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**DETERMINATION OF CORROSION RATES FOR STEEL ALLOYS IN
PROCESS SOLVENT**

Final Technical Report

**By
Edwin J. Latos**

**December 1, 1983
Date Published**

Work Performed Under Contract No. AC05-78OR03054

**UOP, Inc.
Signal UOP Research Center
Des Plaines, Illinois**

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I. ABSTRACT

The objectives of this program were to determine the corrosion rate, under static and dynamic conditions, of AISI 1010, 5 Cr-0.5 Mo, Type 304L and Type 316L steels in a SRC-I, V-178, coal-derived liquid at temperatures ranging from 550°F (288°C) to 700°F (371°C) and to analyze the after-test liquids for metal content, and physical and chemical properties to determine stability under these test conditions. In addition, the program included a study to determine the storage stability of the V-178 coal-derived liquid at 110°F (43.3°C) in air.

II. EXECUTIVE SUMMARY

Preliminary studies and analysis of the circulating streams in the SRC-I Wilsonville Advanced Coal Liquefaction Facility have indicated a corrosion potential for the preferred materials construction. The extent of this corrosion has not been quantitatively defined for certain areas and this program was designed to obtain these data. The test fluid selected was the coal-derived process solvent* from tank V-178 of the Wilsonville SRC-I pilot plant, and the metals were AISI 1010 and 5 Cr-0.5 Mo steels and Type 304L and Type 316L stainless steels. The tests were conducted over a temperature range of 550°F (288°C) to 700°F (371°C) and under both static and dynamic (6 ft sec⁻¹) conditions. Weight loss data yielded corrosion rates (mpy), and scanning electron microscope (SEM), energy dispersive X-ray microprobe (EDX), and metallographic techniques were used to examine corroded coupons. The after-test V-178 coal-derived liquid was analyzed with routine and instrumental techniques for metal content and indications of polymerization or degradation.

It was found that the corrosion rates of 1010 and 5 Cr-0.5 Mo were similar, independent of temperature (550-625°F) and approximately 17 mpy under static and 29 mpy under dynamic (6 ft sec⁻¹) conditions. Only general corrosion of the 1010 steel occurred, but significant pitting of the 5 Cr-0.5 Mo was observed. Analysis of the 5 Cr-0.5 Mo corrosion layer indicated the presence of $\text{Fe}_{(1-x)}\text{S}$ and Cr_7S_8 suspended in an amorphous coal-derived insoluble matrix. Iron content was higher in the after-test liquid, indicating dissolution of these metals. Analysis of after-test liquids also indicated the V-178 coal-derived liquid was stable under these test conditions (550-625°F).

* Contents of V-178 were tray 8 draw-off liquid from Vacuum Column, T-102. This liquid best approximates the process solvent that would be derived in the SRC-I Demonstration Plant.

The corrosion rates for Type 304L and Type 316L stainless steel were found to be approximately 4.7 and 2.1 mpy, respectively, independent of temperature (625-700°F) and static or dynamic conditions. General corrosion with only minor pitting was observed. The V-178 coal-derived liquid appeared to be stable at 625°F, but not at 700°F and there was an increase in iron, chromium, and nickel due to dissolution of the alloys.

It was found that the V-178 coal-derived liquids before and after 625°F static and dynamic tests in the presence of Type 304L were similar when analyzed with mass spectrometry, differential scanning calorimetry, chemiluminescence, and electron spin resonance. These data indicated the V-178 liquid was stable under these test conditions.

After storage for 30 days at 110°F (43.3°C) in air and in the dark, the V-178 coal-derived liquid was analyzed by the techniques listed above and found to have only minor degradation.

A glass liner, a machineable ceramic coupon holder, and a rotating cylinder operated as a coaxial viscometer were developed and utilized in this program to obtain the desired fluid velocity of 6 ft sec⁻¹. A new cleaning procedure using a dimethylformamide + toluene blend as a solvent to aid in removing the corrosion products was also developed for this project.

III. INTRODUCTION

The Solvent-Refined Coal (SRC-I) 6,000 ton-per-day (TPD) Demonstration Plant at Newman, Kentucky was designed to demonstrate the technical, environmental, and economic feasibility of the SRC-I process. The SRC-I plant is intended to allow an industry to be established in the private sector with no additional experimental or demonstration work. Since the plant is planned as the first module of a commercial plant, it must demonstrate process economic feasibility in the demonstration phase and it shall be designed in accordance with good industrial practice for a commercial plant, with the potential for operating 20 years at a cost-effective production rate.

The SRC-I process heats a slurry of pulverized coal, an internally generated solvent, and hydrogen at high pressures and temperatures without catalyst. Hydrocracking occurs in the preheater and in the dissolver units. The process stream is degassed, and the gas is scrubbed to remove sulfur, supplemented with hydrogen, and then recycled to the preheater. The processed coal stream is then treated by the Kerr-McGee critical solvent deashing unit, to separate the solids and liquids. Process solvent is obtained via distillation and recycled for slurring with feed coal. Other liquid products are also separated by fractionation. Figure 1 shows the present Wilsonville, Alabama Pilot Plant.

Preliminary study and analysis of the circulating streams in the SRC Wilsonville Pilot Plant have indicated a corrosion potential for preferred materials of construction for the demonstration plant in certain processing areas. Since the full extent of the corrosion problem has not been defined, it was necessary to obtain accurate corrosion rate data to facilitate proper mechanical equipment design and material selection and gain satisfactory

confidence in the long-term operability of the plant. However, this corrosion rate study was limited to corrosivity of potential materials of construction such as AISI 1010 steel, 5 Cr-0.5 Mo steel and Type 304L and 316L stainless steels in the SRC-I coal-derived process solvent obtained from tank V-178 in the pilot plant.

An excellent review of general corrosion problems at the Wilsonville plant was prepared by J. R. Keiser.⁽⁴⁾ Polythionic acid stress corrosion was concluded as a likely cause of some of the cracking found at a stainless steel Type 304-316L weld joint, and sulfide attack occurred on the carbon steel shell. Keiser⁽⁵⁾ also reported specific details regarding fractionation column corrosion. Corrosion occurred primarily in the lower half in the temperature range of 440-470°F (227-243°C) and appeared to be related to the chlorine content of the feed coal. Chemical analyses of deposits found on Type 316L stainless steel indicated predominately Ni_3S_4 . A new column made of a carbon steel vessel lined with Type 316L stainless steel was installed in 1979.

It should be recognized that this study was conducted on a single "process solvent". It is believed that the composition of the solvent is fundamental to its corrosivity. Since solvent composition is dictated by feed coal type and processing conditions, corrosivity will be run specific. A comparison of this process solvent with others that have been tested is provided in the Appendix, thereby relating this work to the current data base in this area.

IV. EXPERIMENTAL PROGRAM

A. Corrosion Rate Study

1. Corrosion Rate Program

The objective of this program was to determine the corrosion rate of AISI 1010 carbon steel, 5 Cr-0.5 Mo steel, and Types 304L and 316L stainless steels in the V-178 coal-derived SRC-I liquid under static and dynamic conditions from 550°F (288°C) to 700°F (371°C). AISI 1010 carbon steel was evaluated at 550°F (288°C) and 625°F (329°C). 5 Cr-0.5 Mo steel was evaluated at 550°F (288°C) and in the presence of Type 316L stainless steel at 625°F (329°C). Type 304L stainless steel was evaluated at 625°F (329°C) and at 700°F (371°C) in the presence of Type 316L stainless steel. Type 304L and Type 316L were evaluated at 700°F (371°C) under dynamic conditions only. Exposure for these tests was 100 hr.

This program also included, under static conditions only, exposure of AISI 1010 steel at 550°F (288°C) and of Type 304L stainless steel at 625°F (329°C) for 25, 50, and 200 hr. A separate exposure of a combination of Types 304L and 316L stainless steels was made for 400 hr at 625°F (329°C). These data were needed to show the effect of time of exposure on corrosion rate and to determine the development of passivating films.

The test coupons were 4.25 x 0.50 x 0.047 in. (10.8 x 1.27 x 0.119 cm) with a 120-grit finish. The surface roughness (Ra) was found to be 2.5 microns with a Taylor-Hobson Model Talysurf 10 surface profilometer. A 0.063-in. (0.165-cm) hole was drilled 1/2 in. from one end to facilitate handling. The coupons were

precleaned with ultrasonics and vapor-degreasing in a 1:1 blend of toluene and ethanol and stored under nitrogen.

The flow rate of the V-178 coal-derived liquid through the autoclave was 200 mL per hour. For dynamic tests, the liquid was stirred at a relative velocity of 6 ft sec⁻¹. Four coupons were present when only one type of metal was present. In tests in which two types of metal were present at the same time, two coupons of each metal were exposed.

Corrosion rate, in mils per year (mpy), was determined from weight loss data. In addition, corroded metal coupons were examined with visual, metallographic, and scanning electron microscope (SEM) techniques to assess general and pitting corrosion and the elements present (atomic number greater than sodium) in the corrosion layer. The procedure for preparing samples for metallographic examination is given in Table 1.

The after-test V-178 coal-derived liquids were analyzed for evidence of thermal degradation under these test conditions. All liquids exposed during the 100-hr test period were analyzed for heptane- and toluene-insolubles, acid number, dissolved oxygen, chloride content, and viscosity at 100°F (38°C). The heptane- and toluene-insoluble content is an indication of formation of higher (increase in insolubles) or lower (decrease in insolubles) molecular weight fragments. Acid number is an indication of oxidation. Viscosity changes are also indicative of degradation (decrease in viscosity) or polymerization (increase in viscosity) of the test fluid. Chloride content was monitored because it was reported that chlorides were involved in excessive corrosion by coal-derived liquids. In addition, the after-test fluids were analyzed for those metals originally present in the test coupons. The metal content in the original test coupons is given in Table 2.

