

## IMPROVEMENT IN COAL LIQUEFACTION SOLVENT QUALITY BY DEWAXING

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### ABSTRACT

Recycle oils from the Integrated Two-Stage Liquefaction (ITSL), H-Coal and Solvent Refined Coal (SRC) processes were dewaxed by variants of commercial dewaxing processes yielding up to 47 wt % "wax". Dewaxing methods used include the ketone and the urea adduction techniques. The clean waxes are reasonably pure paraffins. The dewaxed oils were substantially better coal liquefaction solvents than the original (non-dewaxed) oils in batch liquefaction tests. For example, in one case, dewaxing improved the conversion of a standard coal to tetrahydrofuran solubles at standard reaction conditions from 71% with the original oil to 87% with the dewaxed oil. These data provide a direct indication of the inimical effect of paraffinic components on solvent quality. The impact of solvent quality is particularly relevant to two-stage liquefaction, in which thermal first-stage reactions proceed in a recycle solvent. In addition, these results indicate the technical feasibility of dewaxing coal liquefaction recycle oils by commercially available technology to improve solvent quality and to produce a useful by-product. Dewaxing could be applied to any liquefaction process that uses a deashed (preferably distillate) recycle stream.

### INTRODUCTION

Paraffinic and other saturated hydrocarbons are well known components of coal and coal liquefaction products (1). The presence of substantial quantities of saturated hydrocarbons in coal liquefaction recycle solvents has been reported (2). Increasing concentrations of these compounds (as well as of highly alkylated compounds) have been linked to a decreasing quality of the recycle oil as a donor solvent for coal liquefaction (2). Other work has demonstrated that the effectiveness of coal liquids as coal liquefaction donor solvents shows a negative correlation with the paraffinic nature of the coal liquid (3). In the development of the Consol Synthetic Fuels (CSF) (4) and Exxon Donor Solvent (EDS) (5) processes, it was recognized that the build-up of saturated hydrocarbons in the recycle solvent resulted in deteriorating solvent quality. Paraffinic and other saturated hydrocarbons are known to be non-donors (6) or at least very poor donors (7,8) at coal liquefaction conditions. Their presence in recycle oils reduces solvent quality, at least by diluting the active solvent molecules and at worst by acting as a detrimental antisolvent that leads to reduced solvent effectiveness.

Saturated hydrocarbons in coal liquefaction recycle oils are formed in part by complete hydrogenation of aromatics to form naphthenes. However, the majority of the paraffins (and particularly the n-paraffins) must ultimately arise either unchanged directly from the coal (9) or as products of the cracking of alkyl side-chains or of larger paraffins. If the recycle solvent is higher boiling than the major liquefaction products, saturates must exit the recycle loop by cracking to lighter products. Paraffins, however, tend to crack selectively to gases (5) thus consuming expensive hydrogen while producing undesirable gas.

The quality and paraffin content of the recycle solvent at equilibrium is fixed for each liquefaction process by the plant configuration, feed coal and operating conditions in use at any time. To reduce the paraffin content of the recycle solvent in order to improve its quality, operating conditions must be changed if the

feed coal and plant configuration are held constant. However, by changing operating conditions, product yield slate and/or product quality may be undesirably affected.

This paper presents a novel application of commonly used commercialized technology to improve the quality of coal liquefaction recycle solvents. That technology, in common use in the petroleum refining industry, is dewaxing. When applied to coal liquefaction recycle solvents, dewaxing improves donor solvent quality by removing predominantly the paraffins and other saturated hydrocarbons that are undesirable components. If applied commercially, a high-value by-product wax could be sold. For example, recent price ranges (10) of related products follow: paraffin wax, \$0.16-0.46/lb; petrolatum, \$0.30-0.40/lb; montan wax, \$0.58-0.65/lb; micro-crystalline wax, \$0.36-0.48 lb; and mineral oil, \$2.68-3.10/gal. These prices are considerably greater than commanded by fuels. Removal of paraffins and other saturates from the liquefaction process this way may also reduce gas production and hydrogen consumption and would reduce the occurrence of wax precipitation at low temperature from products with boiling points similar to the dewaxed stream.

