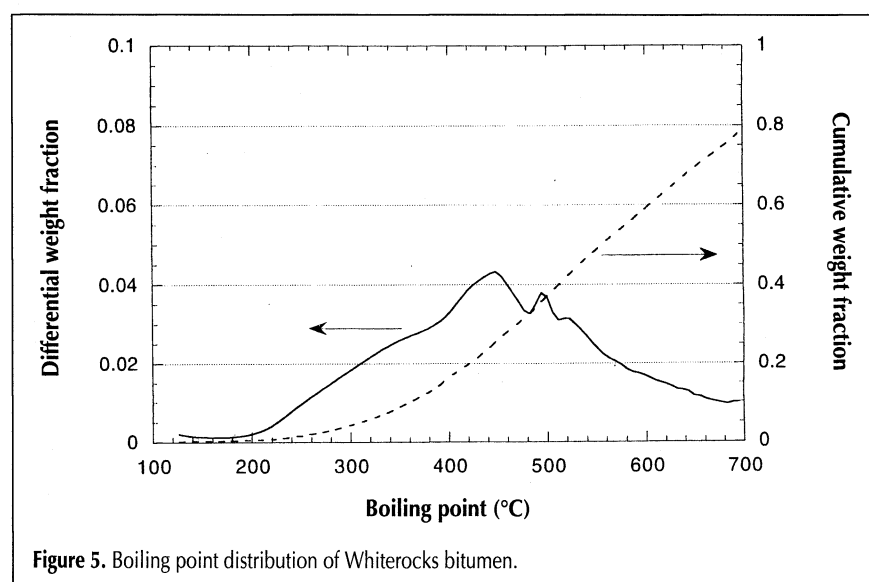


Figure 5. Boiling point distribution of Whiterocks bitumen.

| Table III. Comparison of the Extended Method Results | | |
|--|-----------------|------------|
| Samples | Weight fraction | |
| | 538°C plus | 700°C plus |
| <i>Bitumen</i> | | |
| Whiterocks | 0.53 | 0.20 |
| Asphalt Ridge | 0.45 | 0.11 |
| PR Spring | 0.54 | 0.21 |
| Sunnyside | 0.59 | 0.27 |
| <i>SFE extract</i> | | |
| Whiterocks | 0.08 | 0.0 |
| Asphalt Ridge | 0.16 | 0.0 |
| PR Spring | 0.12 | 0.0 |
| Sunnyside | 0.18 | 0.0 |
| <i>SFE residue</i> | | |
| Whiterocks | 0.88 | 0.58 |
| Asphalt Ridge | 0.87 | 0.61 |
| PR Spring | 0.86 | 0.59 |
| Sunnyside | 0.87 | 0.61 |

was observed. It should be noted that C_{90} , which has a boiling point of 700°C (1292°F), elutes at around 35.5 min. Thus, the procedure essentially divides the sample chromatograms into boiling point fractions up to 35.5 min plus a non-eluted fraction that boils above 700°C (1292°F). It should be noted that packed columns (instead of capillary columns) are employed in ASTM D5307, and the technique extends only to a boiling point of 538°C. The differential and cumulative boiling point distributions of the Whiterocks bitumen are shown in Figure 5. A comparison of the amounts of the 538°C (1000°F) plus fractions of the bitumens, as determined by the conventional D5307 analyses, and the 700°C (1292°F) plus fractions, as determined by the procedure reported herein, is presented in Table III. The Whiterocks bitumen has a 538°C (1000°F) plus fraction of

53 wt% and a 700°C (1292°F) plus fraction of 20 wt%. Thus, by using the technique described in this paper, it was possible to characterize (within the limits of simulated distillation analysis) 80 wt% of the Whiterocks bitumen; that is, 33 wt% more than would have been possible using ASTM D5307. Similarly, 89 wt% of the Asphalt Ridge bitumen (34 wt% more than ASTM D5307), 79 wt% of the PR Spring bitumen (33 wt% more than ASTM D5307), and 73 wt% of the Sunnyside bitumen (32 wt% more than ASTM D5307) were characterized by the proposed method. In general, it was possible to characterize approximately one-third more of these heavy oil samples than would have been possible by conventional ASTM analyses. The distillation cuts, thus obtained, for the four bitumens are presented in Table IV.