2. V-178 Feedstock

The SRC-I derived process V-178 coal liquid is a heart cut from T-102, the vacuum fractionation column.* Illinois 6 Burning Star Coal was used to make this batch of V-178 on August 14, 1983. Typical analyses of Illinois 6 Burning Star Coal are shown in Table 3. This material was blended and delivered to UOP in nitrogen-blanketed 55-gal steel drums. Analysis of a composite sample taken from three drums is given in Table 4. A distillation with a hypercal column is shown in Table 5, boiling range distillation by GC is given in Table 6, and the estimated vapor pressure at various temperatures is provided in Table 7.

3. Test Plant Description

The test equipment used to investigate the corrosion rate of the SRC-I coal-derived liquid on test coupons is shown schematically in Figure 2. The test apparatus consists of a 2-liter, glass-lined stainless steel autoclave containing specially designed machineable ceramic inserts to support the four test coupons. For the dynamic test, the autoclave was equipped with a magnetically coupled stirrer and was operated as a rotating coaxial viscometer by rotating a solid ceramic bob 2.0-in in diameter at 2,000 rpm in order to produce a relative fluid velocity of 6 ft sec^{-1} (Figures 3 and 4).

The fluid flow system for the static and dynamic test apparatus consisted of a 15-gal, nitrogen-blanketed stainless steel reservoir to store the fluid before transferring it to an 8-liter, continuously weight monitored, stainless steel tank. The fluid flow rate for both the static and dynamic tests was controlled at 200 mL/hr by a positive displacement pump. The fluid entered at the bottom of the electrically heated autoclave via a ceramic dip tube. The

*This liquid best approximates the process solvent that would be derived in the SRC-I Demonstration Plant.

fluid level was maintained above the test coupons by a ceramic dip tube attached to the outlet line. A small amount of nitrogen, approximately 0.4 SCFH, entered the autoclave in the vapor space above the liquid level. The fluid and nitrogen gas exiting the autoclave were cooled, passed through a pressure control valve which maintained the operation at 500 psig, and collected in a nitrogen-blanketed product receiver. In each test, the coupons were maintained at a constant temperature and pressure and fluid turnover rate, and, in the case of the dynamic operations, at constant velocity for a prespecified test period.

After the test coupons were loaded into the autoclave, it was purged with nitrogen and pressure-tested for 1 hr at 600 psig. At the conclusion of the pressure test, nitrogen flow was initiated at 0.4 SCFH and the autoclave pressure was controlled at 500 psig. The fluid flow was initiated at 200 mL/hr and the autoclave brought to temperature. Fluid exiting the autoclave was diverted during this line-out phase to an off-test receiver. After the desired internal autoclave temperature was achieved, the test period was begun and the fluid exiting the autoclave was diverted to a pre-tared 9-gal receiver.

At the conclusion of the test period, the heaters were turned off and the liquid product receiver removed. During the cool-down phase, 600 mL of test fluid were pumped into the autoclave to flush the system. When the autoclave temperature was less than 38°C, the test coupons were removed and placed individually into nitrogen-purged jars.

4. Cleaning and Analysis of Corroded Metal Coupons

A review of the literature indicated that coal oil corrosion products on metal coupons were readily cleaned with acids, chemically or cathodically

inhibited. Since no special problems were anticipated, ASTM G-1 cleaning techniques were initially utilized. The coupons were scraped with brass wool, ultrasonically and solvent cleaned with a 1:1 toluene and ethanol blend, and then etched with acid. Stainless steels were etched with 10% nitric acid at 60°C and the 1010 and 5 Cr-0.5 Mo steels were etched with 50% HCl inhibited with Rodine 213 at 72°F (22°C). Examination with SEM indicated the presence of a significant corrosion deposit (Figure 5), indicating inadequate cleaning.

The cleaning process was modified to include extended etching in cathodically protected HCl, and extraction with sodium hypochlorite, 10% H₂SO₄, and carbon tetrachloride. Again, these cleaning techniques were not successful in removing all of the corrosion deposit. A private communication with Gulf Science and Technology Co. indicated similar problems. Methods found to be moderately, but not completely, successful at Gulf included freezing the coupon in liquid nitrogen, or heating, before brushing with wire brushes, blasting with glass beads until a uniform change in color was observed, and etching with HF to dissolve silica deposits. Sand blasting was used at Oak Ridge to clean coupons.

To permit a more judicious selection of a suitable cleaning solvent, the corrosion deposit on the 5 Cr-0.5 Mo steel coupon was analyzed with surface analysis techniques. Attenuated Infrared (ATIR) analyses did not produce an adsorption spectrum. A potassium bromide pellet of the deposit gave a very weak infrared spectrum suggesting the presence of water, inorganic species, and small quantities of CH₂ (or CH₃) at 2,850 and 2,910 cm⁻¹, the presence of phenol or phenol-nitrogen compounds at 1,255 cm⁻¹, water (or nitrogen compounds) at 1,620, 1,640, and 3450 cm⁻¹ and the presence of a metallic species by the band at 580

cm^{-1} (Figure 6). However, these spectra did not resemble any known spectra in the computer files. A photoacoustic spectrum (PAS) obtained with an EG&G Model 6001 photoacoustic spectrometer (see Figure 7) indicated only the presence of -C=C- due to absorption in the 250 to 300 cm^{-1} ultraviolet (UV) region. No other significant absorption bands were present.

Since the UV PAS indicated the presence of organic materials, the corrosion deposit was extracted with a 1:1 dimethylformamide (DMF)-toluene solvent and analyzed by fluorescence spectrophotometry. The spectra of coal-derived liquids after testing are shown in Figures 8 through 12. Both of the after-test liquid extracts from the 5 Cr-0.5 Mo (Figure 9) and 1010 steel (Figure 10) exhibit fluorescence very similar to that of the coal-derived liquid, with the exception of a strong, sharp emission at 620 nm, which does not appear in the coal-derived liquid spectrum. The synchronous fluorescence spectra for the DMF-toluene extracts from the Type 316L stainless steel coupons at 625°F (Figure 11) and 700°F (Figure 12) are substantially different from one another. Most of the fluorescence for the Type 316L steel at 625°F is observed between 300 and 450 nm, similar to both the 5 Cr-0.5 Mo and 1010 steels, whereas the 700°F sample fluorescence is primarily between 400 and 600 nm. The sharp fluorescence at 620 nm seen in the 5 Cr-0.5 Mo and 1010 steel samples is absent in both Type 316 stainless steel samples.

On the basis of the synchronous fluorescence data, it appears that the fluorescence observed for the DMF-toluene extracts for the four coupons is probably due to residual or adsorbed coal oil, which remained on or became a part of the corrosion deposit. The very low concentrations found, as well as the fact that no fluorescence was observed for chlorobenzene extract of the 1010 steel and

Type 316L steel at 700°F deposits, supports the concept that the observed fluorescence in the DMF-toluene extracts is due to residual or adsorbed coal oil or partial dissolution of the corrosion solids. The presence of a sharp emission at 620 nm in the synchronous fluorescence spectrum of the 1010 steel chlorobenzene extract suggests that this unknown compound or compounds could be present in the corrosion solids on the 1010 steel.

Probably the most interesting results obtained from the fluorescence data are that the Type 316L stainless steel at 700°F appears to adsorb preferentially what are believed to be the heavier, larger ring aromatics, which fluoresce at the longer wavelengths (400-600 nm), whereas the 1010 steel, 5 Cr-0.5 Mo steel, and the Type 316L stainless steel at 625°F retain only the lighter or more highly saturated aromatics, which absorb between 300 and 450 nm. There appears to be a very sharp temperature dependence on this selective adsorption.

Total Internal Reflection Laser Raman Spectroscopy (TIR-LRS) of the Type 304L original and corroded specimens, indicated complete absorption and the absence of amorphous carbon or graphite. Complete absorption would not occur with crystalline materials, and this spectra may be consistent with the apparent presence of high-molecular-weight polynuclear materials found with fluorescence.

EDX analysis of the corrosion deposit indicated the presence of the original elements in the base metal as well as sulfur. Spot patterns clearly showed the presence of sulfur, iron, and chromium in the corroded layer of 5 Cr-0.5 Mo at 550°F (Figure 13). The iron found in the corroded layer was less and the chromium deposit equal or perhaps slightly higher than that found in the base metal.

The corrosion layer from a 5 Cr-0.5 Mo coupon, exposed at 625°F (329°C) for

100 hr under dynamic conditions, was evaluated with mass spectrometry-gas chromatography (MS-GC) techniques. It was found that these corrosion products were not volatile under high vacuum at 300°C. Direct insertion probe mass spectrometric analysis of 100 µg of the material showed that water and carbon dioxide were the major gases produced when the sample was programmed from 86°F (30°C) to 572°F (300°C) at a rate of 86°F (30°C)/min. The mass spectra observed during heating showed that very low levels of refractory type aromatics and oxygenates were evolved, along with elemental sulfur (m/z 256 for S₈).

Total ionization (TI) plots of both a blank run and the sample run by direct insertion probe are shown in Figure 14. A comparison of the TI plot vs. selected ions for water and CO₂ is also shown. Water was immediately evolved while CO₂ maximized at 572°F (300°C).

