

TITLE: Characteristics of Process Oils from HTI Coal/Plastics Co-Liquefaction Runs

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OBJECTIVE:

The objective of this project is to provide timely analytical support to DOE's liquefaction development effort. Specific objectives of the work reported here are:

- to determine the fate of the plastics feedstocks, relative to coal-only operation;
- to determine the conversion of the feedstocks;
- to determine the product streams to which the feedstocks are converted (bottoms vs. distillate);
- to determine interactions of feedstocks;
- to determine how use of plastics feedstocks affect product quality; and
- to determine to what degree property differences reflect feedstock differences vs. other (process) condition changes, such as unit operations, space velocity, and catalyst age.

ACCOMPLISHMENTS AND CONCLUSIONS:

Introduction

During a few operating periods of Run POC-2, HTI co-liquefied mixed plastics with coal, and tire rubber with coal. Although steady-state operation was not achieved during these brief test periods, the results indicated that a liquefaction plant could operate with these waste materials as feedstocks. CONSOL analyzed 65 process stream samples from coal-only and coal/waste portions of the run. Some results obtained from characterization of samples from Run POC-2 coal/plastics operation are:^{1,2}

1. Polystyrene (PS) products were identified and quantified in distillate product oil.
2. Incompletely converted high-density polyethylene (HDPE) was found as tetrahydrofuran (THF)-insoluble material in the ash-free-resid recycle stream. It was unclear to what extent this material was present in the ROSE bottoms stream. Unusual solubility behavior seems to be associated with HDPE-derived material in resid-containing streams. The broad implication is that HDPE was not completely converted in the liquefaction process.
3. The unusual presence of a product-oil sediment raised questions about the stability of the product oil.
5. Analytical issues were identified including how to identify and quantify HDPE, the appropriateness of coal liquefaction work-up procedures to

coal/plastics liquefaction, and how to measure the extent of plastics liquefaction.

Along with the analytical difficulties, the brevity of the coal/plastics liquefaction period in Run POC-2 prevented these issues from being resolved. To better evaluate these issues, Run CMSL-8 was performed at a smaller scale and over a longer period than Run POC-2. There were other differences too, such as reactor and temperature configuration and the feed coal used. However, the plant operated in solvent balance, which did not occur during the coal/plastics portion of Run POC-2. Solvent-balanced operation in Run CMSL-8 meant that samples, material balances, and performance results from Run CMSL-8 were representative of operation with the coal/plastics feedstocks. Coal/HDPE liquefaction was tested in Run CMSL-8, in addition to coal/mixed plastics liquefaction. The background and results from characterization of Run CMSL-8 process oil samples is presented below.

HTI Run CMSL-8 Background

A diagram of HTI's bench unit 227 as configured for Run CMSL-8 (also known as Run 227-85) is shown in Figure 1.⁵ CONSOL analyzed feed samples, and samples from sample points 4 through 7, representing recycle and product streams. The operating conditions and process performance summary for the run are given in Table 1.⁵ Operating performance was good early in the run, but as the run continued, the catalyst age increased, and the concentration of polyethylene in the feed was increased. The resid conversion decreased as the run progressed. Several adjustments were made to process conditions after period 16 to maintain performance and operability. Notable events were: the change from coal operation in period 6 to 75% coal and 25% mixed plastics prior to period 11; the increase in second-stage reactor temperature from 830 to 850 °F, an increase in first-stage space velocity from 30 to 40 lb dry feed/hr/ft³ reactor prior to period 16; the increase in mixed plastics concentration to 33%, decrease in space velocity from 40 to 30 lb dry feed/hr/ft³ reactor, and increase in dispersed Mo catalyst concentration from 100 to 200 ppm, prior to period 20; and, prior to period 22, the switch from 33% mixed plastics to 33% HDPE. Over the duration of the run, the supported catalyst reached an age of 966 lb dry feed/lb cat. Samples received as either period 22 or period 23 samples were considered to represent material balance period 22.

Analyses Performed

A brief description of the Run CMSL-8 samples and analyses conducted as CONSOL's baseline characterization is provided in Table 2. In this paper, the samples will be referred to by the abbreviations given in Table 2, e.g., SOH for the product oil, PFL for the recycle liquid, and PFC for the bottoms stream. The baseline analytical methods can be applied to many different kinds of samples, can be performed quickly, and have proven to be suitable for liquefaction process stream characterization. In addition to the routine laboratory analyses, non-routine characterization (such as FTIR characterization of certain samples) was performed, based on the Run POC-2 sample experience. Several samples were selected for specialized analyses, such as plasma desorption mass spectrometry (PDMS) and field ionization mass spectrometry (FIMS).

SOH Product Characteristics and Effects of On-line Hydrotreating

The separator overheads (SOHs) from periods 6 and 11 through 23 were consistently low in aromatic hydrogen and high in paraffinic hydrogen content (Figure 2). There was a small increase in paraffinic hydrogen from periods 16 to 20 to 23

coincident with increases in the HDPE concentration in the feed (8.75 to 11.5 to 33 wt % dry feed in those periods). There was no change in paraffinic hydrogen content from period 6 (coal-only) to period 11 (coal/mixed plastics). However, a substantially lower paraffinic hydrogen content was observed when the on-line hydrotreater was by-passed in period 9. This indicates that, because of extensive upgrading in the hydrotreater, the paraffinic hydrogen content of the SOH may be relatively insensitive to other process changes. The product oil (SOH) sample from period 9, in which the on-line hydrotreater was by-passed, is much poorer in quality than the SOHs produced with the hydrotreater in place. Differences included: medium brown in color vs. colorless, presence of a "coal liquid" odor, more aromatic, less paraffinic, and considerably higher phenolic - OH concentration (Figure 2). The effects of hydrotreating observed in this run were greater than those observed in Run POC-2.¹ This may be because the distillate hydrotreated in Run CMSL-8 is a thermal distillate, and the distillate of Run POC-2 came from a catalytic reactor.

