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Evaluation of the Potential End Use of Oils Produced by the ROPE© Process From California Tar Sand

Topical Report

K.P. Thomas
P.M. Harnsberger

December 1989

Work Performed Under Cooperative Agreement No.: DE-FC21-86MC11076

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Western Research Institute
Laramie, Wyoming

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SUMMARY

The oil products from Process Development Unit (PDU) run SPR-111 were evaluated for potential end use. This particular run was a five-day test using Arroyo Grande tar sand from California as the feed to the PDU. The distillate from knockout #2 was hydrotreated to produce a series of process intermediates. One of the intermediates was evaluated as a feedstock for the production of transportation fuels. The heavy product oil was distilled to produce a residue that was evaluated as an asphalt.

Analysis of a selected process intermediate shows that it is not suitable for the production of gasoline or for use as a gasoline-blending feedstock. In addition, the process intermediate was not suitable for the production of aviation turbine fuels. This is because the oil contains a high concentration of alkanes. However, the presence of alkanes does make the oil valuable as a feedstock for the production of diesel fuel.

The heavy oil product as received from the PDU is not suitable for the production of an asphaltic material because it contains a large amount of very fine solid material. However, after filtration and distillation, the application of ASTM D-3381 specification tests to the +410°C (+770°F) residue shows that all of the requirements are met except for the trichloroethylene solubility requirement. This value is below specification because a small amount of mineral matter was not removed during the filtration process. In addition, the residue had a very high aging index, which suggests that it is quite susceptible to rapid age-hardening or molecular-structuring and that a pavement constructed of this material will become brittle at a faster rate than a conventional asphalt. Results from successive freeze-thaw cycling also show that the residue is comparable to petroleum asphalts when it is coated on the same appropriate aggregate.

INTRODUCTION

The evaluation or production of transportation fuels and asphalts from tar sand has been the subject of numerous publications. Recently, Thomas et al. (1986 and 1987) evaluated oil recovered by wet forward combustion and bitumen recovered by steamflood from the Asphalt Ridge tar sand deposit in Utah. In general, the properties of the distillates from these materials suggested that they were potential feedstocks for the production of aviation turbine fuels. In particular, the distillates contained appreciable concentrations of cyclic-saturate and aromatic structures that, after further processing (hydrogenation), will be beneficial in the production of a high-density aviation turbine fuel. The original bitumen and distillation products from the bitumen and the thermally produced oil met the specifications of viscosity-graded asphalts. Interestingly, the residues had unusually low aging indexes, which suggested that they may not set properly or that they are resistant to rapid age-hardening. Results from successive freeze-thaw cycling showed that the residue from the thermally produced oil was comparable to or better than some petroleum asphalts when it is coated on the same appropriate aggregates. This suggests that the residue is resistant to moisture-induced loss of strength.

Bunger (1979) and Wenger et al. (1952) investigated the potential of producing asphalt from several tar sand bitumens and came to the same conclusion--that the products compared well with petroleum-derived asphalts and ASTM specifications.

Under contract to the U.S. Air Force, the potential for producing transportation fuels from tar sand bitumens, in particular aviation turbine fuels, was studied Ashland Petroleum Company and the Sun Refining and Marketing Company each produced specification-grade JP-4 and JP-8 turbine fuels from both Big Clifty (Kentucky) and Sunnyside (Utah) tar sands. Ashland concluded that high-quality aviation turbine fuels can be produced from tar sand bitumens but that in today's market the cost is not competitive. Their processing scheme included asphalt residual treatment (ART^{SM}), reduced crude conversion (RCC^{SM}), and hydrotreatment (Moore et al. 1987). Sun also produced specification-grade turbine fuels using a process that consisted of hydrovisbreaking and catalytic hydrotreating or hydrocracking (Talbot et al. 1986).

In this report, we present the results of an evaluation of a distillate and a heavy product oil produced from Arroyo Grande tar sand using the ROPE© process developed by Western Research Institute (WRI). The details of the process and this five-day run are reported by Vaughn et al. (1990).