A typical mass spectrum obtained during heating is also shown in Figure 15; all ions above m/z of 60 are enhanced by a factor of 5 and above m/z of 190 by a factor of 20. The more likely possibilities for some of the ions observed were:

m/z	64	S ₂ from disulfides, elemental sulfur, SO ₂
m/z	81	polyunsaturated cyclic hydrocarbons
m/z	95	polyunsaturated cyclic hydrocarbons, aromatic oxygenates
m/z	128	naphthylinic hydrocarbons
m/z	152	phenyl-phenyl, phenyl-CH-phenyl hydrocarbons
m/z	165	phenyl-CH-phenyl-CH-phenyl hydrocarbons
m/z	191-193	anthracenes and/or phenanthrenes and their oxygenated homologues
m/z	256	sulfur (S ₈)

These data do not identify specific compounds, although it was evident that organic material as well as sulfur is present in this corrosion deposit. These data are consistent with other analytical data.

The corrosion film of a 5 Cr-0.5 Mo coupon exposed at 625°F (329°C) for 100 hr was sprinkled onto double-stick tape secured to a glass slide and evaluated with X-ray diffraction techniques. A qualitative goniometer scan was run from 4 to 90°2θ with a counting time of 1 sec per step and a step size of 0.04°2θ. The scan was later rerun (from 20 to 90°2θ) with a counting time of 25 sec per step, which allows the minor peaks to be more easily detected. This pattern is shown in Figure 16. The major crystalline component identified was $\text{Fe}_{(1-x)}\text{S}$ (pyrrhotite), which possibly contains some Cr_7S_8 . Minor amounts of Cr° and Fe_3O_4 (magnetite) may also be present. The high background in the pattern may be due to the glass slide, tape, or some amorphous material present in the sample.

Although sufficient data were not available to define these corrosion deposits, it can be speculated that they were chromium sulfide (Cr_7S_8) and $\text{Fe}_{(1-x)}\text{S}$ imbedded in a cross-linked polymeric network. Although these sulfides are known to be soluble in acids, the sulfide-polymer network appeared to be insoluble in most solvents and acids, and it is not surprising that cleaning techniques always necessitated mechanical methods. Toluene, dimethylformamide (DMF), dimethylsulfoxide (DMSO), and methylene chloride were evaluated, and the most effective method for removal of the corrosion deposit was found to be brushing with a fine bristle brass brush while the coupon was immersed in a 1:1 mixture of toluene and DMF. Acid etching of the stainless steels with 10% nitric acid, at 140°F (60°C) for 20 min and the 1010 and 5 Cr-0.5 Mo steels in

50% hydrochloric acid inhibited with Rodine 213 at 72°F (22.2°C) was the final step. Samples cleaned with this procedure were free of deposits within the grinding grooves and were light to dark gray in appearance. It was noticed that cleaned coupons darkened with aging and had a small gain in weight.

5. Experimental Data

The corrosion rate, metal content in the corroded layer, and metal content in the after-test coal-derived oil for 1010 and 5 Cr-0.5 Mo steels, and Type 304L and Type 316L stainless steels under static and dynamic conditions at various temperatures are summarized in Table 8. The effect of increasing time of exposure of 1010 steel at 550°F (288°C) and of Type 304L stainless steel at 625°F (329°C) under static conditions is shown in Table 9. In addition, the table includes corrosion rate data for Type 316L at 625°F (329°C) after static exposure periods of 100 and 400 hr. Scanning Electron Micrographs (SEM) of the corroded layer and cross-section views, as is and etched, are shown in Figures 17-26. Results from examination of the cleaned metal coupons for pitting according to ASTM G-46 are given in Table 10.

The 1010 steel corrosion rate data showed that under static conditions there was a significant increase, from 18.3 to 30.2 mpy, when the temperature was increased from 550°F (288°C) to 625°F (329°C). On the other hand, no significant change in corrosion rate occurred with the same temperatures under dynamic test conditions (31.9 to 27.7 mpy). A large increase from 18.3 to 31.9 mpy at 550°F (288°C) occurred under dynamic conditions when compared with static conditions. Somewhat surprisingly, the static and dynamic corrosion rates at 625°F (329°C) are similar (30.2 and 27.7 mpy). The corrosion layer was black

and loosely adherent to the base metal, which made it difficult to obtain representative samples for SEM-EDX and metallographic analysis. The SEM-EDX data show that the corrosion layer contains iron and sulfur at an approximate atomic ratio equivalent to Fe:S, although no positive identification of FeS was made. A significant increase in the iron content in the after-test fluids also occurred, indicating dissolution of iron and a larger decrease in viscosity at 625°F (329°C) than observed with those metals containing chromium.

The microstructure of the original 1010 steel consists primarily of a fine-grained ferrite matrix with a highly spheroidized and dispersed carbide network (no pearlite found). Some sulfide-type inclusions were present. There was very little pitting and no change in microstructure in the corroded 1010 coupons; only general corrosion was observed. The SEM and metallographic photographs show a continuous amorphous film ranging in thickness from 5 to 10 microns.

The corrosion rate for 5 Cr-0.5 Mo in the V-178 coal-derived liquid was similar at 550°F (288°C) and 625°F (329°C), although it was significantly lower (18.8-14.2 mpy) under static than dynamic (29.2-26.1 mpy) conditions. This corrosion rate appeared to be similar to that of 1010 steel, although this black corrosion layer was tightly adherent to the base metal. The EDX data indicate the deposit was depleted in iron, enriched in chromium, and contained sulfur when compared to the base metal. Significantly more iron and chromium and less sulfur were present in the deposit formed at 550°F (288°C) than at 625°F (329°C). There was also a significant increase in the iron content in the after-test V-178 coal-derived liquid, and perhaps more iron after the 550°F (288°C) than the 625°F (329°) test. X-ray diffraction identified $\text{Fe}_{(1-x)}\text{S}$, although Cr_7S_8 was suspected, but not positively identified. Molybdenum could

not be detected in the presence of sulfur. Loss in viscosity in the after-test fluids was also greater with 1010 steel than with 5 Cr-0.5 Mo. The microstructure of the 5 Cr-0.5 Mo was of alloy carbides in a ferrite matrix; some sulfide type inclusions were present. The microstructure in the corroded coupons showed no change, although pitting was evident and increased with increasing temperature. The corrosion layer was approximately 5-7 microns thick and appeared to have an amorphous structure. The corrosion layer from the static 625°F (329°C) experiment contained a needle-like deposit (Figure 20).

The corrosion rate of Type 304L was 3.7 to 4.1 mpy and was independent of temperature (625-700°F) and static or dynamic conditions. At 750°F (399°C), corrosion rate significantly increased from 4 to 8 mpy. The thickness of the black corrosion layer increased with increasing temperature from 2-3 microns at 625°F (329°C) to 10-12 microns at 700°F (371°C) and to 15 microns at 750°F (399°C). The EDX elemental analysis of these films also showed a significant change in iron, chromium, nickel, and sulfur content with increasing temperature; iron content increased, chromium content decreased, the sulfur content increased, and nickel content decreased. The viscosity of the after-test fluids also decreased with increasing test temperatures. The iron and chromium contents decreased and the nickel and molybdenum contents remained relatively constant with increasing temperature in the after-test V-178 coal-derived liquid.

The original Type 304L had an annealed microstructure with equiaxed, twinned austenitic grains, and showed no apparent change in the corroded samples. Pitting was minor, and although it was observed in the coupon after the 100-hr static test, none was found in the coupon exposed for 400 hr. The

corroded film was tightly adherent to the base metal and had an amorphous-like structure with small particle size debris attached to the surface.

The corrosion rate of Type 316L stainless steel in the V-178 coal-derived liquid was low (0.7-3.2 mpy), and apparently independent of temperature (625-700°F) under static or dynamic conditions. The black corrosion layer was difficult to remove, and at 700°F (371°C) it ranged in thickness from 2 to 10 microns. The iron, chromium, nickel, and sulfur content of the corrosion layer was constant under these conditions, and at 700°F (371°C), it contained considerably more chromium and sulfur than the base metal. The iron, chromium, nickel, and molybdenum content in the after-test V-178 coal-derived liquid was similar to that found with Type 304L. The microstructure was annealed and consisted of equiaxed, twinned austenitic grains. This batch of Type 316L was not fully annealed since cold-flow lines were detectable, and there was a minor amount of precipitate along some of the austenitic grain boundaries. Pitting was insignificant, appearing to a minor degree in the 100-hr test at 700°F (371°C), but not in the 400-hr, 625°F (329°C) test coupon. The corrosion layer had an amorphous glassy-like appearance with fine particle debris adhering to the surface. Viscosity changes in the after-test fluids were similar to those obtained with Type 304L stainless steel.

To determine the degree of passivation occurring in the 1010 steel at 550°F and in Type 304L stainless steel at 625°F, the time of exposure was varied at 25, 50, 100 and 200 hr. These data as well as those obtained for Types 304L and 316L at 625°F after exposure for 400 hr are given in Table 9. It was apparent that equilibrium was attained rapidly at a constant corrosion rate of 18 mpy for 1010 steel at 550°F, and at approximately 1 mpy for Type 316L steel at 625°F. The

corrosion rate for Type 304L was constant at 2.6 to 4 mpy for the first 200 hr, and then decreased to approximately 1 mpy after 400 hr. The 6.3 mpy corrosion rate for the 100-hr test was obtained with coupons excessively etched with acid and was suspected of being abnormally high.