Gas chromatography-mass spectrometry (GC-MS) total ion chromatograms of SOH samples (Figure 3) show that replacing a portion of the coal with mixed plastics (from period 6 to period 11) and the switch from mixed plastics to HDPE (from period 11 to period 22) increased the concentrations of n-paraffins in the SOHs, and shifted the n-paraffins to higher molecular weight. Thus, HDPE appears to be an important source of the n-paraffins in the SOHs produced after period 6. Two peaks corresponding to ethylbenzene and cumene (isopropylbenzene) are marked in Figure 3. These components are polystyrene (PS) liquefaction products. Cumene was not found in the coal-only period SOH, and ethylbenzene was present at about 1% concentration in the coal-only and coal/HDPE periods 6 and 23. ¹H-NMR results indicate that PS products persisted in the SOH product from the coal/HDPE period. In the NMR spectra of the SOHs, ethylbenzene features are nonexistent in the coal period SOH, quite prominent in the coal/mixed plastics period SOHs, and observable, but small, in the coal/HDPE period SOH.

The PS products were quantified by GC-MS and ¹H-NMR (Table 3). The area of the ethylbenzene and cumene peaks, as a percentage of the total ion chromatogram was used to estimate the concentration of these components in the SOHs. The alkylbenzene concentration of the SOHs was estimated (as ethylbenzene) by integration of the ¹H-NMR peak near 7.1 ppm. Based on these estimates, ethylbenzene and cumene constitute about 8-15 wt % of the coal/mixed plastic period SOHs (with the HTU in use), less than 1 wt % of the coal/HDPE period SOH, and about 2 wt % or less of the coal period SOH. When the hydrotreater was by-passed with the coal/mixed plastics feed, the concentration increased to about 15 to 23 wt % of the SOH. Approximately 50% of the PS fed to the process can be accounted for as these alkylbenzene products (with the hydrotreater operating).

HDPE in Recycle and Resid Samples

The PFLs from the coal/plastics periods 11, 16, 20, and 22 contained 15 to 30 wt % THF insolubles. These insolubles were tan with white specks early in the run and dark brown later in the run. The presence of THF insolubles in the PFL is a unique feature of coal/plastics processing. PFLs from coal-only operations (including period 6 of this run) typically contain little or no THF-insoluble material. The FTIR spectra of insolubles from coal/plastics periods 11 and 22 were similar and indicated that they are polyethylene-like material (Figure 4). PFL 850 °F distillation bottoms from two of three coal/mixed-plastics periods separated into two solid phases upon cooling; none of the other PFL resids behaved in this way. The two phases differed in physical characteristics and

color. Diffuse reflectance FTIR (Figure 4) was used to examine both phases of one of the resids. The upper phase appeared to be predominantly plastic derived (much of it PE), and the lower phase is predominantly coal derived. The spectrum of the upper brown phase indicated primarily aliphatic hydrocarbons with PE-like features. Aromatic hydrocarbon peaks also were significant, but no features indicated the presence of heteroatomic functional groups. The spectrum of the lower black phase showed more intense aromatic hydrocarbon peaks than did the upper phase, and a significant amount of aliphatic hydrocarbon in the lower phase, but no distinctive PE-like features. The spectrum of the lower phase also contains prominent peaks from heteroatomic functionality, perhaps N-H and O-H.

Samples of both PFL resid phases, along with other samples from Run CMSL-8, also were characterized by field-ionization mass spectrometry (FIMS) at SRI International.⁴⁻⁶ The pyrolysis profiles are shown in Figures 5a-b and the FIMS spectra in Figures 5c-h. Volatilization of each sample was nearly complete. The pyrolysis profiles show that HDPE pyrolyses to low molecular weight components at about 430 °C (Figure 5a), and that the THF-insoluble sample from the period 22 PFL is nearly all HDPE (Figure 5b). In the mass spectra, the HDPE pyrolysis products are lower in molecular weight and generally distinct from the coal-derived resid components (Figure 5c-h). These spectra also confirm the identification of the period 22 PFL THF insolubles as nearly pure HDPE (Figure 5c-d), and show that HDPE is present to varying degrees in the other samples from coal/plastic operating periods (Figure 5c-h). The plastic layer (Figure 5g) contains more HDPE than the corresponding coal layer (Figure 5h). Furthermore, the odd/even mass ratio is higher for the coal layer, suggesting that it contains more heteroatomic species. This is consistent with the FTIR results. A simple quantitation method was tried with the FIMS data (Table 4), and it appears to work fairly well (to the extent determinable at this stage). This method is compared with another method below.

In Table 5, the results of two methods for estimating the concentration of HDPE in liquefaction process streams are compared. In the first method, the THF-insoluble content of a PFL sample was measured and assumed to be unconverted HDPE. In the second method, a linear relationship between the HDPE concentration and the number average molecular weight (M_n) determined by FIMS was assumed. The methods for this limited sample set agree quite well. The FIMS approach offers the potential to quantify the amount of unconverted HDPE present in the bottoms (PFC) stream. This would allow a more accurate determination of HDPE conversion than is presently available.

Conversion of HDPE During Run CMSL-8

CONSOL and others have found indications that high-density polyethylene (HDPE) is less reactive than coal and other plastics feedstocks toward liquefaction at conventional liquefaction conditions. Since adequate conversion of HDPE is an important factor in the development of coal/plastics coprocessing, it is important to know the conversion of the HDPE during Run CMSL-8 and other coal/plastics coprocessing runs. Upper limits for both single-pass and overall conversions of HDPE during Run CMSL-8 were estimated (Table 6). It was assumed that: 1) the HTI unit was operating at steady-state, 2) that the PFL THF-insolubles are HDPE, and 3) that there was no unconverted HDPE in the PFC. During Run CMSL-8, PFL was both the recycle liquid (Figure 1) and a liquid product. Overall conversion is a measure of fresh HDPE which is not present as unconverted HDPE in the net products; in overall HDPE conversion, recycled HDPE is considered an internal stream and does not need to be explicitly accounted

for. The single-pass conversion of HDPE is a measure of the disappearance of both the recycled and fresh HDPE fed (recycled HDPE is explicitly accounted for).