If an oil contains a small amount of material boiling above 538°C (1000°F), it should be possible to characterize 100% of the sample using the proposed method. This is illustrated using the extract phases obtained by the SFE of four bitumens with propane. The chromatogram for the extract phase from the Whiterocks bitumen is shown in Figure 6. Analyses with internal standard (analogous to those described in the previous paragraph) for these extract samples revealed that these extracts did not contain any hydrocarbon heavier than C_{90} . The 538°C (1000°F) plus weight fractions of the Whiterocks, Asphalt Ridge, PR Spring, and Sunnyside extracts were 0.08, 0.16, 0.12, and 0.18, respectively. However, all four extract phases were totally eluted with the upper boiling point of 700°C (1292°F). Thus, the procedure developed in this paper allows for complete characterization of samples that consist of relatively small fractions boiling above 538°C (1000°F). The boiling point distribution of the extract phase from Whiterocks bitumen is shown in Figure 7.

The method can also be applied to extremely heavy petroleum residue. Residual fractions of the four bitumens recovered after SFE with propane were used to illustrate this point. Approximately 90 wt% of each of these residues boiled above 538°C (1000°F), as shown in Table III. The extended SIMDIS technique helped characterize 42 wt% of the Whiterocks residue, 39 wt% of the Asphalt Ridge residue, 41% of the PR Spring residue, and 39% of the Sunnyside residue (Table III). Thus, the technique permitted the characterization of an additional 30 wt% of the residual fractions. The chromatograms of the Whiterocks bitumen residual fraction and the boiling point distributions are presented in Figures 8 and 9, respectively.

Table IV. Distillation Cuts for Four Bitumens Analyzed

| Properties | Bitumen source | | | |
|--|----------------|---------------|-----------|-----------|
| | Whiterocks | Asphalt Ridge | PR Spring | Sunnyside |
| <i>ASTM D5307</i> | | | | |
| Volatility ($< 538^{\circ}\text{C}$), wt% | 46.6 | 53.5 | 45.4 | 40.9 |
| Distillation cuts (wt%) | | | | |
| $< 204^{\circ}\text{C}$ | 0.5 | 1.3 | 0.4 | 0.6 |
| 204–344°C | 7.4 | 11.8 | 8.2 | 7.8 |
| 344–538°C | 38.7 | 40.4 | 36.8 | 32.5 |
| $> 538^{\circ}\text{C}$ | 53.4 | 46.5 | 54.6 | 59.1 |
| <i>Modified SIMDIS procedure</i> | | | | |
| Volatility ($< 538^{\circ}\text{C}$), wt% | 46.6 | 53.5 | 45.4 | 40.9 |
| Distillation cuts (wt%) | | | | |
| $< 204^{\circ}\text{C}$ | 0.5 | 1.3 | 0.4 | 0.6 |
| 204–344°C | 7.4 | 11.8 | 8.2 | 7.8 |
| 344–538°C | 38.7 | 40.4 | 36.8 | 32.5 |
| 538–700°C | 33.0 | 34.0 | 33.0 | 32.0 |
| $> 700^{\circ}\text{C}$ | 20.0 | 11.0 | 21.0 | 27.0 |