B. Stability Analyses of After-Test Fluids

1. Routine Analyses

The V-178 coal-derived liquids in the 100-hr test were monitored for evidence of degradation, oxidation, or polymerization by analysis for heptane- and toluene-insolubles, acid number, dissolved oxygen, and viscosity at 100°F (38°C). These data are shown in Table 11. There was no significant change in the chloride content or acid number, suggesting no detectable oxidation (no formation of acids) or sorption of chloride by the test metals. The dissolved oxygen content was also reasonably constant, indicating that the nitrogen pressure and sparging system successfully eliminated potential oxidation of the test liquid by traces of oxygen. There was a significant decrease in viscosity with increasing test temperature, although there was no apparent effect on the corrosion rate of the metal coupons. These data indicate an apparent depolymerization, degradation, or precipitation of the higher molecular weight fragments. There was no correlation between viscosity changes and corrosion rate data, and there was no apparent trend between corrosion rate and the changes in heptane- and toluene-insolubles, or type of metal under test. It appeared that these changes were within experimental error.

It can be concluded that V-178 coal-derived liquid is stable in the 550°F

(288°C) to 625°F (329°C) temperature range in the presence of the 1010, 5 Cr-0.5 Mo, Type 316L, and Type 304L test coupons. Degradation, as evidenced by viscosity changes, occurs at temperatures above 625°F.

2. Instrumental Analyses

Since routine chemical analyses for stability cannot detect subtle changes during the thermal treatment of fuels, the V-178 coal-derived liquid and test liquids after exposure at 625°F (329°C) in the presence of Type 304L under static and dynamic conditions were analyzed by several instrumental techniques capable of discerning small compositional changes. These included high-resolution mass spectrometry (MS) to identify any new species formed during the thermal treatment; electron spin resonance (ESR) to determine the nature and concentration of free radicals developed as a result of the thermal treatment; chemiluminescence (CL) to monitor the potential for oxidation; and high-pressure differential scanning calorimetry (DSC) to detect easily oxidizable materials.

a. Mass Spectrometry

To determine if test liquid components were changing during the corrosion studies, high-resolution mass spectrometry was used to directly analyze the composition of the V-178 coal-derived liquid in its original form and immediately after static and dynamic corrosion tests. A low-ionizing voltage technique was used to obtain a complete detailed compositional analysis of the volatile aromatic material.

A Kratos AEI high-resolution mass spectrometer (HRMS) Model MS-30 coupled with a Data General DS-55 on-line computer data acquisition system was used to obtain detailed molecular composition analysis of the total coal liquid sample.

Samples were quantitatively introduced into the MS through a specially designed, all-glass heated sampling system operated at 275°C to minimize thermal degradation of the samples while they were being analyzed.

In the application of mass spectra obtained at 10,000 resolution* to the analysis of the coal liquids, the following approach was used. The method for the analysis of the aromatic types present used a low-ionizing voltage (~11 eV). This method produced spectra composed mainly of molecular ion peaks that were accurately measured; the primary data were the carbon number distribution of the various aromatic types present.

The coal-derived liquids were analyzed shortly after receipt of the sample to minimize possible oxidation that might occur during long storage. Since the saturated hydrocarbon content is typically very low in these types of samples, analyses are reported on a saturates-free basis.

The original V-178 coal-derived liquid was analyzed and the detailed results obtained are shown in Tables 12-14. The HRMS analysis showed the coal-derived liquid to contain 87.78 wt.% aromatic hydrocarbons, 3.00 wt.% aromatic nitrogens, 9.18 wt.% aromatic oxygenates, and a trace (0.04 wt.%) of aromatic sulfurs. Since only semiquantitative information could be obtained on compounds having more than one heteroatom per molecule, these compounds were not included in the analysis. Only trace amounts of compounds having two oxygen atoms per molecule were determined, and the total concentration was estimated to be <0.2 wt.%. Since the concentration of sulfur-containing compounds was also low, only those

* This resolution is sufficient to separate most of the multiplet peaks except those containing sulfur present in the spectrum.

compounds detected where there was no overlap were reported separately. All others present were included in the corresponding aromatic hydrocarbon concentration.

The detailed HRMS analyses of the coal-derived liquid after the dynamic and static tests are shown in Tables 15-17 and in Tables 18-20, respectively. A comparison of the summaries of compound types is shown in Table 21. The concentrations of the aromatic oxygenates and aromatic nitrogenates increased after both the static and dynamic tests compared with the original coal-derived liquid. Comparison of the coal-derived liquids after static and dynamic testing revealed essentially no differences within experimental error. Based on the comparison of standard elemental analysis for oxygen in the original coal-liquid (1.19 ± 0.09 wt.%) with MS elemental analysis of each test liquid (shown in Table 21), it appeared that the increases seen in the aromatic oxygenate and nitrogenate concentrations might be due to increased solubility, thermal breakdown, and/or sampling. The trace dioxygenated compounds detected in the original coal-liquid were also seen in the liquids after testing.

b. Differential Scanning Calorimetry

High-pressure differential scanning calorimetry (DSC) is a thermal analytical method for measuring the heat of reaction. This is a direct display of the physiochemical processes that are actually occurring in the sample under the conditions of the experiment.

The purpose of this thermal study was to determine the oxidation onset temperature of the original coal-derived liquid sample compared with both the static and dynamic after-test samples. The oxidation method was adapted from the

method proposed by Cummings et al.⁽²⁾ at the National Bureau of Standards. The experimental procedure was to drop ~4 microliters of the coal-derived liquid into an aluminum DSC pan from a syringe. The open, uncovered pan was placed in the high-pressure DSC cell of a DuPont Model 990 Thermal Analyzer, the temperature scanned from 77 to 752°F (25 to 400°C) at 68°F (20°C)/min under an atmosphere of 520 psig air, and the oxidation onset temperature was determined by graphically extrapolating the maximum slope of the exotherm to the baseline. The samples were run immediately upon receipt.

Duplicate DSC runs under identical conditions were made, and the data are shown in Figures 27, 28, and 29. The exotherm, showing oxidation of the original V-178 coal-derived liquid, corresponded to an average temperature of 372°F (189°C). The exotherms for both the static and dynamic after-test samples corresponded to a temperature of 363°F (184°C). Oxidation of the original sample occurred at a slightly higher temperature than the static or dynamic test samples, but this was considered within experimental error. Therefore, it was concluded that no difference with respect to oxidation potential exists between the original and after-test samples.

c. Chemiluminescence

The chemiluminescence (CL) technique has been previously used in the study of food deterioration, plastics stability, lubrication oil stability and in determining the effectiveness of antioxidants. These studies have been reviewed by Mendenhall⁽⁶⁾ and Clark et al.⁽¹⁾. In a recent investigation, CL was used to characterize the changes that took place in a coal-derived liquid before and after hydrogenation and during storage⁽³⁾. It was reported that

rather large increases in CL were obtained after hydrogenation of the raw coal-derived liquid. In addition, the CL decreased as the level of hydrotreatment increased, and finally, the CL decreased with time of storage for all samples. Thus, it seemed clear that the CL technique was a potentially useful tool for the study of coal-derived liquids.

The instrument used in this study was a custom-built Battelle-Columbus chemiluminescence apparatus consisting of a photomultiplier, heater, and temperature controller. The output from the photomultiplier was fed to a Hewlett-Packard 5311B digital-analog converter, which was connected to a Fisher Recordall® Series 500 recorder. The photocurrent could be taken directly from the converter or read off the curve. A block diagram of the apparatus is shown in Figure 30.

All samples were stored in a refrigerator until ready for analysis. The runs were carried out by weighing 15 g of the liquid into a quartz cell having inlet and outlet ports so that air could be bubbled through the sample. The sample was blanketed with nitrogen while it was weighed and was kept under nitrogen until it was inserted into the heating block of the chemiluminescence apparatus.

The runs were carried out in the following manner. The block was heated to the desired temperature. The sample was then inserted into the receptacle in the block and kept under nitrogen purge while being heated to the desired temperature. When temperature equilibrium was reached, the gas manifold was switched to air flow (180 cc/min) and the zero time noted.

The first sample was of the original V-178 coal-derived liquid, the second was taken from a static run under 625°F test conditions, and the third was the

result of a 625°F dynamic run. Chemiluminescence runs were made on all samples at 212 and 302°F (100 and 150°C). As seen in Tables 22 and 23, there was essentially no difference in the results obtained from the various samples. Likewise, there was no temperature effect, since the same results were obtained at both 100 and 150°C. In Table 24, the results of a run made using only nitrogen are given, and are identical to those obtained with air.

These results are in sharp contrast to those obtained from the previous study on coal-derived liquids. As mentioned above, the photon counts were large, and thus the changes that occurred with a particular mode of treatment could be readily followed. Without a detailed analysis of the coal-derived liquid used in this study, one can only speculate as to why there was no observable CL response in this instance. For example, it was shown in the previous study that the removal of materials that were known oxidation inhibitors and quenchers led to large increases in the CL output. It is possible then, to conceive of a situation in which the presence of materials of this type could result in no observable chemiluminescence; alternatively, the coal-derived liquid used in this case might be very deficient in those species that interact with oxygen to show CL.

In summary, CL indicated no significant changes in potential to form free radicals in the original and after-test fluids.

d. Electron Spin Resonance

A Varian model 109E electron spin resonance spectrometer (ESR) was used to quantitate the relative amount of carbon radicals in the original sample, as well as in samples from static and dynamic tests after exposure at 625°F (329°C). The samples were placed in the ESR and irradiated with microwaves of a

fixed energy. The magnetic field surrounding the samples was then scanned from 3,200 to 3,300 gauss. In this range, the energy needed to change the spin of the electron in a carbon radical in relation to the magnetic field becomes equal to the energy of the microwaves. The electron then absorbs energy and changes its spin. The first derivative of this absorption peak is recorded and its height should be proportional to the concentration of carbon radicals in the sample.