The conversion calculations require material balance data for the HTI run periods,³ and an estimate of the amount of HDPE in the pressure-filter liquid (PFL). Details of the method used are provided elsewhere.⁷ These results (Table 6) represent an upper limit for conversion, because the HDPE concentration in the pressure-filter cake (PFC) product could not be determined. The overall conversion of HDPE ranged from 40-80% during the run (Table 6), lower than the 90-95% coal conversion and 80-85% resid conversion typically observed for coal liquefaction. The single-pass HDPE conversions averaged around 25%. Both overall and single-pass conversions were lowest during period 16, after an increase in second-stage reactor temperature and space velocity. Measures taken by HTI to improve performance after period 16, such as reducing the space velocity and doubling the dispersed Mo catalyst concentration, restored the conversions observed in period 11. The single-pass HDPE conversion in period 22 was much higher at about 50%. Measures that HTI took to maintain operability in that period of the run when HDPE and coal were fed seemed to provide the high single-pass conversion, and high overall conversion of HDPE.

Conclusions

The major conclusions from characterization of Run CMSL-8 samples are listed below.

- PS products are identifiable and quantifiable in the SOH distillate product from coal/mixed plastics co-liquefaction.
- HDPE appears to be an important source of n-paraffins in the SOHs from coal/plastics co-liquefaction.
- The SOH sample from period 9 in which the on-line hydrotreater was bypassed was much poorer in quality than the SOHs produced with the hydrotreater in operation.
- Identification of some PS and polyethylene terephthalate (PET) products in the SOHs may be masked by highly effective on-line hydrotreating. Addition of a hydrotreater feed sample point, or of more off-line hydrotreater reference periods may help in identification of plastics liquefaction components in the SOHs.
- Incompletely converted HDPE constituted 15 to 30 wt % of the PFL recycle streams, and was found as THF insolubles; virtually no THF insolubles were present in the coal-only period PFL.
- Phase separation in some PFL distillation resids indicates that HDPE products have complex phase behavior.
- HDPE conversions were estimated to be ca. 80 % overall, and ca. 25 % single-pass, and the conversions were responsive to changes in process conditions.
- THF insolubility is currently the best way to separate HDPE in liquid samples which contain no other solids.
- FTIR is useful for the identification of HDPE products.
- FIMS allows distinction of coal-derived material and HDPE-derived material in process stream samples. Quantification of HDPE seems possible using the FIMS technique, but additional development is needed.

PLANS:**CONSOL Support to DOE Coal/Plastics Co-Liquefaction Development**

We will do similar sample collection, distribution, and characterization work for future runs. Specialized analyses will supplement baseline characterization techniques. Additional analytical work, such as method development and evaluation, will be performed, as needed, to address key issues in coal/waste coprocessing. This will include evaluation of methods for characterization of plastic liquefaction products. Additional work could include development of alternative liquefaction work-up schemes to accommodate plastic components which are not amenable to conventional coal liquefaction work-up schemes. It is anticipated that at some future point, a distillate product oil from coal/waste co-liquefaction will be selected by DOE for a full set of product inspection tests. CONSOL will assist DOE in conducting these tests.

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**TABLE 1. RUN CONDITIONS AND PROCESS PERFORMANCE SUMMARY
FOR HTI RUN CMSL-8 (227-85)**

Condition	1	2	3	4	5
Period No.	6	11(b)	16	20	22
Hours of Run (End of Period)	144	264	384	480	528
wt % Plastics in Feed (a)	0	25	25	33	33
Stage 1 Cat. Age, lb Feed/lb Cat	252	457	711	884	966
Stage 1 Feed Space Velocity lb Feed/hr/ft ³ Reactor Vol.	32.2	29.6	39	30.1(c)	29.6
Oil/Solids Ratio	1.5	1.6	1.8	2.1	1.9
<u>Temperature, °F</u>					
Stage 1	810	810	810	810	810
Stage 2	830	830	850	850	850
HTU	715	715	715	715	715
Dispersed Mo Concentration, ppm of Dry Feed	100	100	100	200(c)	200
Total Material Recovery, % (Gross)	102.2	98.4	96.7	101.2	99.6
<u>Estimated Normalized Yields, wt % MAF Feed</u>					
C ₁ -C ₃ in Gases	11.35	9.14	9.02	7.41	5.17
C ₄ -C ₇ in Gases	4.81	3.27	3.41	3.17	3.27
IBP-350 °F	15.86	20.48	19.00	17.63	8.80
350-500 °F	17.99	12.57	8.59	11.16	7.60
500-650 °F	21.14	19.85	12.27	16.88	10.72
650-850 °F	10.18	11.84	15.18	11.54	14.24
850-975 °F	2.29	2.94	5.60	4.22	6.43
975 °F	4.74	10.53	17.15	19.67	33.83
Unconverted Feed	3.90	4.07	4.50	4.40	4.22
Water	9.04	7.34	6.90	5.92	4.85
COx	0.67	0.80	0.86	0.57	0.16
NH ₃	1.50	1.08	1.04	0.82	0.27
H ₂ S	3.98	2.98	2.84	2.52	2.24
Hydrogen Consumption	7.46	6.91	6.35	5.71	1.80
<u>Process Performance</u>					
Feed Conversion, wt % MAF Feed	96.10	95.90	95.50	95.60	95.80
975 °F ⁺ Conversion, wt % MAF Feed	91.40	85.40	78.40	75.90	62.00
C ₄ -975 °F Distillate, wt % MAF Feed	72.30	71.00	64.00	64.40	51.00
Hydrogen Efficiency, lb Dist/lb H ₂	9.69	10.27	10.08	11.28	28.33

Feeds:

Illinois No. 6 Crown II Mine coal, HDPE, Polystyrene, and PET

Back Pressure: 2500 psig

Catalysts:

K-1: Shell 317 Supported + Dispersed Sulfated Fe/Mo Oxide (100 ppm Mo)

K-2: Only Dispersed Sulfated Fe/Mo Oxide Introduced in Feed to K-1

Hydrotreater: HRI-6135 (Criterion C-411 Trilobe)

- (a) Conditions 2-4 used a 50/35/15 w/wt % ratio of HDPE/PS/PET; Condition 5 used HDPE alone w/coal.
- (b) Although not specifically listed here, in period 9 the on-line hydrotreater (HTU) was by-passed; otherwise conditions were the same as in period 11.
- (c) The total space velocity was reduced from 40 to 30 beginning in Period 18 as a result of operating difficulties at higher space velocities; the dispersed catalyst addition rate also was increased from 100 ppm Mo to 200 ppm Mo beginning in Period 19 to improve process performance.