EXPERIMENTAL

The distillate and heavy product oil were obtained during the conduct of PDU run SPR-111. The experimental details of this run are

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reported by Vaughn et al. (1990). Tar sand from the Arroyo Grande deposit near Edna, California (T31S, R13E, Sec. 31, NW 1/4), was processed in the reactor. The distillate was obtained from knockout #2, and the heavy product oil was obtained from the heavy oil outlet of the screw reactor.

Hydrogenation of the distillate was conducted in a bench-scale, fixed-bed system, operated in the downflow, once-through mode. The operating conditions for the hydrogenation experiments were as follows: pressure of 2000 and 2200 psig, liquid hourly space velocity of 1 and 0.5, hydrogen feed ratio of 5000 and 6000 scfb, and temperatures varying from 329 to 399°C (625 to 750°F). All experiments were conducted using Shell 424 nickel-molybdenum catalyst in the presulfided form. This catalyst has good saturation and cracking capabilities and is resistant to deactivation by nitrogen and sulfur. The catalyst bed was 20 mL and was contained in the bottom of the hot zone of the reactor. The remaining volume of the reactor was filled with the inert material, Cohart grain. The reactor was 9.5 mm i.d. by 105.4 cm long. The six process intermediates were collected during steady-state operation of the reactor. The process intermediates were washed with cold water to remove hydrogen sulfide and ammonia. The organic phase was then dried by stirring for 16 hours with anhydrous magnesium sulfate and filtered to remove the salt.

The chemical and physical properties (elemental composition, specific gravity, and distillation range) of the distillate and the process intermediates were obtained using ASTM or standard procedures developed by WRI. The hydrocarbon-group-type distribution of the distillate and a process intermediate were determined using WRI's proprietary gas chromatographic/mass spectrometric (GC/MS) procedure. In this procedure, the hydrocarbon-group-type composition is determined by using selected ions that are representative of each hydrocarbon-group type. The procedure relies on the gas chromatographic separation of the compounds to minimize interferences from fragment ions of other hydrocarbon groups. Prior to GC/MS analysis, the distillate was chromatographically separated to minimize the concentration of heteroatoms in the hydrocarbon fraction. This was accomplished by chromatographing 0.2 g of distillate on a 1.3- x 61.0-cm column containing Bio-Rad neutral alumina (100 to 200 mesh). The hydrocarbon fraction was eluted with 75 mL of benzene. The polar fraction was not eluted from the column, its contribution to the distillate was determined by difference. The benzene was removed from the hydrocarbon fraction by rotary evaporation. A trace amount of benzene remaining in the hydrocarbon fraction was identified and quantified during GC/MS analysis, and its contribution was accounted for in the recovery. The distillation data determined by ASTM D-2887 is not comparable with data determined by GC/MS. The former method employs low-resolution chromatography and unit sensitivity, whereas the latter method employs high-resolution chromatography and the actual sensitivities determined for various hydrocarbon types.

At the onset, we noted that the heavy product oil contained a substantial amount of very fine solid material. Therefore, the heavy product oil was dissolved in toluene with mechanical agitation, allowed to stand overnight, decanted, centrifuged at 2000 rpm for 20 minutes, and then vacuum filtered through Whatman #50 filter paper. The toluene was then removed by rotary evaporation. Because the initial simulated distillation (before solids removal) showed that the initial boiling point of the heavy oil was 223°C (433°F), loss of the light-end material was not a significant problem during solvent removal.

The heavy product oil was subjected to vacuum distillation to produce a residue suitable for evaluation as an asphalt. The vacuum distillation apparatus consisted of a short-path, all-glass still equipped with a Claisen head and a water-jacketed condenser connected to a fraction cutter. The distillation pot and head temperatures were monitored by Fluke digital thermometers connected to chromel-alumel thermocouples. The pressure was monitored by a Datametrics barocel with a digital readout. The final pot temperature was 252°C (486°F) and the overhead temperature was 210°C (410°F). The pressure was 1.3 mm of mercury. When corrected to standard pressure this resulted in a distillation overhead temperature of 416°C (780°F).