The peak heights and calculated g-values are listed in Table 25. A definite drop in radical concentration occurred after the feed was processed through a dynamic or static corrosion test, although it is not known whether this loss of carbon radicals was due strictly to high temperature or to thermal effects in the presence of stainless steel as well. Another point of interest was the difference in radical concentration after static and dynamic tests. The static test was apparently more effective at reducing the carbon radical concentration than was the dynamic test.

In summary, mass spectrometry, differential scanning calorimetry, chemiluminescence, and electron spin resonance indicate that V-178 coal-derived liquid has relatively good stability at 625°F in the presence of Type 304L stainless steel at relatively short residence times.

C. Storage Stability Study at 110°F

The storage stability of the V-178 coal-derived liquid was evaluated in the 110°F (43.3°C) Storage Fuel Oil Stability Test. The preparation of samples was as follows:

A 1-gal sample was placed in a water bath sonifier held at 80°F (26.7°C) and sonified for 30 min while being continuously purged at 0.7 SCFH with nitrogen

(dried over high surface sodium) and simultaneously stirred at a rate just below that which would produce a vortex. After the mixing operation, the sample was transferred to an acetone-rinsed bottle which was dried while the nitrogen blanket was maintained inside it.

The sample was stored in a 500-mL centrifuge Pyrex bottle exposed to air for a period of 30 days at 110°F (43.3°C). The storage stability was measured by chemical and instrumental analyses, which included mass spectrometry (MS), differential scanning calorimetry (DSC), chemiluminescence (CL), and electron spin resonance (ESR).

The chemical analyses presented in Table 27 showed that aging of the coal-derived liquid resulted in an increase in the viscosity at 100°F, in toluene-insolubles and particulates. These changes indicated that some degradation of the V-178 coal-derived liquid took place during the storage period.

MS, DSC, and CL detected no change as a result of the 30-day storage (Tables 28 to 40 and Figures 31 and 32). ESR did detect a significant increase in free radical concentration (Table 41), which was consistent with the data obtained with the SRC-I coal-derived liquid product heated at 625°F.

V. CONCLUSIONS

The corrosion rates of AISI 1010 and 5 Cr-0.5 Mo steels at 550°F (288°C) and 625°F (329°C), and stainless steel Types 304L and 316L at 625°F (329°C) and 700°F (371°C) in a V-178 coal-derived liquid were determined under static and dynamic (6 ft sec⁻¹) conditions. The corrosion rates for 1010 and 5 Cr-0.5 Mo were similar (approximately 17 mpy static and 29 mpy dynamic) at 550°F (288°C) and 625°F (329°C), except that the corrosion rate for 1010 steel was significantly higher than 5 Cr-0.5 Mo at 625°F (329°C) under static conditions. The 1010 steel showed general corrosion only and 5 Cr-0.5 Mo had significant pitting that increased with increasing temperature. The corrosion rate for 1010 and 5 Cr-0.5 Mo was significantly higher under dynamic versus static conditions, and except for the conditions noted above, increasing the temperature from 550°F (288°C) to 625°F (329°C) had no measurable effect on the corrosion rate. The corrosion rate for 1010 steel was reasonably constant when evaluated after 25, 50, 100, and 200 hrs, indicating an equilibrium condition with an average corrosion rate of approximately 16.7 mpy at 550°F under static conditions. Iron and sulfur in the 1010, and iron, chromium, and sulfur in the 5 Cr-0.5 Mo were found in the corrosion layer with SEM-EDX. These data suggest iron and chromium sulfides, although positive identification was not made. The iron content of the after-test V-178 coal-derived liquid increased, indicating dissolution of the 1010 and 5 Cr-0.5 Mo.

The corrosion rates for Type 304L (4.7 mpy) and Type 316L (2.1 mpy) stainless steels were significantly lower than those for 1010 and 5 Cr-0.5 Mo steels. The corrosion rate for Type 304L was found to be slightly higher than that for Type 316L and independent of temperature (625-700°F) and static or

dynamic conditions. No significant pitting of Type 304L and Type 316L was found. Increasing the test period from 100 to 400 hr resulted in a small decrease in corrosion rate, although the statistical significance of this decrease was not apparent. Analysis of the Type 304L and Type 316L corrosion layers with EDX indicated the presence of iron, chromium, nickel, and sulfur. No specific identification of the actual compounds was made, although it could be speculated that these compounds are sulfides. At 625°F (329°C), iron content of the test fluid increased slightly, but at 700°F (371°C), iron content decreased slightly. These data suggest dissolution of the stainless steels and at 700°F (371°F) a precipitation of an iron compound. These corrosion rate data are summarized in Table 26.

Analysis of the after-test fluids indicated that the V-178 coal-derived liquid was stable at 550°F (288°C) and 625°F (329°C) under static and dynamic conditions in the presence of 1010, 5 Cr-0.5 Mo, Type 304L, and Type 316L [except in the presence of 1010 steel at 625°F (329°C)]. These conclusions were made on the basis of viscosity changes only. Any apparent changes in heptane- and toluene-insolubles, acid number, or chloride content were not statistically significant. It was also found that the V-178 coal-derived liquid after the Type 304L, 625°F (328°C) static and dynamic tests was not different from the original V-178 coal-derived liquid when analyzed with MS, DSC, CL, and ESR, indicating that the V-178 coal-derived liquid was stable under these test conditions. On the other hand, there was a significant decrease in viscosity at 700°F, revealing potential degradation or precipitation of the higher molecular species.

Two new experimental techniques were developed in this program. A glass and machineable ceramic coupon holder and a rotating cylinder operated as a coaxial

viscometer to obtain the desired fluid flow of 6 ft sec⁻¹ were designed. A new cleaning technique to remove the corrosion layer was also developed.

Based on several analyses of the corrosion layer from a 5.0 Cr-0.5 Mo coupon, it was concluded that the corrosion layer consisted of metal sulfides ($\text{Fe}_{(1-x)}\text{S} + \text{Cr}_7\text{S}_8$) suspended in a coal-derived liquid insoluble compound. The most efficient cleaning technique was found to be brushing the coupon while it was immersed in a 1:1 blend of dimethylformamide and toluene with a fine bristle brass brush before etching it with acid.

The storage stability of V-178 coal-derived liquid was evaluated in the 110°F (43.3°C) Storage Fuel Oil Stability Test. Analysis with MS, CL, and DSC of the coal-derived liquid after a 30-day storage period indicated no change. However, increases in free radical concentration (ESR), viscosity, toluene-insolubles, and particulates indicated some degradation.

VI. : REFERENCES

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4. Keiser, J. R., et al., "Analysis of Metallurgical Failures in Solvent Refined Coal Oil Pilot Plant at Wilsonville, Alabama" NACE Paper No. 149, Corrosion 1982.
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Table 1

Metallographic Sample Preparation for Edge Retention

1. Mounting: Vacuum impregnated Buehler's® Epoxide 6:1 ratio of resin to hardener. Let Cure for at least 24 hrs.
2. Rough Grinding: 120, 240, 320, 400, and 600 grit silicone carbide grinding papers.
3. Fine Grinding:
 - A. 6 micron diamond on Buehler's® texmet covered wheel using kerosene as a lubricant.
 - B. 3 micron silicone carbide lapping film using kerosene as a lubricant. The lapping film is supplied by 3M Co.
 - C. Back to 6 micron diamond on Buehler's® texmet covered wheel using kerosene as a lubricant.
4. Polishing:
 - A. 1 micron alumina on Buehler® Chemomet covered wheel. With the wheel stationary, the sample is rotated approximately 10 to 20 times around the wheel.
 - B. 0.05 micron alumina on Buehler's® microcloth. The sample is final polished for about 1 minute at low RPM, rotating the sample opposite to the direction of the wheel rotation.

Table 2

Chemical Composition of Test Coupons, Wt. %

<u>Metal</u>	<u>Fe</u>	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Mo</u>	<u>Ni</u>	<u>Cr</u>	<u>Cu</u>	<u>Other</u>
AISI 1010	99+	0.08	0.30	0.006	0.007						
5Cr-0.5Mo	93.5	0.15	0.44	0.010	0.021	0.19	0.49	0.23	4.95		
304L	70	0.03	1.85	0.021	0.002	0.57	0.20	8.70	18.46	0.11	0.08 Co, 0.015 N
316L	67.5	0.02	1.54	0.032	0.004	0.36	2.17	11.32	17.11		0.11 V

Table 3

Feed Coal Analyses - Special Product Workup Periods^(c)