**TABLE 2. CONSOL ANALYSES OF SAMPLES
FROM HTI COAL/PLASTICS CO-LIQUEFACTION RUN CMSL-8**

Sample Description; Name (Abbrev.); Sample Point	Periods	Technique & Information Sought (Refer to Key)
Product Distillate; Separator Overheads (SOH); SP-4	6,9,11,16,20,23	A,B,C
Recycle Oil; Pressure Filter Liquid (PFL); SP-6	6,11,16,20,22	A,E,F,G; THF Extract - B; THF Insols - D; Dist. - A,B,E; Resid - G; Resid THF Extract - A,B,H
Solid Residue; Pressure Filter Cake (PFC); SP-7	6,11,16,20,22	G; THF Extract -A,B,H

KEY TO TECHNIQUES AND INFORMATION SOUGHT:

- A** = ¹H-NMR for hydrogen distribution (7 classes), aromaticity (degree of hydrogenation), paraffinicity, hydrogen donors
- B** = FTIR in THF solution for phenolic -OH content
- C** = GC-MS for composition, carbon numbers of paraffins
- D** = special analyses
- E** = microautoclave test with standard coal for donor solvent quality
- F** = 850°F distillation for distillate content
- G** = THF extraction and ash for resid, ash and IOM content, for coal and resid conversion
- H** = solvent fractionation (oils, asphaltenes, preasphaltenes) for resid composition.

TABLE 3. QUANTITATION OF POLYSTYRENE LIQUEFACTION PRODUCTS IN SOH PRODUCT OILS FROM HTI RUN CML-8

Period	Analysis by GC-MS, Area % of SOH Total Ion Chromatogram			Analysis by ¹ H-NMR		wt % PS In Dry Feed	SOH Yield, wt % of Dry Feed	EB+IPB by GC-MS, as wt % of PS Fed	EB by ¹ H-NMR, as wt % of PS Fed
	Ethylbenzene, Ref. Time ca. 16.7 min.	Cumene (Isopropylbenzene), Ref. Time ca. 21.8 min.	Total, Area % (assumed to equal wt % of SOH)	As Ethylbenzene, wt % from Integration of Peak at 7.1 ppm					
6 (Coal)	0.55	-	0.55	-	0	50.06	- (b)	-	-
11 (Coal/Mixed Plastics)	6.53	1.91	8.4	8.8	8.75	47.65	45.7	47.9	
16 (Coal/Mixed Plastics)	8.32	3.38	11.7	15.1	8.75	33.52	44.8	57.8	
20 (Coal/Mixed Plastics)	6.94	2.01	9.0	12.1	11.55	43.28	33.7	45.3	
23 (Coal/HDPE)	1.38	0.29	1.7	3.4	0	25.29	(c)	(d)	
9 (Coal/Mixed Plastics - HTU Off-line)	13.52	4.03	17.6	15.4 (a)	8.75	35.02	70.4	61.6	

- (a) Assumed 11 wt % H in SOH for NMR estimate, other periods used wt % H reported by HTI.³
 (b) Represents 0.3 wt % of dry coal fed; equivalent to 3.1 wt % of PS fed in period 11.
 (c) Represents 0.6 wt % of dry coal fed; equivalent to 3.7 wt % of PS fed in period 20.
 (d) Represents 1.2 wt % of dry coal fed; equivalent to 7.4 wt % of PS fed in period 20.

TABLE 4. ESTIMATION OF HDPE CONCENTRATION WITH FIMS DATA

Sample	FIMS M_n , Da	FIMS M_w , Da	Estimate of wt % HDPE ^(a)	M_w/M_n
HDPE	154	558	100	3.6
PFL 22 THFI ^(b)	184	662	93	3.6
PFC 22	304	591	66	1.9
PFL 22	329	493	14 ^(c) (61 ^(d))	1.5
PFL 11 Resid Top Layer	404	627	44	1.6
PFL 11 Resid Bottom Layer	466	580	30	1.2
THF-Soluble Coal Resids ^{4,5}	600	710	-	1.2

Note: FIMS analyses were performed by R. Malhotra, at SRI International.

- (a) It was assumed that wt % HDPE is linearly related to M_n , and that $M_n = 154$ Da for 100% HDPE, and $M_n = 600$ Da for 100% coal resid.^{4,5}
- (b) THFI = THF insolubles.
- (c) It was assumed that $M_n = 515$ Da for the non-HDPE portion of the sample, rather than 600 Da, as in the other samples. This value was calculated from 21.1 wt % of the THF-soluble PFL as 850 °F distillate with an assumed $M_n = 200$ Da, and 78.9 wt % of the THF-soluble PFL as 850 °F resid with an assumed $M_n = 600$ Da.
- (d) Value if uncorrected for 850 °F distillate.

TABLE 5. COMPARISON OF METHODS TO ESTIMATE HDPE CONCENTRATION

Sample	Estimate of HDPE as THF Insolubles, wt % of Sample		Estimate of wt % HDPE, Based on M_n
	From Whole Sample	From Resid	
PFL 22 THFI	100	-	93
PFL 22	18.6	15.8	14
PFL 11 Resid Top Layer	-	-	44
PFL 11 Resid Bottom Layer	-	-	30
PFL 11	20.0	20.3	23 ^(a)

- (a) Calculated from wt % HDPE in each resid layer, the wt % of each layer in the resid (66.7 wt % top layer, 33.3 wt % bottom layer), and 59.3 wt % resid in the PFL.