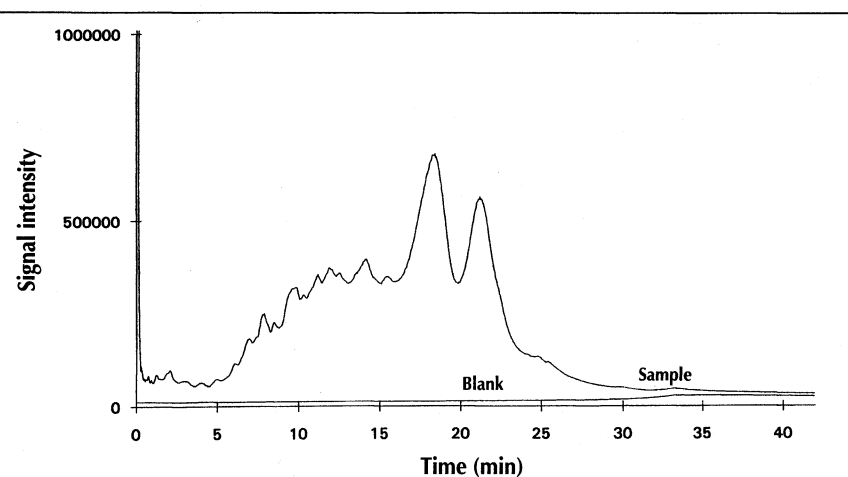


Figure 6. Chromatogram of Whiterocks bitumen extract. A Petrocol EX-2887 column (Supelco) was used with helium as the carrier gas (20 cc/min). The initial temperature was 35°C. It was held for 4.5 min, increased at 12°C/min to 380°C, and held for 8.75 min.

Conclusion

Boiling point distributions up to 700°C (1292°F) and an estimate of the 700°C (1292°F) plus fraction for the ultraheavy oils can be obtained using capillary columns with high phase ratios. Applicability of this technique was demonstrated using ultraheavy oils (bitumens) from the Uinta Basin in Utah, which had volatilities (538°C minus fractions) of 55 wt% or less. The method allowed for the characterization of 30–35 wt% more of the bitumen and bitumen residual fractions obtained by SFE than would have been possible using the ASTM D5307-92 method. Characterization of extract phases with volatilities of 90 wt% was also demonstrated.

Acknowledgments

The financial support of the U.S. Department of Energy through the Laramie Project Office of the Morgantown Energy Technology Center is gratefully acknowledged. The authors also wish to thank Professor A.G. Oblad, for his constant support and encouragement. Mr. John Fausett is thanked for mining the oil sand ores.

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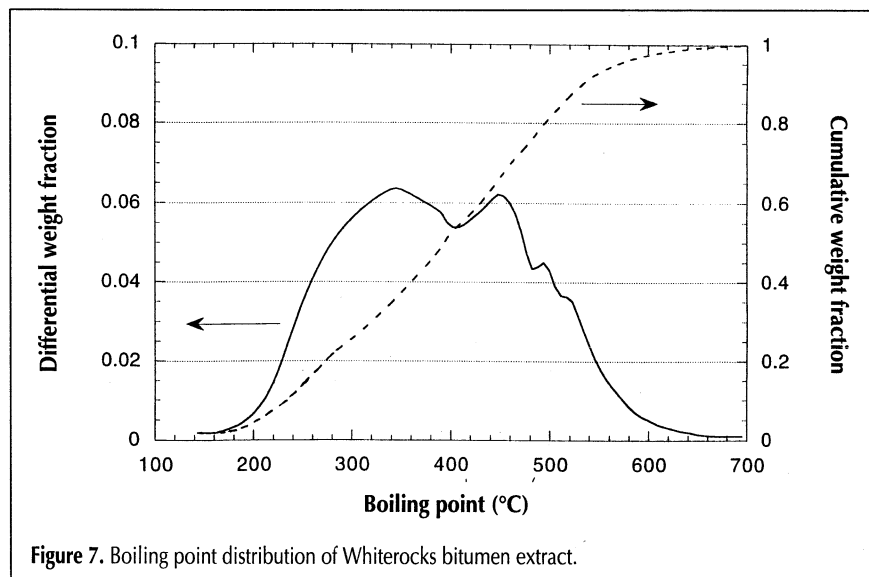


Figure 7. Boiling point distribution of Whiterocks bitumen extract.