Coal Mine Date, 1983 Run	-----Illinois 6----- -----Burning Star-----						
	29 May 243J	31 May 243K	13 Jun 243L	19 Jun 243M	10 Jul 244A	21 Jul 244B	28 Jul 244C
<u>Proximate analysis, wt %</u>							
Volatile matter	-	35.20	-	-	-	-	35.68
Fixed carbon	-	50.29	-	-	-	-	49.95
Ash	-	10.78	-	-	-	-	10.18
Moisture	-	3.73	-	-	-	-	4.19
<u>Ultimate analysis, wt %</u>							
Carbon	68.13	68.16	68.78	68.51	67.73	68.08	68.54
Hydrogen	4.63	4.35	4.54	4.50	4.54	4.48	4.42
Nitrogen	1.30	1.32	1.39	0.99	1.00	1.32	1.56
Sulfur	3.29	3.15	3.06	3.28	3.29	3.12	3.28
Chlorine	-	0.02	-	-	0.05	0.00	0.05
Ash	11.18	11.18	10.59	10.84	10.65	10.78	10.61
Oxygen (by difference)	11.47	11.82	11.64	11.88	12.74	12.16	11.54
H/C atomic ratio	0.82	0.77	0.79	0.79	0.80	0.79	0.77
Dry heating value, Btu/lb	-	12,384	-	-	-	-	12,662
<u>Sulfur forms, wt % (a)</u>							
Pyritic	-	1.26	-	1.18	1.28	1.22	1.15
Sulfate	-	0.07	-	0.07	0.05	0.03	0.12
Sulfide	-	<0.01	-	0.02	<0.01	<0.01	<0.01
Organic	-	1.98	-	2.22	2.19	2.20	2.08

Table 3 (continued)

Coal Mine Date, 1983 Run	-----Illinois 6----- -----Burning Star-----						
	29 May 243J	31 May 243K	13 Jun 243L	19 Jun 243M	10 Jul 244A	21 Jul 244B	28 Jul 244C
<u>Mineral analysis, wt % (Ignited basis)</u>							
Phos. pentoxide, P_2O_5	-	0.07	-	0.10	0.07	0.07	0.05
Silica, SiO_2	-	49.92	-	49.02	47.86	46.54	49.02
Ferric oxide, Fe_2O_3	-	17.39	-	17.26	17.70	18.18	18.20
Alumina, Al_2O_3	-	18.44	-	17.35	18.19	18.89	19.13
Titania, TiO_2	-	0.94	-	0.82	0.92	0.98	0.85
Lime, CaO	-	6.82	-	6.08	5.44	5.85	5.19
Magnesia, MgO	-	0.89	-	0.88	0.86	0.86	0.81
Sulfur trioxide, SO_3	-	2.82	-	6.19	6.17	4.60	4.20
Potassium oxide, K_2O	-	1.76	-	1.66	1.83	1.77	2.06
Sodium oxide Na_2O	-	0.39	-	0.40	0.58	0.57	0.45
Vanadium oxide, V_2O_5	-	0.05	-	0.04	0.04	0.04	0.04
Undetermined	-	0.51	-	0.20	0.34	1.65	0.00
<u>Trace metal analysis, ppm</u>							
Arsenic	-	5	-	2	1	2	-
<u>Reactivity, % THF conversion</u> (by the equilibrium test)(b)							
	79.0	77.6	76.4	77.9	75.5	76.5	79.7

(a) Data from Galbraith analyses.

(b) Thirty minutes at 750°F; 2:1 solvent-to-coal ratio. The solvent is 25% tetralin and 75% 1-methylnaphthalene.

(c) ICRC analyses.

Table 4

Analysis of V-178 Coal-Derived Process Liquid

API Gravity (60°F)	7.0
Specific Gravity (60°F)	1.022
Viscosity at 100°F (38.8°C)	
cSt	78.26
SUS	363
Acid Number, mg KOH/g	0.11
Heptane-Insoluble, wt.%	1.67
Toluene-Insoluble, wt.%	0.10
Elemental Analysis, wt.%	
Carbon	89.40
Hydrogen	9.80
Oxygen	1.19
Sulfur	0.27
Nitrogen	0.27
Chloride, ppm	71
Dissolved Metals, ppm	
Chromium	1.7
Nickel	0.3
Molybdenum	0.1
Iron	19.0
Phenol, Total Oxygenates, wt.%	
UV	0.8
Infrared	0.8

Table 5

High Temperature Distillation
of V-178 Coal-Derived Process Liquid

<u>O.H.</u> <u>°C</u>	<u>Press.</u> <u>mm</u>	<u>Corr.</u> <u>°C</u>	<u>Still</u> <u>°C</u>	<u>Accum.</u> <u>mL</u>	<u>L.V.</u> <u>%</u>
111	7.8	248	243	INIT.	-
120	7.8	259	245	5	2.2
132	7.8	274	246	10	4.4
140	7.8	284	247	15	6.7
147	7.8	292	250	20	8.9
153	7.8	299	252	25	11.1
159	7.8	307	254	30	13.3
163.5	7.8	312	256	35	15.6
168	7.8	318	258	40	17.8
172	7.8	322	262	45	20.0
176.5	7.8	328	262	50	22.2
181	7.8	333	263	55	24.4
185	7.8	338	266	60	26.7
185.5	6.8	343	267	65	28.9
190	6.8	348	270	70	31.1
190.5	6.0	352	270	75	33.3
190.5	5.0	357	273	80	35.6
194	5.0	361	273	85	37.9
196	4.6	366	274	90	40.0
200	4.6	370	278	95	42.2
203.5	4.6	374	280	100	44.4
206	4.3	379	290	105	46.7
209.5	4.3	384	293	110	48.9
212.5	4.3	387	296	115	51.1
215.5	4.2	391	298	120	53.3
218.5	4.2	395	301	125	55.6
				<u>100</u>	<u>44.4</u>
				225	100.0

Table 6

Boiling Range Distillation by GC,
wt.% of V-178 Coal-Derived Process Liquid,
UOP Method 194-NB

<u>% OFF</u>	<u>DEG F</u>
IBP	445
5	520
10	547
15	574
20	599
25	620
30	639
35	655
40	675
45	692
50	710
55	728
60	745
65	763
70	780
75	797
80	816
85	838
90	865
95	899
FBP	1043

Table 7

Estimated Vapor Pressure on V-178
Coal-Derived Process Liquid

<u>Temp., °F</u>	<u>Pressure, psia</u>
550	73
625	135
700	230
750	340

Table 8

Corrosion Rate, and Analysis of Corrosion Deposits and After-test
Coal-Derived Liquids After 100 Hour Tests

Metal	Type of Test	Temp., °F	Corrosion Rate, mpy	Metal Content, Corrosion Layer, Wt.%				Metal Content in After-test Oil, ppm			
				Fe	Cr	Ni	S	Fe	Cr	Ni	Mo
1010	- - - -Original-	- - - -	- - - -	100	-	-	-	19	1.7	0.3	0.1
"	Static	550	18.3	65.7	-	-	33.1	32	-	-	-
"	Dynamic	550	31.9	73.0	-	-	26.0	48	-	-	-
"	Static	625	30.2	96.6	-	-	0.3	29	-	-	-
1010	Dynamic	625	27.7	87.6	-	-	11.2	37	-	-	-
5Cr-0.5Mo	- - - -Original	- - - -	- - - -	94.5	5.0	-	-	19	1.7	0.3	0.1
"	Static	550	18.8	74.0	20.0	1.0	2.0	37	1.4	-	<0.1
"	Dynamic	550	29.2	65.0	20.0	1.0	12.0	45	2.0	-	0.4
"	Static	625	14.2	58.5	9.0	0.5	39.5	23	1.8	-	0.3
5Cr-0.5Mo	Dynamic	625	26.1	51.8	8.5	1.4	36.0	32	2.1	-	0.1
Type 304L	- - - -Original	- - - -	- - - -	70.0	18.5	8.7	-	19	1.7	0.3	0.1
"	Static	625	6.3	47.0	33.0	11.0	6.0	23	1.7	0.3	-
"	Dynamic	625	4.1	51.5	31.3	10.1	2.5	22	2.1	0.3	-
"	Static	700	4.5	-	-	-	-	11	1.2	0.2	0.2
"	Dynamic	700	4.1	-	-	-	-	14	1.3	0.4	0.2
"	Dynamic	700	3.7	58.3	12.1	7.3	21.5	-	-	-	-
"	Dynamic*	700	7	-	-	-	-	-	-	-	-
Type 304L	Static	750	8.0	64.6	2.3	4.5	27.8	6	1.1	0.3	-
Type 316L	- - - -Original	- - - -	- - - -	67.5	17.1	11.3	-	19	1.7	0.3	0.1
"	Static	625	1.5	50.6	21.6	11.8	9.3	23	1.8	-	0.3
"	Dynamic	625	0.7	56.9	22.0	9.9	3.9	32	2.1	-	0.1
"	Static	700	3.2	56.0	25.0	12.0	5.0	11	1.2	0.2	0.2
"	Dynamic	700	3.0	54	31.0	9.0	4.0	14	1.3	0.4	0.2
"	Dynamic	700	2.9	49.0	27.0	13.4	9.0	17	1.4	-	0.5
Type 316L	Dynamic	700	*	-	-	-	-	-	-	-	-

*Run 10 aborted due to breakage of ceramic protective layer.