TABLE 6. OVERALL AND SINGLE-PASS CONVERSIONS OF HDPE DURING HTI RUN CMSL-8

Period	wt % HDPE in PFL (a)	Overall Conversion, % (b)	Single-Pass Conversion, % (b)
Using THF insolubles in whole PFL as estimate for HDPE in PFL:			
11	20.0	80.7	23.2
16	30.4	44.6	9.1
20	14.5	71.6	26.2
22	18.6	73.9	49.5
Using THF insolubles in PFL resid as estimate for HDPE in PFL:			
11	20.3	80.5	22.9
16	37.4	32.0	5.9
20	16.5	67.7	22.9
22	15.8	77.8	53.3

(a) Assuming that THF insolubles in PFL are unconverted HDPE.

(b) Calculations are described in Reference 7.

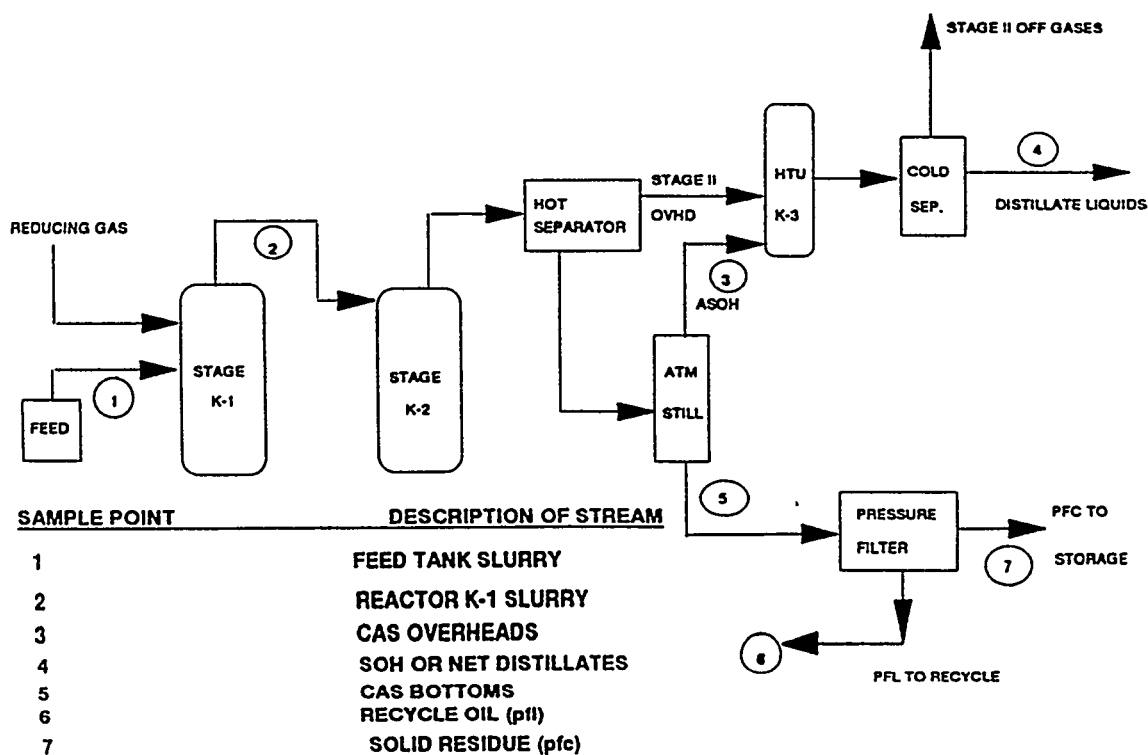


Figure 1. Simplified Plant Diagram for HTI Run CMSL-8.
(Source: Reference 3)

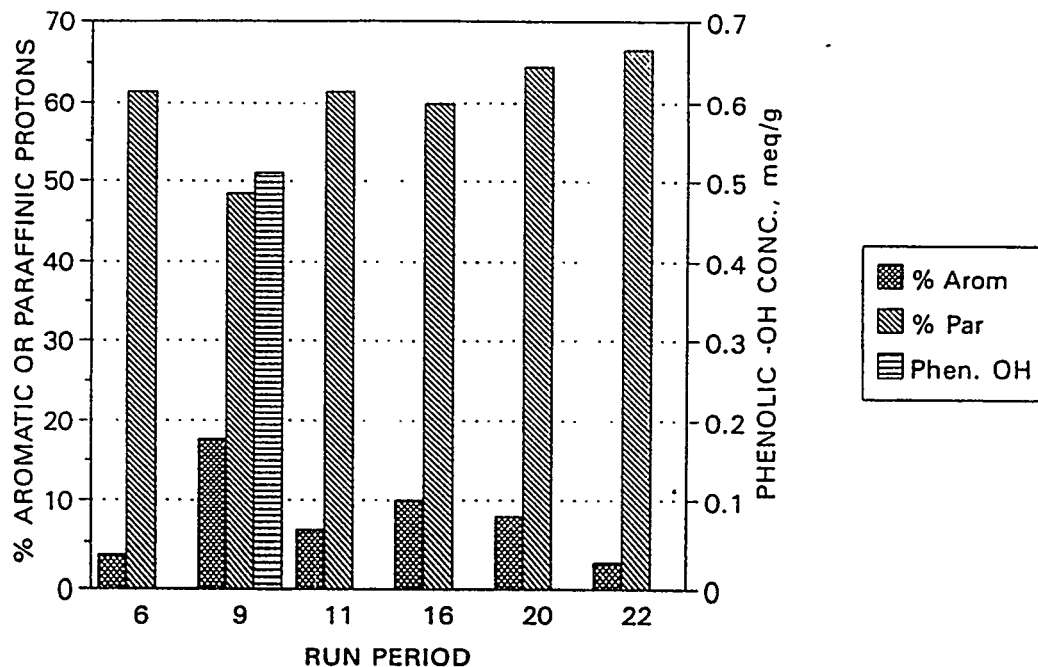


Figure 2. Characteristics of SOH Samples from Run CMSL-8.