Various dewaxing methods are now, or have been, in commercial use in the petroleum industry (11) including pressing and sweating, centrifugation, solvent dewaxing (e.g., the propane and ketone processes) and urea adduction methods. The bulk of the experiments reported here used a laboratory version of the ketone process, though the urea adduction process was also tested, both with promising results. Commercially, methyl ethyl ketone is typically employed in the ketone process (11); acetone was used in our experiments for convenience. These experiments demonstrate on a small scale the technical feasibility of improving coal liquefaction solvent quality by dewaxing using commercially available technology.

The impact of solvent quality is particularly relevant to processes using a thermal reactor in which coal liquefaction proceeds in and depends upon a recycle solvent such as the ITSL, SRC-I, SRC-II and EDS processes. Dewaxing should be directly applicable to processes that recycle at least one distillate-only stream such as ITSL (the reconfigured mode in use at Wilsonville), SRC-I, EDS (without bottoms recycle) and H-Coal. It should also be possible to dewax any intermediate distillate stream if the recycle does not contain a separate distillate component. One experiment demonstrated that a deasphalted residual oil could also be dewaxed.

## EXPERIMENTAL

### Ketone Dewaxing

A weighed amount (about 85 g) of the oil to be dewaxed was mixed with acetone (Fisher HPLC grade) in the desired ratio (1/2 to 1/3.3 by volume) in a beaker equipped with a magnetic stirrer. All oils tested, except the single 850°F<sup>+</sup> resid, dissolved readily at room temperature. The contents were cooled while stirring in a dry ice/acetone bath to the desired temperature to precipitate the waxes. When the desired temperature (-20 or -50°C) was reached, the mixture was immediately filtered, while still cold, in a Buchner funnel equipped with a glass-fiber filter (Reeve Angel #934AH). The filter cake (wax) was washed with additional cold acetone approximately equaling the volume of the original oil/acetone mixture. This filtrate was set aside. The wax cake was washed through the filter with freshly distilled tetrahydrofuran (THF). Each filtrate (acetone and THF) was rotary evaporated at about 60°C to constant weight to remove all traces of solvents, leaving the dewaxed oil and the waxes, respectively, which were then weighed and analyzed. The product waxes were usually a white solid though some of the less pure waxes were discolored.

One sample, a solid deasphalted 850°F<sup>+</sup> resid, was ketone dewaxed using a somewhat different method. The solid (32g) was dissolved in 120g of an 80/20 v/v solution of acetone and freshly distilled toluene, then dewaxed as above at -50°C. These

waxes (12% of oil) were very impure and so were subjected to a second dewaxing procedure similar to the first except that the solvent used was 100 mL of a 70/30 v/v mixture of acetone and toluene. This product was a very hard brown waxy solid.

#### Urea Dewaxing

One oil sample was dewaxed by the urea adduction method. About 50g of the oil was weighed and diluted with an equal weight of  $\text{CH}_2\text{Cl}_2$  (MCB reagent). To this mixture was slowly added about 50 mls of an aqueous solution of urea (Fisher certified) saturated at 80°C which crystallized upon cooling on contact with the oil/ $\text{CH}_2\text{Cl}_2$  solution. The mixture was stirred for one hour, filtered in a Buchner funnel equipped with a glass-fiber filter, then washed with several aliquots of  $\text{CH}_2\text{Cl}_2$ . This filtrate was set aside. The filter-cake was washed with warm water to dissolve the urea leaving the waxes on the filter. The water wash was discarded. The waxes were washed through the filter with freshly distilled THF. The  $\text{CH}_2\text{Cl}_2$  and THF filtrates were stripped of solvent by rotary evaporation to produce the dewaxed oil and the waxes, respectively, which were weighed and analyzed. The product wax was a white solid.

#### Solvent Quality (Microautoclave) Tests

Samples were tested for their effectiveness as coal liquefaction donor solvents using a standard microautoclave test. This test, called the modified equilibrium test, has been described in detail previously (3). Briefly, 6g of a standard coal and 9g of the sample to be tested are heated to 750°F for 30 min without added gas in a 30 mL microautoclave. The contents are cooled and extracted with THF to determine the conversion of coal to solubles. This test is an authentic coal liquefaction experiment. Solvent effects can be tested easily because there is no interfering catalyst or extraneous gas present. The results of this test serve as an empirical measure of donor solvent quality. Coal conversions obtained with several pure model compounds follow: n-tetradecane, 25.4%, 1-methylnaphthalene, 48.2%, tetralin, 85.4%. This test is reproducible to 1.2% absolute (standard deviation).