Viscosities of the distillation residue were determined using a Brookfield viscometer model RVT using a number 27 spindle for viscosities measured at 60°C (140°F) and a number 21 spindle for viscosities measured at 135°C (275°F). This is in accordance with ASTM method D-4402. The viscosity of the aged material from the thin-film accelerated-aging test (TFAAT) was determined on a Rheometrics mechanical spectrometer model RMS-605. Measurements were made in the dynamic mode at 60°C (140°F) using 25-mm parallel plates spaced 1.0 mm apart, a shear frequency range of 0.1 to 100 rad/s, and a strain setting of 50%.

The asphalt ASTM specification tests were performed in accordance with the procedures set forth in method D-3381 (ASTM 1985c) for viscosity-graded asphalts. However, viscosities were determined as described above, on a Brookfield viscometer instead of by methods D-2170 and D-2171 (ASTM 1985a and 1985b). Method D-3381 contains two tables that define the minimum (min) and maximum (max) specification values for paving asphalts. The second table contains the more restrictive set of values.

The thin-film accelerated-aging test (TFAAT) is a modification of the ASTM rolling thin-film oven test (RTFO). The TFAAT test subjects 4 g of asphalt to oxidative aging for 24 hours at 130°C (266°F). The aged samples are analyzed by infrared spectrometry (Petersen 1975; Petersen and Plancher 1981) to measure the changes in oxygen-containing functional groups. The aged asphalt from the TFAAT is also subjected to rheological analysis. As part of this analysis, an aging index (the aged viscosity divided by the initial viscosity) is calculated. One part of the rheological analysis provides a ratio of the viscous flow component to the elastic component (G"/G') called tan delta. The degree of aging that takes place in this test appears to simulate the amount of oxidative aging that occurs in a pavement after an extended time of normal service. The thin-film oven test referred to in Table 5 simulates the amount of oxidative aging that occurs in an asphalt mix plant before the preparation of a road.

Test briquets to evaluate water susceptibility (WST) were prepared by the method described by Plancher et al. (1980). Asphalt-aggregate briquets were prepared using a 5 wt % coating of binder on 20- to 35-mesh aggregate particles. The asphalt-aggregate mixture was compacted in a laboratory press to form a 1.90- x 4.13-cm briquet. The test subjects the briquet submerged in water to alternating freeze and thaw cycles while it is suspended on a beveled plastic support. The freeze and thaw cycles were each 12 hours long, the freeze temperature was -10 to -15°C (14 to 5°F), and the thaw temperature was 60°C (140°F). The test was concluded when the briquet broke.

RESULTS AND DISCUSSION

Transportation Fuel Potential

The conditions used for the hydrogenation of the distillate are listed in Table 1. The variables included reactor pressures of 2000 and 2200 psig, liquid hourly space velocities of 1 and 0.5, hydrogen feed ratios of 5000 and 6000 scfb, and reactor temperatures of 329, 349, 371, 385, and 399°C (625, 660, 700, 725, and 750°F). In general, the hydrogen consumption increased with an increase in the severity of the reactor operating conditions. The six process intermediates were taken during steady-state operation of the reactor. As they were produced, the intermediates were brown due to the presence of coke suspended in the oil. This finely divided coke was removed during the subsequent washing, drying, and filtration process. This unexpected production of coke in the oil is probably the result of processing of this highly aromatic feedstock.

Table 1. Hydrogenation Conditions Used for the Conversion of the Distillate

Property	Test #1	Test #2	Test #3	Test #4	Test #5	Test #6
Pressure, psig	2000	2000	2000	2000	2200	2200
Temperature, °C	329	349	371	371	385	399
Liquid Hourly Space Velocity, V_o/V_c/hr	1	1	1	0.5	0.5	0.5
Hydrogen Feed Ratio, scfb	5000	5000	5000	5000	6000	6000
Hydrogen Consumption, scfb	6 6	652	886	942	873	1149

The chemical and physical properties of the distillate and three of the process intermediates are listed in Table 2. The sample number of the process intermediate corresponds to the test number listed in Table

1. In general, the trace amounts of nitrogen and sulfur decreased with increasing severity of the reactor operating conditions and were less than that contained in the original distillate. In addition, the percentage of oil distilling above 371°C (700°F) decreased from 19.1 to 13.0 wt % with increasing severity and was less than that contained in the distillate. The distillate originally contained about 0.5 wt % nitrogen, 1.7 wt % sulfur, and 26.7 wt % distilling above 371°C (700°F). The hydrogen-to-carbon atomic ratio of a process intermediate, sample #6, increased from 1.70 for the distillate to 1.89, and the specific gravity decreased from 0.8972 to 0.8355.