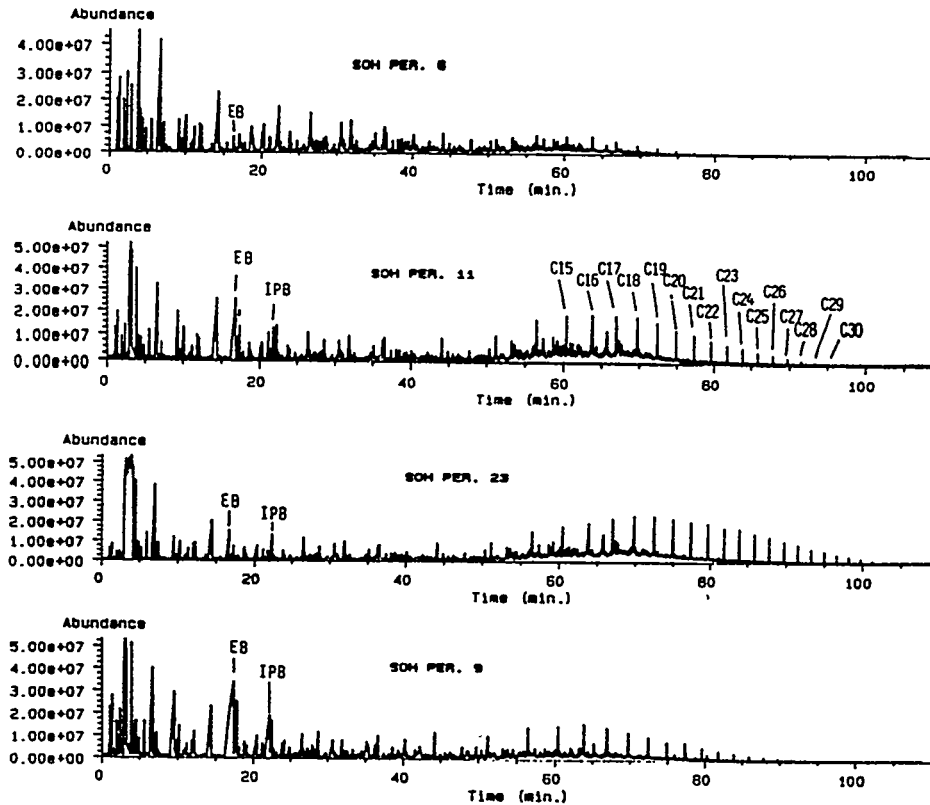


Figure 3. Gas Chromatography-Mass Spectrometry (GC-MS) Total Ion Chromatograms of Selected SOH Samples from Run CMSL-8.