#### Other Analyses

Samples were analyzed by  $^1\text{H-NMR}$  using a procedure described in detail elsewhere (3) to determine the effectiveness of dewaxing and to determine the purity of the waxes. Briefly, the  $^1\text{H-NMR}$  spectrum is divided in regions roughly corresponding to different proton types. For example, the region between 10.0 and 4.7 ppm is assigned to aromatic protons and the region between 1.4 and 0.5 ppm is assigned to paraffinic protons. Paraffinic protons are protons on internal  $-\text{CH}_2-$  groups or  $-\text{CH}_3$  groups of paraffins and long alkyl chains. Reproducibility is 0.2% absolute (standard deviation).

Gas chromatography (GC) was performed with a Perkin-Elmer Sigma 2000 instrument equipped with dual flame ionization detectors. The column, a 30m x 0.25mm DB-5 column from J&W Scientific, was initially at 50°C for 4 min, then programmed to 280°C at 4°C/min and held for 20 min. Carrier gas was 20 psig  $\text{H}_2$ . Injector and detector temperatures were 300°C. A split injection of 0.2 $\mu\text{L}$  of sample (0.1g/mL in THF) was used. Quantitation was based on peak areas referred to an n-decane internal standard.

Elemental analyses (C, H, N and S) were performed with Leco CHN-600 and SC32 instruments. Though these instruments were designed for the analysis of coals, not oils, reliable N and S determinations can be made. C and H values are less reliable, but are probably accurate to  $\pm 1\%$  absolute.

#### Feed Oils

All samples were authentic coal liquefaction recycle oils.

- #1 ITSL subbituminous distillate - A composite of the 850°F<sup>-</sup> distillate portions of twenty daily samples taken between 5/6 and 8/6/84 of the recycle solvent (V-131B) from Wilsonville Run 246 made with Wyoming (Clovis Point mine) subbituminous coal.
- #2 ITSL subbituminous distillate - The 850°F<sup>-</sup> distillate portion of a sample of hydrotreater flashed bottoms (V-1067) taken 9/14/85 from Wilsonville Run 249 made with Wyoming (Clovis Point mine) subbituminous coal.
- #3 ITSL bituminous distillate - A composite of the 850°F<sup>-</sup> distillate portions of fourteen daily samples taken between 9/12 and 12/9/84 of the recycle solvent (V-131B) from Wilsonville Run 247 made with Illinois 6 (Burning Star mine) bituminous coal.
- #4 H-Coal subbituminous distillate - the 1000°F<sup>-</sup> distillate portion (96.1%) of a composite of seventeen daily samples taken between 9/1 and 9/17/80 of the "clean oil" (a component of the recycle solvent) from H-Coal PDU Run 10 made with Wyoming (Wyodak mine) subbituminous coal.
- #5 SRC-1 bituminous distillate - the 1000°F<sup>-</sup> distillate portion (95.6%) of a sample of recycle solvent taken 10/2/78 from Wilsonville Run 149 made with Kentucky #9 bituminous coal.
- #6 ITSL subbituminous deasphalted resid - the hexane-solubles of a composite of the 850°F<sup>+</sup> resid portions of twenty-one daily samples taken between 5/6 and 8/6/84 of the recycle solvent (V-131B) from Wilsonville Run 246 made with Wyoming (Clovis Point mine) subbituminous coal.

## DISCUSSION

Results from dewaxing experiments using authentic coal liquefaction recycle oils are discussed below. In this report, "wax" refers to the precipitated portion of the oil obtained in the procedure, "dewaxed oil" refers to the non-precipitated portion and "feed oil" refers to the original, untreated oil. It should be recognized that the ketone process as commercially practiced is performed in two stages called dewaxing and de-oiling (11). Except in one case, these experiments were done in a single stage. Therefore, it is expected that these results could be further improved.

Experimental conditions and yields are shown in Table 1. Results of microautoclave liquefaction tests are shown in Table 2. Analyses of the various oils are shown in Table 3. <sup>1</sup>H-NMR spectra and gas chromatograms of the feed oil, dewaxed oil and waxes from experiment 5 are shown in Figures 1 and 2.

### Wax Yields

Wax yields ranged from 3 to 47 wt % (Table 1). Wax purity spanned a range as well. Generally, the greatest yields of wax and the purest waxes were produced from oils derived from subbituminous coal (feed oils #1, 2 and 4). Those oils were very paraffinic, as determined by the paraffinic hydrogen content from <sup>1</sup>H-NMR and by GC (Table 3) and would be expected to produce the most wax. This is consistent with the concept that lower rank coals tend to be more paraffinic. One highly paraffinic oil produced from subbituminous coal (feed oil #2) yielded 47% of a reasonably pure wax (Tables 1 and 3).