Table 2. Chemical and Physical Properties of the Distillate and Selected Process Intermediates

Property	Distillate	Sample #1	Sample #3	Sample #6
Elemental Composition, wt % or (ppm)				
Carbon	84.8	86.4	86.0	85.6
Hydrogen	12.1	13.2	13.4	13.6
Nitrogen	0.5	(400)	(<20)	(<20)
Sulfur	1.7	(910)	(66)	(140)
Oxygen	1.4	ND	ND	ND
Hydrogen-to-Carbon Atomic Ratio	1.70	1.82	1.86	1.89
Specific Gravity, 16°C	0.8972	ND	ND	0.8355
Distillation Data, vol %, ASTM D-2887				
IBP-177°C	6.8	11.5	11.2	13.6
177-371°C	66.5	69.4	71.3	73.4
+371°C	26.7	19.1	17.5	13.0

ND = not determined

Because sample #6 contained part-per-million levels of heteroatoms and resulted from the most severe hydrogenation conditions, it was analyzed by GC/MS for hydrocarbon-group types. The data for the original distillate are listed in Table 3, and the data for a process intermediate are listed in Table 4. The distillate contained 30.9 wt % of polar material, which was not analyzed by the GC/MS procedure. The data in Tables 3 and 4 are divided into the approximate boiling ranges for the production of gasoline (IBP-177°C/IBP-350°F) and jet and diesel fuel (177-371°C/350-700°F). The distillate contained no material distilling in the gasoline feedstock range, whereas the process intermediate contained about 47 wt % distilling in this range. This

Table 3. Results[a] of Hydrocarbon-Group-Type Analysis of the Distillate Determined by GC/MS, wt %

Hydrocarbon Type	IBP-177°C	177-371°C	+371°C
Alkanes	0	32.0	2.1
Alkenes	0	26.6	0.3
Monocyclic Alkanes	0	0	0
Dicyclic Alkanes	0	0	0
Tricyclic Alkanes	0	0	0
Tetracyclic Alkanes	0	0	0
Pentacyclic Alkanes	0	0	0
Total Saturate Hydrocarbons	0	58.6	2.4
Alkylbenzenes	0	0.4	0
Indanes/Tetralins	0	4.7	0
Naphthalenes	0	1.6	0
Fluorenes	0	1.1	0
Anthracenes/Phenanthrenes	0	0.3	0
Pyrenes/Fluoranthenes	0	0	0
Total Aromatic Hydrocarbons	0	8.1	0
Total Hydrocarbons	0	66.7	2.4

[a] 30.9 wt % of the sample was not analyzed by GC/MS because it was in the polar fraction.

Table 4. Results of Hydrocarbon-Group-Type Analysis of a Process Intermediate, Test Fuel #6, Determined by GC/MS, wt %

Hydrocarbon Type	IBP-177°C	177-371°C	+371°C
Alkanes	7.7	27.7	0.8
Alkenes	14.2	15.4	0
Monocyclic Alkanes	19.0	2.2	0
Dicyclic Alkanes	0	1.2	0
Tricyclic Alkanes	0	0	0
Tetracyclic Alkanes	0	0	0
Pentacyclic Alkanes	0	0	0
Total Saturate Hydrocarbons	40.9	46.5	0.8
Alkylbenzenes	5.9	2.1	0
Indanes/Tetralins	0	3.8	0
Naphthalenes	0	0	0
Fluorenes	0	0	0
Anthracenes/Phenanthrenes	0	0	0
Pyrenes/Fluoranthenes	0	0	0
Total Aromatic Hydrocarbons	5.9	5.9	0
Total Hydrocarbons	46.8	52.4	0.8