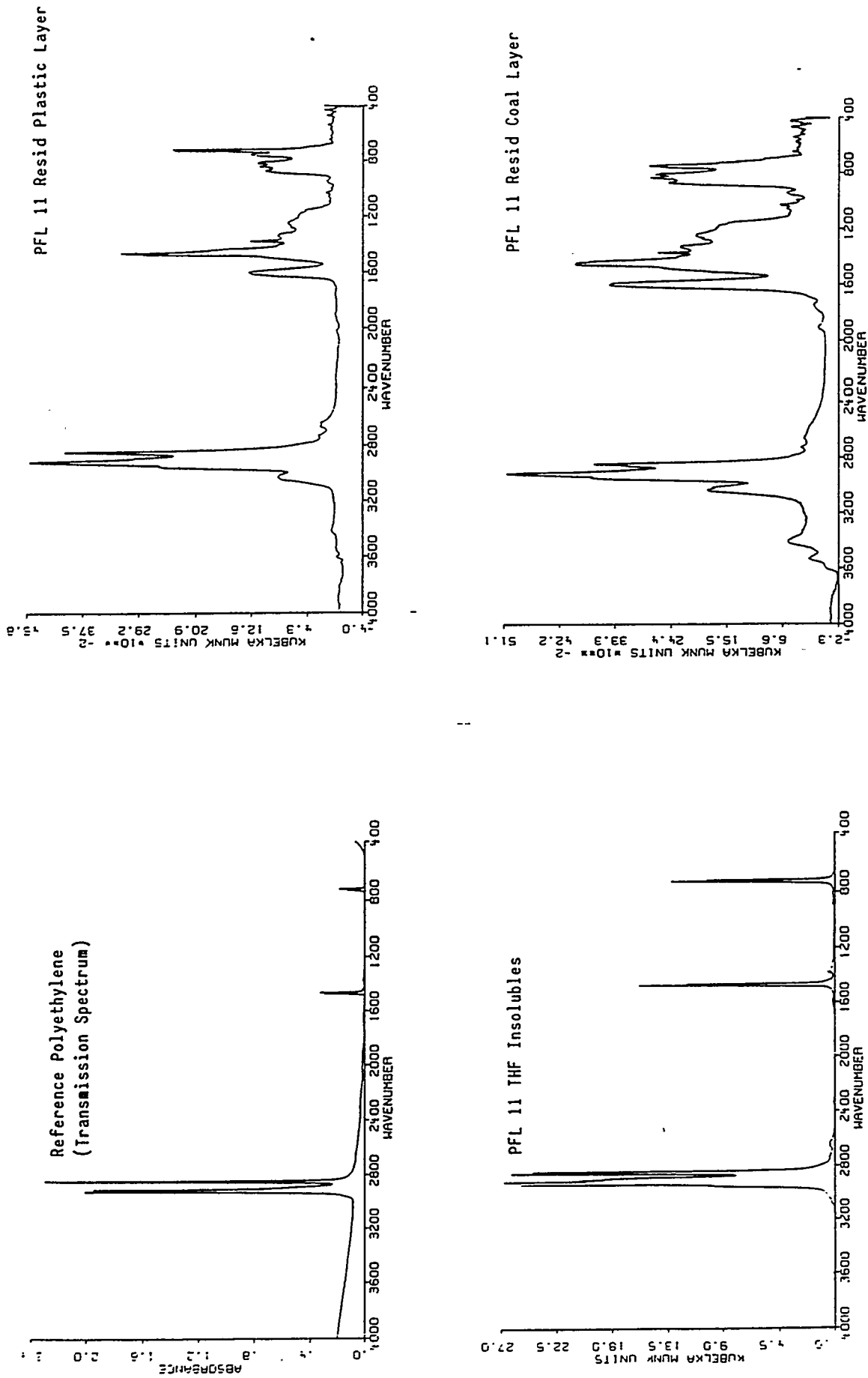
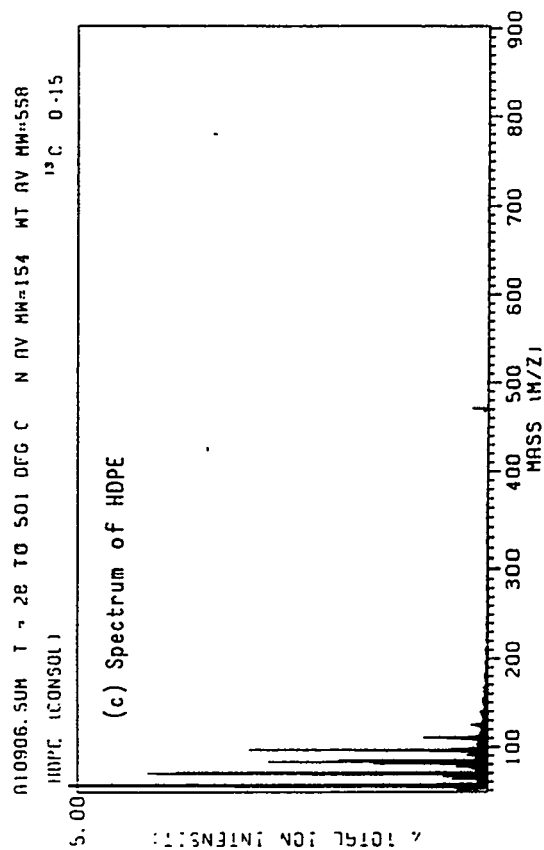
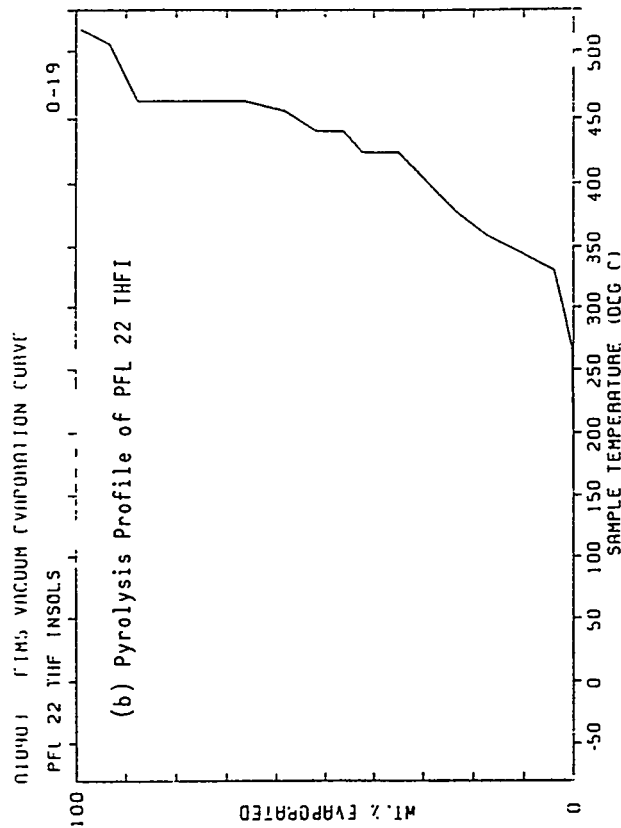
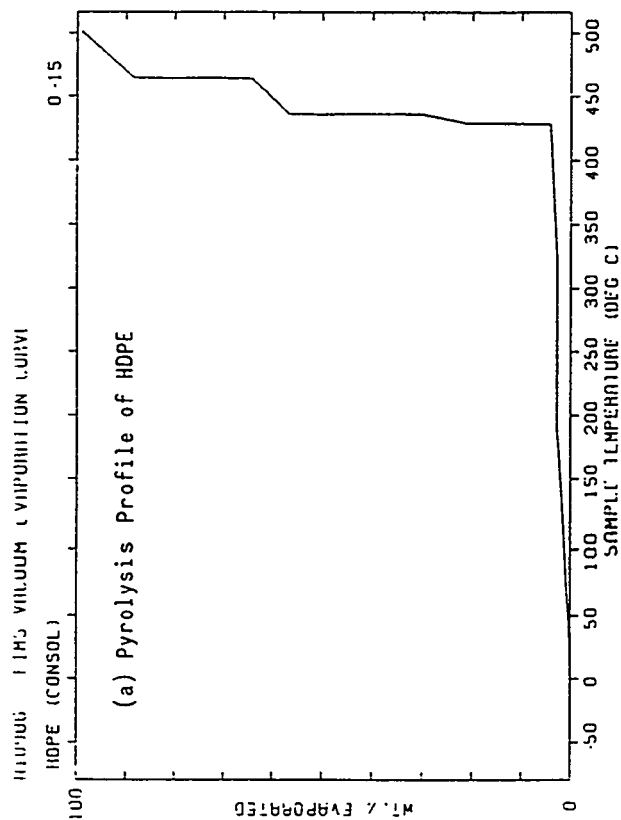


Figure 4. Fourier-Transform Infrared (FTIR) Spectra of Reference Polyethylene and Selected Run CMSL-8 Samples.