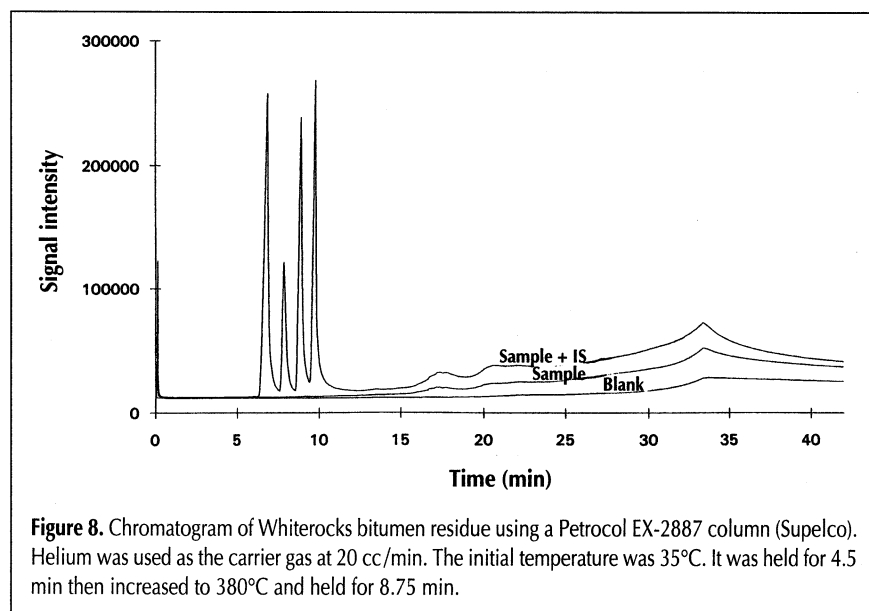


Figure 8. Chromatogram of Whiterocks bitumen residue using a Petrocol EX-2887 column (Supelco). Helium was used as the carrier gas at 20 cc/min. The initial temperature was 35°C. It was held for 4.5 min then increased to 380°C and held for 8.75 min.

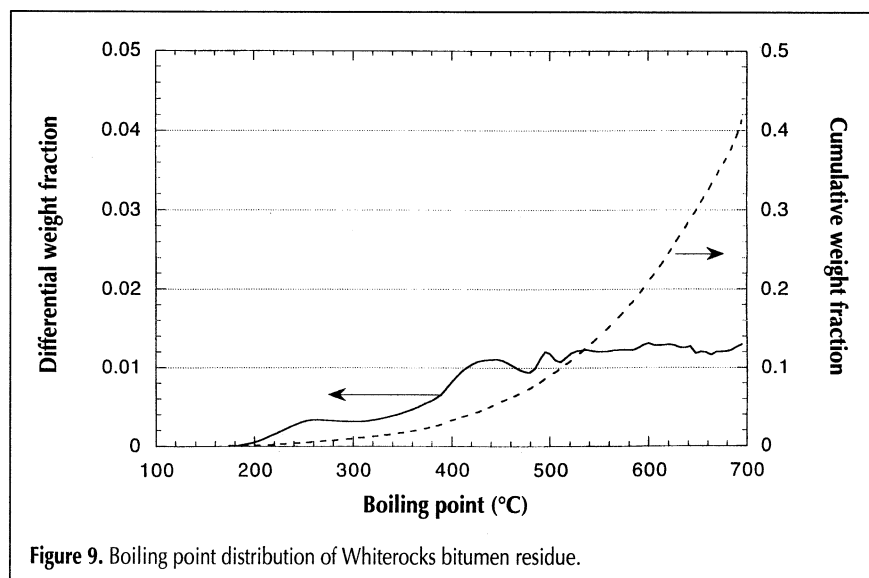


Figure 9. Boiling point distribution of Whiterocks bitumen residue.

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Manuscript accepted May 31, 1995.