Table 9

Effect of Time of Exposure on Corrosion Rate of
1010 Steel and Types 304L and 316L Stainless Steels

<u>Metal</u>	<u>Temp., °F</u>	<u>Corrosion Rate, mpy, After Various Hrs. of Exposure</u>					
		<u>Hrs. →</u>	<u>25</u>	<u>50</u>	<u>100</u>	<u>200</u>	<u>400</u>
1010	550		17.0	17.7	18.3	14.0	-
304L	625		4.2	2.6	6.3	3.9	0.83
316L	625		-	-	1.5	-	0.86

Table 10

Pitting and Intergranular Corrosion According to ASTM G-46

Type of Metal	Temp., °F	Type of Test	Pitting		Depth Ave, μ	Depth Max, μ
			Area, %	Ave. Size		
1010	550	Static	20		3	5
"	550	Dynamic	-	-	-	<3
"	625	Static	-	-	-	<5
"	625	Dynamic			General Corrosion	
5Cr-0.5Mo	550	Static	10	3	2	5
"	550	Static	60	5	3	8
"	550	Dynamic	30	4	2	6
304L	625	Static	30	2-3	3	5
	625	Dynamic	10	2	2	4
	700	Dynamic	30	5	3	8
	700	Dynamic	30	5	2	5
	750	Static	40	10	5	10*
	625	Static (400 hrs)	25	10	2	4
316L	700	Static	10	10-15	2	5
	700	Dynamic	<5	10	1-2	3
	700	Dynamic	10	10	2	5
	700	Dynamic	20	10	2	5
	625	Static (400 hrs)	20	5	2	3

* Intergranular Depth of 10 μ

Table 11

Select Properties of V-178 Coal-Derived Liquid After 100 Hour Test

Metal	Temp., °F	Insolubles		Acid No., mg KOH/g	Chloride, ppm	Dissolved Oxygen, ppm	Viscosity at 100°F Centistok
		Heptane, wt.%	Toluene, wt.%				
Static							
Original	-	1.67	0.10	0.11	71	<1	78.3
1010	550	1.07	0.10	0.11	64		
1010	625	1.99	0.09	0.10	72	0.4	70.1
5 Cr-0.5 Mo	550	2.06	0.13	0.10	38	0.3	78.6
5 Cr-0.5 Mo	550	2.35	0.10	0.10	70	0.5	
5 Cr-0.5 Mo + 316L	625	2.53	0.04	0.08	41	0.4	81.2
304L	625	1.62	0.04	0.04	65	0.9	77.6
304L + 316L	700	2.35	0.02	0.10	59	0.3	69.8
304L	750	0.93	0.03	0.08	57	0.8	48.0
Dynamic							
1010	550	1.24	0.02	0.11	65	0.4	77.0
1010	625	2.19	0.13		59	0.5	70.4
5 Cr-0.5 Mo	550	1.69	0.05	0.12	62	0.4	81.8
5 Cr-0.5 Mo + 316L	625	0.88	0.05	0.11	49	0.4	81.0
304L	625	1.56	0.12	0.05	69	0.8	76.5
304L + 316	700	1.76	0.11	0.07	57	0.3	70.8
304L	700	1.34	0.01	0.11	66	0.5	69.6
316L	700	1.65		0.11	72	0.4	69.2

Table 12
Analysis by High Resolution Mass Spectrometry
V-178 Coal-Derived Liquid Original

Sample Analysis Summary

<u>Compound Types, C_nH_{2n-z}(X)</u>	<u>Avg. C No.</u>	<u>Avg. Mol. Wt.</u>	<u>Avg. z No.</u>	<u>Avg. Rings</u>	<u>Wt. %</u>
Saturated Hydrocarbons (P+N)			NOT DETERMINED		
Aromatic Hydrocarbons	20.58	269.76	18.60	3.00	87.78
Aromatic Nitrogens	18.90	260.11	18.64	3.23	3.00
Aromatic Oxygens	17.09	240.29	15.11	2.24	9.18
Aromatic Sulfurs	13.49	203.07	15.09	2.66	0.04
TOTAL	20.21	266.74	18.28	2.94	100.00

<u>Distribution of Aromatic Rings, C_nH_{2n-z}(X), Wt. %</u>	<u>C-H</u>	<u>C-H-N*</u>	<u>C-H-O*</u>	<u>C-H-S*</u>	<u>Total*</u>
1 Ring Aromatics	12.78	0.0	3.10	0.01	15.89
2 Ring Aromatics	26.17	0.57	2.98	0.02	29.74
3 Ring Aromatics	16.76	0.84	1.42	0.0	19.02
4 Ring Aromatics	16.94	1.25	1.23	0.00	19.42
5 Ring Aromatics	11.14	0.34	0.37	0.01	11.86
6 Ring Aromatics	3.13	0.0	0.08	0.0	3.21
7+ Ring Aromatics	0.86	0.0	0.0	0.0	0.86
					100.00

<u>Elemental Analysis</u>	<u>Wt. %</u>
Atomic Carbon	90.89
Atomic Hydrogen	8.31
Atomic Nitrogen	0.18
Atomic Oxygen	0.61
Atomic Sulfur	0.01
TOTAL	100.00

*Analysis is approximate

NOTE: Hundredths are shown for normalization purposes only.

(X) Indicates appropriate heteroatom.

Table 13

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Original

Hydrocarbon Types Analysis

Aromatic Hydrocarbons

<u>z No.</u>	<u>Compound Types, $C_nH_{2n-z}^*$</u>	<u>Sample, Wt. %</u>	<u>Avg. Mol. Wt.</u>
z=6	Benzenes	1.79	270.96
z=8	Indans/Tetralins	4.43	251.66
z=10	Dinaphthene Benzenes	6.56	249.76
z=12	Naphthalenes	5.72	255.11
z=14	Acenaphthenes/Biphenyls	9.55	241.80
z=16	Fluorenes	10.90	258.18
z=18	Phenanthrenes/Anthracenes	8.74	263.36
z=20	Aceanthrenes	8.02	268.64
z=22	Pyrenes	10.20	267.32
z=24	Chrysenes	6.74	285.66
z=26	Cholanthrenes	4.99	301.06
z=28	Benzopyrenes	4.01	304.47
z=30	Dibenzanthracenes	2.14	324.50
z=32	Benzoperylenes	2.18	320.85
z=34	Dibenzopyrenes	0.95	342.68
z=36	Coronenes	0.62	356.38
z=38	Naphthcoronenes	0.17	358.26
z=40	Dibenzoperylenes	0.06	363.80
z=42	Benzocoronenes	0.01	378.14
TOTAL		87.78	269.76

Aromatic Nitrogens

<u>z No.</u>	<u>Compound Types, $C_nH_{2n-z}N^*$</u>		
z=13 ^N	Naphthenoquinolines	0.57	237.69
z=15 ^N	Carbazoles	0.29	231.84
z=17 ^N	Acridines	0.55	250.68
z=19 ^N	Naphthenobenzoquinolines	0.45	253.99
z=21 ^N	Benzocarbazoles	0.53	274.74
z=23 ^N	Benzacridines	0.27	282.90
z=25 ^N	Naphthenobenzacridines	0.18	281.03
z=27 ^N	Dibenzocarbazoles	0.12	302.15
z=29 ^N	Dibenzacridines	0.04	415.44
TOTAL		3.00	260.11

*Suggested Compound Types

Estimated Accuracy $\pm$ 10% Relative

Table 13(Cont.)

Aromatic Oxygens

<u>z No.</u>	<u>Compound Types, C_nH_{2n-2}O*</u>	<u>Sample, Wt. %</u>	<u>Avg. Mol. Wt.</u>
z=6 ^O	Phenols	0.50	160.76
z=8 ^O	Naphthenophenols	1.16	188.43
z=10 ^O	Benzofurans	1.44	232.42
z=12 ^O	Naphthols	0.50	195.92
z=14 ^O	Naphthenonaphthols	0.64	234.01
z=16 ^O	Dibenzofurans	1.84	243.73
z=18 ^O	Hydroxyanthracenes	0.86	258.87
z=20 ^O	Hydroxynaphthenoanthracenes	0.56	277.81
z=22 ^O	Hydroxypyrenes	0.73	281.82
z=24 ^O	Hydroxychrysenes	0.50	297.63
z=26 ^O	Hydroxycholanthrenes	0.15	304.68
z=28 ^O	Hydroxybenzopyrenes	0.12	314.81
z=30 ^O	Hydroxydibenzanthracenes	0.10	330.54
z=32 ^O	Hydroxybenzoperylene	0.07	322.12
z=34 ^O	Hydroxybenzopyrenes	0.01	388.18
TOTAL		9.18	240.30

Aromatic Sulfurs

<u>z No.</u>	<u>Compound Types, C_nH_{2n-2}S*</u>		
z=8 ^S	Dihydrothionaphthenes	0.01	164.07
z=10 ^S	Benzo thiophenes	0.01	176.07
z=12 ^S	Naphthenobenzothiophenes	0.01	188.07
z=10 ^S	Fluoreno thiophenes	0.00	236.07
z=22 ^S	Tribenzothiophenes	0.00	234.05
z=28 ^S	Dinaphthothiophenes	0.01	284.07
TOTAL		0.04	203.07