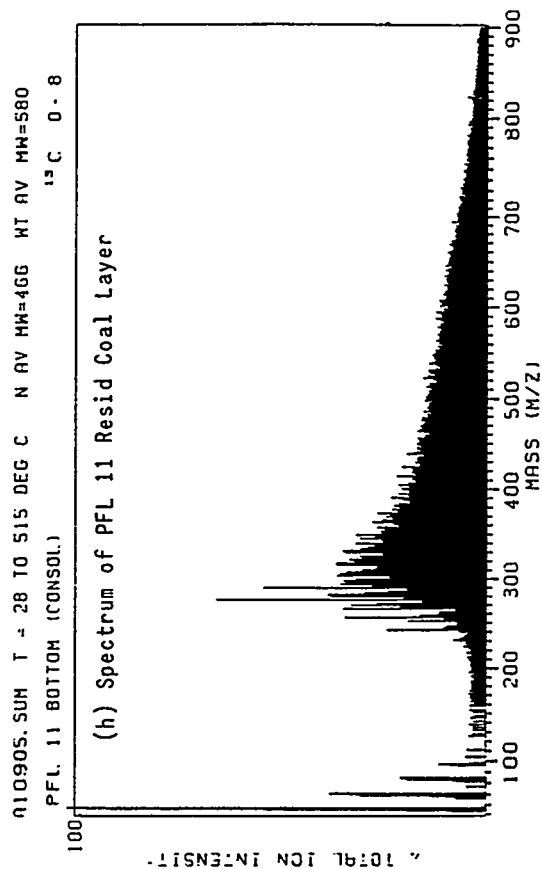
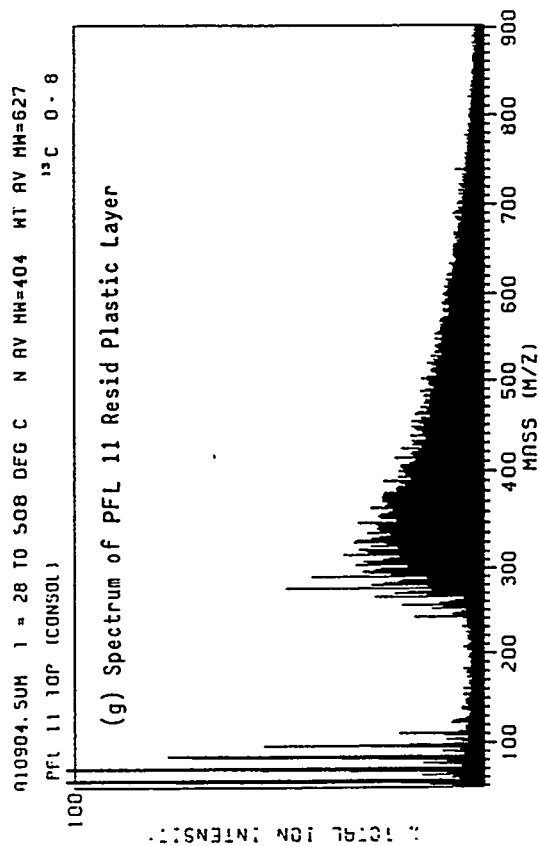
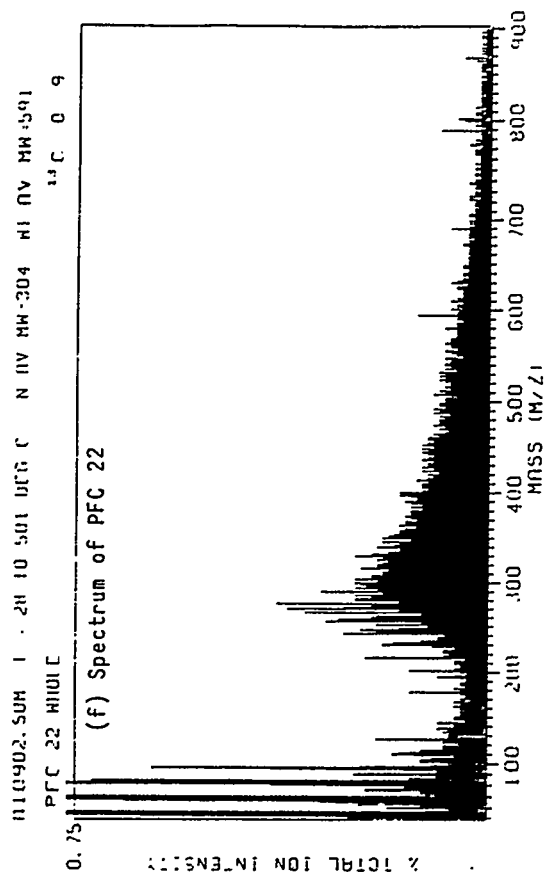
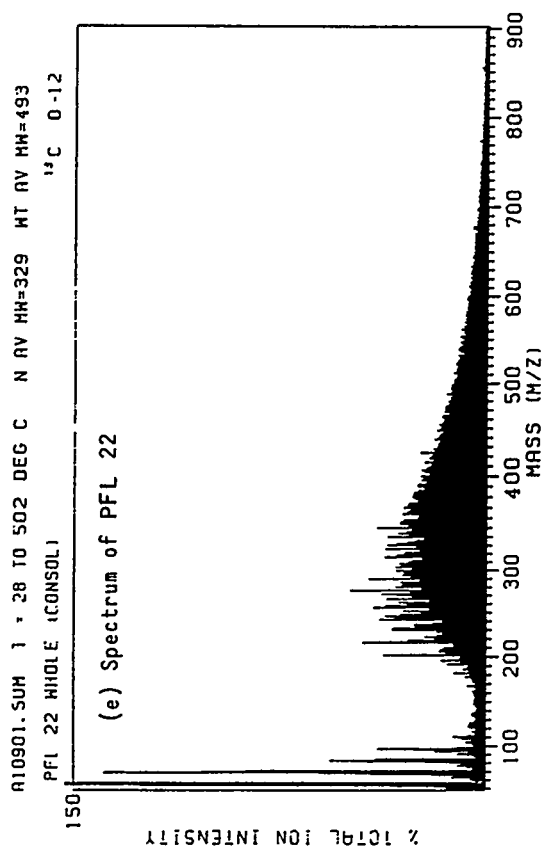


Figure 5 (Continued). Pyrolysis Profiles and Field-Ionization Mass Spectrometry (FIMS) Spectra of Selected Run CMSL-8 Samples.