The wax yields obtained in these experiments would not be expected to be attained at equilibrium in a liquefaction process employing dewaxing. The liquefaction processes from which these samples were taken all employed recycle and therefore, the total feed to these liquefaction processes included varying amounts of wax components. If dewaxing of all or part of the recycle were used, the wax content of the feed would be reduced thereby reducing the wax content of the product. It may be possible to dewax only that portion of the recycle solvent that is necessary

to keep wax levels below some set point. Dewaxing only a portion of the recycle solvent could reduce both capital and operating costs of the dewaxing unit.

#### Improvement in Donor Solvent Quality

In all cases, donor solvent quality, as measured by microautoclave tests, increased upon dewaxing. In general, the improvement in solvent quality upon dewaxing, as measured by the difference in the microautoclave tests with the feed oil and the corresponding dewaxed oil, increased with increasing wax yield. Thus, only 3% wax was removed in experiment 11 giving an improvement in donor solvent quality from 63 to 66%, whereas 47% wax was removed in experiment 14 giving an improvement in donor solvent quality from 71 to 87%. Clearly, the increase in donor solvent quality results from reducing the concentration of paraffins and other saturates which are non-donors and are generally considered to be poor physical solvents for coal liquids. In fact, paraffins have been found to be inimical to solvent quality in both microautoclave tests (3) and in the development of the CSF (4) and EDS (5) processes.

In direct coal liquefaction processes, the quality of the recycle oil generally is fixed by the feed coal, the operating conditions and the characteristics of the process. Making changes in operating conditions to improve the product slate or to compensate for catalyst deactivation can have the undesirable side effect of reducing solvent quality which, in turn, can affect product yields unexpectedly. Alternately, operating conditions that provide a high quality recycle solvent may not be desirable from a product yield or product quality standpoint. Dewaxing provides a means of improving recycle solvent quality that is independent of liquefaction conditions and may permit simultaneous optimization of product and recycle-solvent qualities.

It is interesting to note that the three dewaxed distillate ITSL recycle solvents all gave similar coal conversions in the microautoclave tests, even though the non-dewaxed feed oils gave significantly different results as shown below.

Run No.	Feed Oil	Coal Conversion, wt % MAF	
		Feed	Dewaxed
5	#1	80.9	88.2
13	#3	79.2	86.2
14	#2	70.8	87.1

This result would indicate that not only can donor solvent quality of recycle oils be improved by dewaxing, but that donor solvent quality can also be made more constant regardless of feed coal.

#### Effect of Temperature on Ketone Dewaxing

Experiments were performed with three feed oils at both -20 and -50°C. In each case, the lower temperature produced about twice as much wax (Table 1). The dewaxed oils produced at -50°C were better coal liquefaction donor solvents as measured by microautoclave tests (Table 2). This is consistent with the lower paraffinic content of those dewaxed oils (Table 3). However, the waxes produced at -50°C were lower purity paraffins than those produced at -20°C as evidenced by the increased aromaticities and carbon contents and decreased hydrogen and paraffinic hydrogen contents and by the GC results (Table 3). Even though the additional material removed at -50°C was largely not paraffinic, its removal further improved donor solvent quality. It is believed that the additional material removed at -50°C largely consists of highly saturated and alkylated compounds. Clearly, ketone dewaxing can be performed to maximize the improvement in solvent quality or to maximize the purity of the product wax depending on operating temperature. It should be possible to optimize both features simultaneously by selecting appropriate

temperature, time and solvent power conditions. These experiments were all operated with no hold time at temperature. Commercial petroleum dewaxing operations tailor solvent power by using solutions of varying ratios of ketone and toluene as the dewaxing solvent (11). Commercial petroleum operations also improve the selectivity of the process by operating in two stages in which the wax is separated and then de-oiled (11).