hydrotreated product still contained some alkenes (14 wt %), which need to be hydrogenated before a stable fuel can be produced from this distillate fraction. Unfortunately, this distillate fraction, after hydrotreating to convert the alkenes to alkanes, will have too high of a concentration of alkanes to be suitable for the production of gasoline or gasoline-blending stock. Too high of a concentration of these alkanes will have a detrimental effect on the octane rating of the finished fuel. The distillate also contained about 67 wt % distilling in the jet and diesel fuel feedstock range, whereas the process intermediate contained about 52 wt % distilling in this range. Again the hydrotreated product still contained some alkenes (15 wt %), which will have to be hydrogenated before a stable fuel can be produced. Upon hydrogenation, the alkenes are be converted to alkanes. Consequently, the 177-371°C (350-700°F) distillate from the process intermediate is not suitable for the production of conventional or high-density aviation turbine fuels because the alkane content is too high. The limit for this class of compounds in a high-density turbine fuel is 10 vol %. High concentrations of alkanes in aviation turbine fuels can have an adverse effect on the freeze point of the fuel. However, because of the high alkane content of this distillate range, this fraction is valuable as a diesel fuel blending stock. In diesel fuels alkanes are valuable because they have a positive effect on the cetane number.

Asphalt Potential

The heavy oil product from SPR-111 contained a large amount of very fine solid material, which was removed by filtration. The amount of solids removed represented 27.9 wt % of the total material. The original heavy oil product had a viscosity of 79.4 P at 60°C (140°F) and after filtration it had a viscosity of 45.3 P at 60°C (140°F). A particle-size analysis was performed on the solid material that was removed by filtration, and this analysis showed that the bulk of the particles range in size from 2 to 10 μm. Combustion of the solid material at 750°C (1382°F) showed that 81.5 wt % of the solid material was mineral matter. These data imply that some of the organic material in the tar sand cokes on the mineral particles during the ROPE® process. The large amount of fine solid material that was contained in the original heavy oil product probably make it economically infeasible for its use in the production of an asphalt cement for paving applications. The cost of preparing 99+% of solid-free oil will be excessive in a commercial application.

After vacuum distillation of the filtered heavy oil product to an equivalent temperature of 416°C (780°F), the viscosity of the residue was 1381 P at 60°C (140°F), slightly high for an AC-10 asphalt. Therefore, 6.06 grams of the 385 to 416°C (725 to 780°F) distillate (which is 0.9% of the total material, 11.5% of the 385 to 416°C/725 to 780°F distillate, or 4.4% of the total distillate) were added back to the residue and blended. This resulted in a residue with a viscosity of 1060 P at 60°C (140°F).

Comparison of the vacuum distillation data and the simulated distillation data obtained by gas chromatography shows a good correlation. The vacuum distillation resulted in 79.8 wt % residue boiling above 416°C (780°F). This compares well with the 79.2 wt % determined by simulated distillation. After blending back the small amount of the 385 to 416°C (725 to 780°F) distillate, the amount of residue is 80.7 wt %. Using the data from simulated distillation, 80.7 wt % residue is reached at 410°C (770°F).

Examination of the data in Table 5, obtained in the evaluation of the residue shows that it did not pass one of the ASTM method D-3381 requirements for an AC-10 viscosity-graded asphalt. The requirement not met was the trichloroethylene solubility test. This is an indication of the difficulty in removing solids to a level of 99+% solubility of the residue and the extreme fineness of the solids in the original heavy oil. All other values obtained in the ASTM method D-3381 testing show that the residue meets the requirements in Table 1 but not the more stringent requirements in Table 2. With the exception of the solubility value, these results are quite similar to findings previously reported on other tar sand asphalts (Thomas et al. 1986; Thomas et al. 1987).