#### Ketone Dewaxing of Resid

One deasphalted 850°F<sup>+</sup> resid sample was dewaxed (experiment 12) yielding 7.9% of a very hard wax. The results of <sup>1</sup>H-NMR and elemental analyses (Table 3) indicate the wax is reasonably pure paraffin. This wax was too high boiling for complete GC analysis, but the eluted components were predominantly n-paraffins containing 24 to over 40 carbon atoms. This wax was very hard and had a freezing point upon cooling of 54°C by differential scanning calorimetry. This experiment showed that deasphalted resids can be dewaxed and may indicate that full-range coal liquefaction recycle oils can be dewaxed providing that they are solids-free and asphaltene-free. This may have applicability in ITSL and the developing Catalytic Two-Stage Liquefaction process in which the full-range recycle oil is typically solids-free and, when processing subbituminous coal, contains relatively low levels of asphaltenes (12,13).

#### Nature and Quality of Product Waxes

The product waxes were predominantly saturated hydrocarbons. This is evidenced by their very low H-aromaticities (as low as 0.3% aromatic/total hydrogen) and their elemental analyses (Table 3). Specifically, these saturated hydrocarbons were mostly paraffins as shown by the high concentration of paraffinic protons from <sup>1</sup>H-NMR analysis (as high as 91% paraffinic/total hydrogen). Even pure paraffins do not give 100% paraffinic protons in this <sup>1</sup>H-NMR analysis because of spinning side bands. For example, pure n-tetracosane gives 92.1% paraffinic protons in this analysis. n-Paraffins predominate in the gas chromatograms of the waxes (Table 3 and Figure 3), accounting for as much as 60% of the wax. The carbon preference indices (14) of the waxes were near unity, averaging 1.03 with a standard deviation of 0.04 (range = 0.92 to 1.09).

Dewaxing is most efficient for the higher molecular weight n-paraffins as seen by the GC data in Table 3. For all but one experiment, the n-paraffin of greatest concentration has the highest carbon number in the wax and the lowest carbon number in the dewaxed oil.

Enough wax was produced in experiments 13 and 14 to test the effectiveness of the wax as a donor solvent. The waxes from these experiments were also two of the least pure waxes recovered. As shown in Table 2, these waxes performed considerably more poorly than the corresponding feed oils, though not as poorly as pure n-tetracosane which gave 25.4% coal conversion in the same test. It is expected that the purer waxes would behave more similarly to the n-tetracosane.

#### ACKNOWLEDGEMENT

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TABLE 1  
EXPERIMENTAL CONDITIONS AND PRODUCT YIELDS

Experiment No.	Feed Oil I.D.	Conditions		Yields, wt %		
		T, °C	Acetone/Oil, v/v	Wax	Dewaxed Oil	Mass Balance
<u>Urea Adduction Method</u>						
2	#1	Room	(b)	5.0	88.8	93.8
<u>Ketone Method</u>						
4	#1	-20	2.0	9.1	90.7	99.8
5	#1	-50	3.3	20.6	79.3	99.9
14	#2	-50	3.0	47.4	52.0	99.4
7	#3	-20	3.0	14.5	83.2	97.7
13	#3(a)	-50	3.0	32.7	66.5	99.1
9	#4	-20	3.0	5.2	93.9	99.1
10	#4	-50	3.0	9.2	90.0	99.1
11	#5	-50	3.0	3.2	96.3	99.5
12	#6	-50	(c)	7.9	90.7	98.6

- (a) Redistilled to 1000°F immediately before experiment (98.3% distillate).  
 (b) Urea method used, see Experimental section.  
 (c) 850°F residual oil used as feed, see Experimental section.

TABLE 2  
MICROAUTOCLAVE TEST RESULTS

Experiment No.	Coal Conversion, wt % MAF		
	Feed Oil	Dewaxed Oil	Waxes
2	80.9	84.9	-
4	80.9	87.1	-
5	80.9	88.2	-
14	70.8	87.1	55.1
7	80.1	83.7	-
13	79.2	86.2	62.5
9	55.7	55.4/57.2	-
10	55.7	60.5	-
11	63.3	65.6	-