Table 5. Results of ASTM D-3381 Specification Tests on +410°C Residue

Test	Specifications for AC-10	+410°C Residue
Viscosity, 60°C, P	1000 ± 200[a]	1060
Viscosity, 135°C, cP, min	150[b], 250[c]	200
Penetration, 25°C, 100 g, 5 s, dmm, min	70[b], 80[c]	97
Flash point, Cleveland open cup, °C, min	219[a]	290
Solubility in trichloroethylene, %, min	99.0[a]	98.1
Tests on residue from thin-film oven test		
Viscosity, 60°C, P, max	5000[a]	3153
Ductility, 25°C, 5 cm/min, cm, min	50[b], 75[c]	80

[a] Value common to ASTM D-3381 Tables 1 and 2
[b] ASTM D-3381, Table 1
[c] ASTM D-3381, Table 2

The data from infrared spectroscopic analysis of the +410°C (+770°F) residue before and after the thin-film accelerated-aging test (TFAAT) are listed in Table 6 and suggest that the residue experienced oxidation before TFAAT aging because it contained 0.145 mol/L ketones. This is expected from an oil that was thermally produced. The amount of sulfoxide present before aging, <0.239 mol/L, also suggests prior oxidation. The exact amount of sulfoxide was masked by an absorption band at about 1060 cm^{-1}. There was only a relatively small increase in the amount of sulfoxide formed during TFAAT aging. This suggests that the most reactive sulfides in the original tar sand were oxidized during the recovery process or that the sulfoxides did not survive the high temperature of the process. No phenolic or pyrrolic compound types were detected in either the +410°C (+770°F) residue or the TFAAT-aged residue. The only other oxidation products formed during the TFAAT were small amounts of carboxylic acids and carboxylic acid anhydrides.

Table 6. Infrared Spectroscopic Analysis of Unaged and TFAAT-Aged +410°C Residue

Functional Group	Unaged	Aged
Ketones, mol/L	0.145	0.320
Anhydrides	0.000	0.045
Carboxylic Acids	0.000	0.008
2-Quinolone Types	0.017	0.012
Sulfoxides	<0.239	0.381
Pyrrolic NH	0.000	0.000
Phenolic OH	Trace	Trace

The data from rheological analysis of the sample before and after TFAAT aging are listed in Table 7. These data suggest that the residue had an extremely high aging index. This sample has an aging index that is about two orders of magnitude higher than most petroleum asphalts. An asphalt with such a high aging index will be very susceptible to cracking and embrittlement even in the most favorable climates. The extremely high aging index may be the result of the fine solid material in the residue catalyzing the oxidation process, or perhaps the solid material promotes molecular structuring very rapidly after aging producing the large observed viscosity increase. These possibilities can be evaluated through further testing.

Rheological measurements were made in the dynamic mode, which facilitates obtaining the elastic and viscous flow components of viscosity. The elastic modulus and viscous flow modulus are related to viscosity by the following equation:

$$\eta^* = \frac{\sqrt{(G')^2 + (G'')^2}}{\omega}$$

Table 7. Rheological Characteristics of Unaged and TFAAT-Aged Petroleum Asphalts and +410°C Residue

Sample	Dynamic Viscosity, Poise, 60°C		Tan Delta		Aging Index
	Unaged	Aged	Unaged[a]	Aged[b]	
+410°C Residue	1.06×10^3	1.66×10^7	13.8	1.08	15,660
Wyoming Sour	1.50×10^3	3.04×10^5	17.8	1.52	202.6
Boscan Asphalt	8.11×10^2	1.79×10^5	8.5	1.19	327.3
California Coastal Asphalt	1.27×10^3	1.72×10^5	10.4	1.21	557.3