TABLE 3  
ANALYSES OF FEEDS AND PRODUCTS

Experiment No.	Fraction	Hydrogen Types by <sup>1</sup> H-NMR, %		Elemental Analysis, wt % (a)				n-Paraffins by GC		
		Aromatic	Paraffinic	C	H	N	S	Conc., wt %	Range	Max. Conc.
2	Feed	14.6	42.6	89.1	10.0	0.3	<0.1	5.7	15-33	22
	Dewaxed	17.0	34.2	88.4	9.8	0.4	<0.1	3.2	15-32	22
	Wax	2.6	82.5	80.9	13.9	0.9	<0.1	47.7	13-35	25
4	Feed	14.6	42.6	89.1	10.0	0.3	<0.1	5.7	15-33	22
	Dewaxed	19.5	32.5	88.4	9.9	0.4	<0.1	1.7	15-24	17
	Wax	2.2	88.4	84.7	14.6	0.1	<0.1	51.4	15-35	25
5	Feed	14.6	42.6	89.1	10.0	0.3	<0.1	5.7	15-33	22
	Dewaxed	19.3	28.8	89.4	9.1	0.3	<0.1	0.5	15-28	18
	Wax	2.7	71.2	87.0	12.8	0.3	<0.1	29.2	13-35	25
14	Feed	9.3	49.4	87.9	11.0	0.3	<0.1	6.2	15-34	26
	Dewaxed	15.3	33.6	89.4	10.2	0.4	<0.1	1.0	15-23	17
	Wax	4.1	62.0	87.4	12.7	0.2	<0.1	15.9	15-34	26
7	Feed	10.8	40.7	88.5	10.2	0.2	<0.1	2.3	15-31	20
	Dewaxed	12.5	38.4	88.9	10.0	0.2	<0.1	1.2	15-28	19
	Wax	4.6	57.6	87.6	11.9	0.1	<0.1	11.8	15-35	25
13	Feed	10.8	42.5	88.4	10.8	0.2	<0.1	1.9	15-31	20
	Dewaxed	14.1	35.2	89.1	10.2	0.3	<0.1	0.6	15-24	17
	Wax	5.0	55.4	88.0	12.4	0.2	<0.1	5.5	15-32	23
9	Feed	21.3	40.3	88.9	10.3	0.4	<0.1	6.2	16-31	16
	Dewaxed	22.9	35.6	88.6	10.3	0.4	<0.1	3.5	16-23	17
	Wax	0.3	91.3	84.7	15.3	0.1	<0.1	58.9	14-33	19
10	Feed	21.3	40.3	88.9	10.3	0.4	<0.1	6.2	16-31	16
	Dewaxed	24.6	32.5	88.6	9.8	0.5	<0.1	2.3	14-24	15
	Wax	1.9	90.9	84.4	15.4	0.1	<0.1	60.1	14-35	16
11	Feed	25.9	31.4	86.6	9.5	0.8	0.3	2.8	15-30	15
	Dewaxed	26.9	30.1	86.4	9.3	0.8	0.3	0.9	15-24	15
	Wax	0.8	86.7	84.6	14.5	0.1	<0.1	53.1	12-36	17
12	Feed	20.7	33.8	90.0	8.5	0.6	<0.1	-	-	-
	Dewaxed	23.8	26.4	90.2	8.0	0.4	<0.1	-	-	-
	Wax	2.8	76.9	87.0	12.7	0.4	<0.1	-	24->40	-

(a) carbon and hydrogen values probably only accurate to  $\pm 1\%$  absolute.

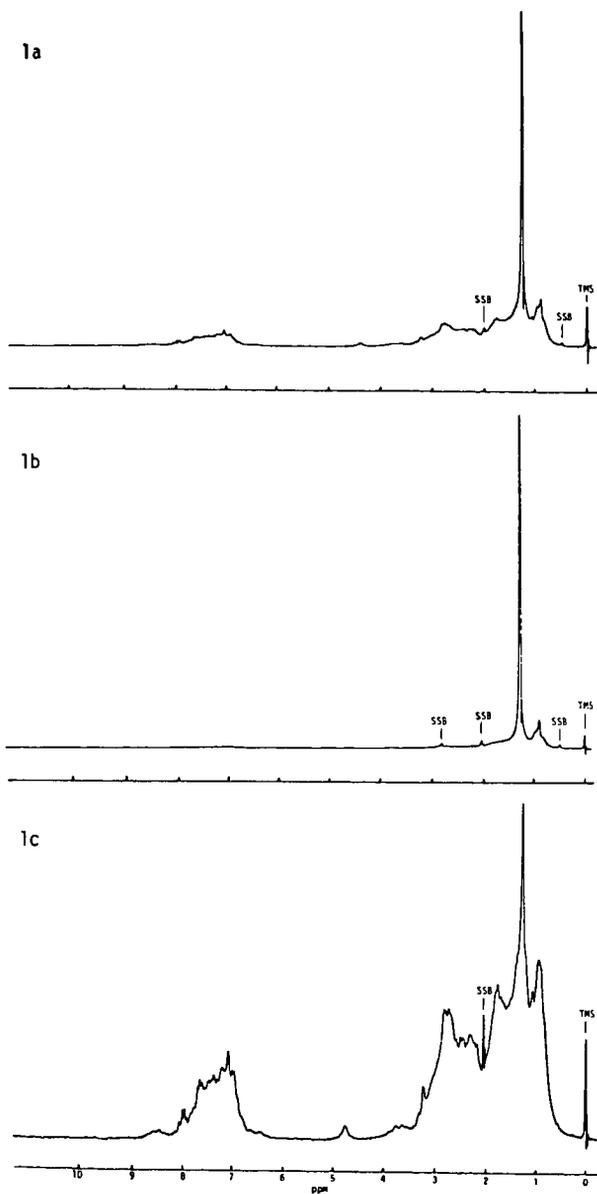


Figure 1.  $^1\text{H-NMR}$  spectra of samples from experiment 5. (a) feed oil, (b) wax, (c) dewaxed oil. TMS - tetramethylsilane internal reference, SSB - spinning side band.

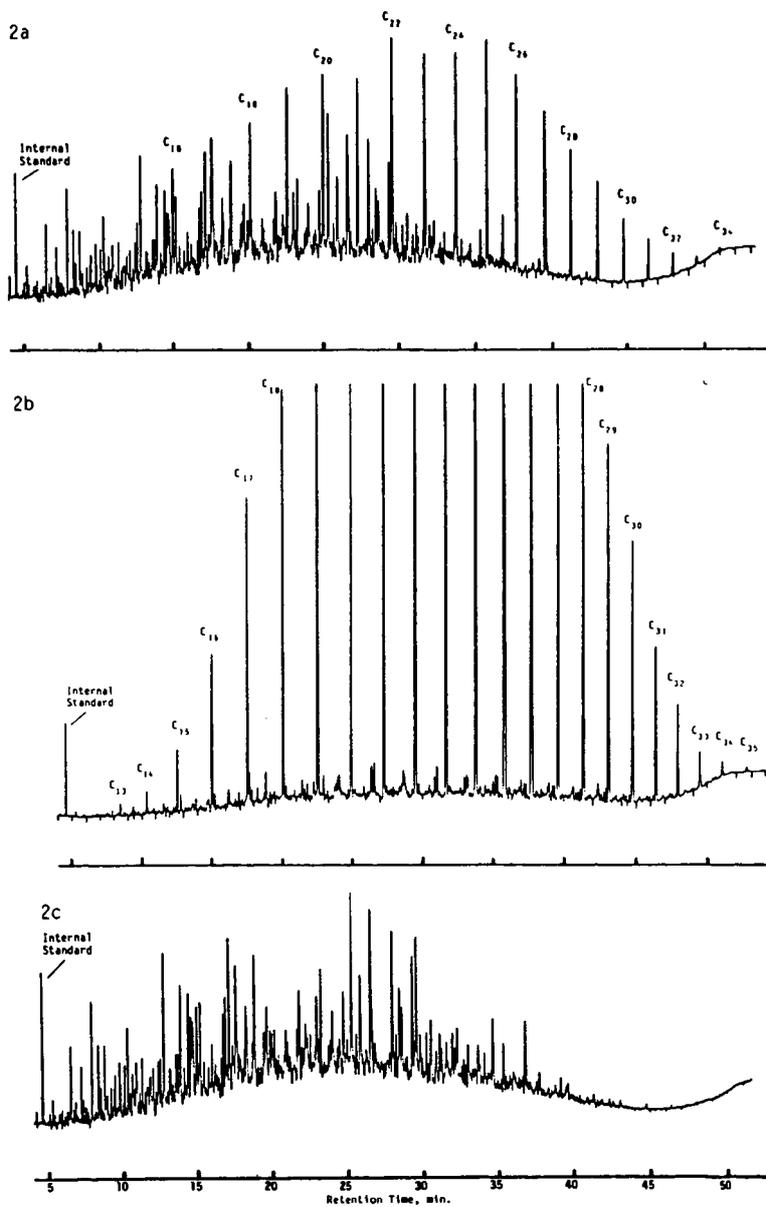


Figure 2. Gas chromatograms of samples from experiment 5. (a) feed oil, (b) wax, (c) dewaxed oil. (internal standard, n-decane)