Tan delta = viscous flow modulus/elastic modulus
Aging Index = aged viscosity/unaged viscosity
[a] Tan delta determined at $G^* = 1.9 \times 10^4$
[b] Tan delta determined at $G^* = 2.1 \times 10^6$

where η^* is the dynamic viscosity, G' is the elastic modulus, G'' is the viscous flow modulus, and ω is the shear frequency. The elastic modulus is related to the ability of a system to recover from deformation caused by the rapid loading and unloading of traffic. The viscous flow modulus is related to plastic or permanent deformation of a system caused by a load. These are also related to the viscous flow versus elastic storage in pavement when it undergoes thermal cycling from day to night. The tan delta (viscous flow modulus divided by the elastic modulus) is a ratio used to measure the relative contribution of each component to the viscosity (Ferry 1961). A high tan delta at low temperatures is related to the ability of a system to relieve low temperature thermal stress by creep flow (very slow viscous flow). However, at higher temperatures (such as 60°C/140°F), a high tan delta may suggest a tender asphalt (an asphalt that does not set properly). Poor setting characteristics in pavement result in a permanently deformed (damaged) roadway.

The tan delta value for the unaged residue falls in the range typical of petroleum asphalts; however, the tan delta value for the aged residue is significantly lower than the aged petroleum asphalts. This is another indication that the residue will be subject to cracking and embrittlement. The tan delta values are compared at the same G^*, or complex dynamic modulus. The G^* value:

$$G^* = \sqrt{(G')^2 + (G'')^2}$$

can be called the stiffness value; therefore, the tan deltas are all compared at the same asphalt stiffness. In effect, the extremely high viscosity of the aged residue is factored out in this comparison.

The water susceptibility test (WST) is an indirect measure of the resistance of an asphalt-aggregate mixture to moisture-induced damage. The data from WST analysis are listed in Table 8. The +410°C (+770°F) residue performed adequately when compared with petroleum asphalts. The Teton aggregate used in the test is very susceptible to water stripping, whereas the Wyoming limestone shows moderate resistance to water stripping. Although the sample prepared with the Teton aggregate broke on the first cycle as did the petroleum asphalts, other binders that have been tested on this aggregate have virtually disintegrated on the first cycle. The sample prepared with the +410°C (+770°F) residue on the Wyoming limestone aggregate showed improvement over the petroleum asphalt. The petroleum asphalt briquet broke after 14 cycles, whereas the briquet prepared with the +410°C (+770°F) residue survived more than 30 cycles without breaking. The Wyoming Sour petroleum asphalt is a common asphalt used in the region where both aggregates are obtained. The California Coastal petroleum asphalt is another common asphalt that was chosen for comparison. These two petroleum asphalts were chosen for comparison purposes only and do not necessarily represent the results of all petroleum asphalts on these two selected aggregates.

Table 8. Results of Water Susceptibility Tests of Petroleum Asphalts and +410°C Residue

Aggregate	Binder	Cycles to Failure
Wyoming Limestone	+410°C Residue	>30
	Wyoming Sour	14
Teton Mountain Gravel	+410°C Residue	1
	Wyoming Sour	1
	California Coastal	1

CONCLUSIONS

A series of hydrotreating experiments was conducted on the distillate obtained from PDU run SPR-111. In general, the amounts of heteroatoms decreased with increasing severity of the reactor operating conditions. A process intermediate was selected and analyzed for hydrocarbon-group types, and the subsequent data were arranged according to gasoline and jet and diesel fuel production. These data show that the fraction of the process intermediate distilling in the gasoline feedstock range is inappropriate for the production of either gasoline or gasoline-blending stock because it is too high in alkanes. The fraction of material distilling in the jet and diesel fuel feedstock range is also inappropriate for the production of aviation turbine fuels because it is too high in alkanes. However, the presence of the alkanes makes this distillate fraction valuable for the production of diesel fuel.

The heavy oil product contained a large amount of very fine solid material that makes it impractical to use for the production of an asphaltic material. After filtration and distillation of the heavy oil product, the +410°C (+770°F) residue met all of the ASTM specification tests for viscosity-graded asphalts except the solubility specification. This specification was not met because the residue still contained a small amount of fine solid material. The +410°C (+770°F) residue had a very high aging index, which suggests that it is quite susceptible to rapid age hardening or molecular structuring and that a pavement constructed with this material will be subject to excessive embrittlement. Results from successive freeze-thaw cycling show that the residue is comparable to petroleum asphalts when it is coated on the same appropriate aggregates.

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DISCLAIMER

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