*Suggested Compound Types

Estimated Accuracy ± 10% Relative

Table 14

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Original

Carbon Number Distribution, Wt.% Aromatic Hydrocarbons
 C_nH_{2n-z}

C No.	z=6	z=8	z=10	z=12	z=14	z=16	z=18	z=20	z=22	z=24	z=26	z=28	z=30	z=32	z=34	z=36	z=38	z=40	z=42
C 11	0.02	0.10	0.04	0.08															
C 12	0.03	0.18	0.08	0.19	0.04														
C 13	0.04	0.21	0.22	0.22	0.38	0.02													
C 14	0.07	0.35	0.50	0.31	0.97	0.18	0.15												
C 15	0.07	0.40	0.59	0.31	1.22	0.46	0.43	0.03											
C 16	0.12	0.41	0.71	0.40	1.15	0.99	0.59	0.27	0.64										
C 17	0.18	0.44	0.77	0.62	1.07	1.61	0.88	0.88	1.04										
C 18	0.20	0.36	0.82	0.61	0.99	1.69	1.04	1.32	1.12	0.30									
C 19	0.19	0.32	0.66	0.53	0.76	1.34	1.05	1.11	1.24	0.99	0.26								
C 20	0.15	0.26	0.42	0.52	0.58	1.05	1.16	0.90	1.44	1.12	0.55	0.35							
C 21	0.13	0.22	0.39	0.47	0.47	0.82	0.93	0.85	1.34	0.99	0.65	0.62	0.10						
C 22	0.15	0.21	0.33	0.35	0.51	0.75	0.68	0.71	0.92	0.84	0.78	0.66	0.17	0.27					
C 23	0.12	0.30	0.26	0.27	0.35	0.54	0.58	0.47	0.67	0.61	0.68	0.52	0.35	0.33					
C 24	0.10	0.14	0.23	0.23	0.32	0.49	0.25	0.39	0.58	0.57	0.57	0.47	0.32	0.38	0.11				
C 25	0.07	0.17	0.12	0.20	0.21	0.31	0.24	0.30	0.33	0.39	0.45	0.42	0.31	0.32	0.19	0.05			
C 26	0.06	0.16	0.19	0.13	0.18	0.23	0.27	0.32	0.26	0.27	0.36	0.31	0.27	0.31	0.18	0.12	0.02		
C 27	0.04	0.15	0.10	0.08	0.15	0.14	0.21	0.20	0.26	0.24	0.16	0.22	0.18	0.21	0.14	0.10	0.05		
C 28	0.05	0.05	0.05	0.08	0.10	0.11	0.10	0.14	0.18	0.19	0.20	0.17	0.17	0.13	0.12	0.11	0.05	0.02	
C 29			0.08	0.06	0.05	0.09	0.08	0.08	0.07	0.13	0.12	0.12	0.12	0.11	0.08	0.09		0.03	
C 30				0.06	0.05	0.05	0.04	0.05	0.06	0.06	0.12	0.06	0.05	0.05	0.05	0.07	0.01	0.01	
C 31						0.03	0.06		0.05	0.04	0.06	0.06	0.03	0.04	0.04	0.05	0.04		
C 32											0.03	0.03	0.04	0.03	0.04	0.03			0.01
C 33													0.03						
TOTAL	1.79	4.43	6.56	5.72	9.55	10.90	8.74	8.02	10.20	6.74	4.99	4.01	2.14	2.18	0.95	0.62	0.17	0.06	0.01

 $\Sigma=87.78$

Table 14 (Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Original

Carbon Number Distribution, Wt. % Aromatic Nitrogens
 $C_nH_{2n-z}N$

<u>C No.</u>	<u>$z=13^N$</u>	<u>$z=15^N$</u>	<u>$z=17^N$</u>	<u>$z=19^N$</u>	<u>$z=21^N$</u>	<u>$z=23^N$</u>	<u>$z=25^N$</u>	<u>$z=27^N$</u>	<u>$z=29^N$</u>
C 14	0.10	0.02							
C 15	0.07	0.07	0.06	0.03	0.01				
C 16	0.12	0.06	0.09	0.05	0.04				
C 17	0.08	0.06	0.09	0.08	0.06				
C 18	0.05	0.02	0.08	0.09	0.07	0.02	0.03		
C 19	0.06	0.05	0.02	0.08	0.06	0.03	0.02		
C 20	0.05	0.01	0.17	0.04	0.05	0.04	0.04	0.02	
C 21	0.02		0.03	0.03	0.05	0.04	0.03	0.02	
C 22	0.02		0.01	0.03	0.05	0.05	0.03	0.02	
C 23			0.01	0.02	0.09	0.02	0.02	0.02	
C 24			0.00	0.00	0.02	0.02	0.01	0.02	0.01
C 25					0.03	0.03		0.02	0.00
C 26						0.02			0.01
C 27									0.00
C 28									0.01
C 30									0.01
TOTAL	0.57	0.29	0.55	0.45	0.53	0.27	0.18	0.12	0.04

$\Sigma = 3.00$

Table 14(Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Original

Carbon Number Distribution, Wt. % of Aromatic Oxygens
 $C_nH_{2n-2}O$

C No.	<u>z=6⁰</u>	<u>z=8⁰</u>	<u>z=10⁰</u>	<u>z=12⁰</u>	<u>z=14⁰</u>	<u>z=16⁰</u>	<u>z=18⁰</u>	<u>z=20⁰</u>	<u>z=22⁰</u>	<u>z=24⁰</u>	<u>z=26⁰</u>	<u>z=28⁰</u>	<u>z=30⁰</u>	<u>z=32⁰</u>	<u>z=34⁰</u>
C 7	0.02														
C 8	0.04	0.01													
C 9	0.08	0.05													
C 10	0.10	0.14	0.01	0.02											
C 11	0.08	0.19	0.02	0.08											
C 12	0.07	0.18	0.08	0.09	0.00										
C 13	0.06	0.16	0.11	0.08	0.04	0.07									
C 14	0.05	0.13	0.20	0.07	0.10	0.16	0.02								
C 15		0.09	0.20	0.04	0.12	0.26	0.04	0.00							
C 16		0.10	0.22	0.03	0.11	0.31	0.10	0.01							
C 17		0.05	0.17	0.03	0.07	0.25	0.13	0.07	0.06						
C 18		0.06	0.13	0.06	0.07	0.23	0.15	0.10	0.09						
C 19			0.11		0.04	0.17	0.13	0.08	0.10	0.07					
C 20			0.10		0.04	0.13	0.10	0.08	0.13	0.06	0.02				
C 21			0.06			0.03	0.09	0.09	0.07	0.14	0.09	0.02	0.01		
C 22			0.03			0.02	0.07	0.06	0.06	0.07	0.09	0.04	0.03	0.01	
C 23							0.06	0.02	0.05	0.05	0.09	0.03	0.03	0.02	0.02
C 24							0.04	0.02	0.04	0.04	0.06	0.02	0.02	0.02	0.03
C 25										0.03	0.04	0.02	0.02	0.02	0.01
C 26									0.02			0.01	0.01	0.01	
C 27													0.02		
C 29															0.01
TOTAL	0.50	1.16	1.44	0.50	0.64	1.84	0.86	0.56	0.73	0.50	0.15	0.12	0.10	0.07	0.01

Σ=9.18

Table 14 (Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Original

Carbon Number Distribution, Wt. % of Aromatic Sulfurs
 $C_nH_{2n-z}S$

<u>C No.</u>	<u>$z=8^S$</u>	<u>$z=10^S$</u>	<u>$z=12^S$</u>	<u>$z=20^S$</u>	<u>$z=22^S$</u>	<u>$z=28^S$</u>
C 10	0.01					
C 11		0.01				
C 12			0.01			
C 16				0.00	0.00	
C 20						0.01
TOTAL	0.01	0.01	0.01	0.00	0.00	0.01

$\Sigma=0.04$

Table 15

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 Hrs. Dynamic Test

Sample Analysis Summary

<u>Compound Types, C_nH_{2n-2}(X)</u>	<u>Avg. C No.</u>	<u>Avg. Mol. Wt.</u>	<u>Avg. z No.</u>	<u>Avg. Rings</u>	<u>Wt. %</u>
Saturated Hydrocarbons (P+N)			NOT DETERMINED		
Aromatic Hydrocarbons	19.84	261.02	16.91	2.65	79.51
Aromatic Nitrogens	19.27	264.82	18.78	3.53	4.49
Aromatic Oxygens	16.87	237.40	14.91	2.24	15.95
Aromatic Sulfurs	25.40	195.67	12.80	2.00	0.05
TOTAL	19.35	257.39	16.67	2.62	100.00
<u>Distribution of Aromatic Rings, C_nH_{2n-2}(X), Wt. %</u>	<u>C-H</u>	<u>C-H-N*</u>	<u>C-H-O*</u>	<u>C-H-S*</u>	<u>Total*</u>
1 Ring Aromatics	16.24	0.0	5.40	0.02	21.66
2 Ring Aromatics	29.31	0.66	5.26	0.02	35.25
3 Ring Aromatics	12.42	1.48	2.55	0.0	16.45
4 Ring Aromatics	11.99	1.85	1.74	0.01	15.59
5 Ring Aromatics	7.40	0.46	0.88	0.0	8.74
6 Ring Aromatics	1.75	0.04	0.12	0.0	1.91
7+ Ring Aromatics	0.40	0.0	0.0	0.0	0.40
					100.00
<u>Elemental Analysis</u>	<u>Wt. %</u>				
Atomic Carbon	90.13				
Atomic Hydrogen	8.55				
Atomic Nitrogen	0.24				
Atomic Oxygen	1.07				
Atomic Sulfur	0.01				
TOTAL	100.00				

*Analysis is approximate

NOTE: Hundredths are shown for normalization purposes only.

(X) Indicates appropriate heteroatom.

Table 16

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 Hrs. Dynamic Test

Hydrocarbon Types Analysis

Aromatic Hydrocarbons

<u>z No.</u>	<u>Compound Types, C_nH_{2n-z}*</u>	<u>Sample, Wt. %</u>	<u>Avg. Mol. Wt.</u>
z=6	Benzenes	2.48	270.70
z=8	Indans/Tetralins	6.35	238.68
z=10	Dinaphthene Benzenes	7.41	243.22
z=12	Naphthalenes	6.66	242.92
z=14	Acenaphthenes/Biphenyls	10.89	234.62
z=16	Fluorenes	11.76	252.23
z=18	Phenanthrenes/Anthracenes	6.24	261.88
z=20	Aceanthrenes	6.18	271.54
z=22	Pyrenes	7.13	269.36
z=24	Chrysenes	4.86	286.41
z=26	Cholanthrenes	3.41	301.91
z=28	Benzopyrenes	2.57	307.83
z=30	Dibenzanthracenes	1.42	325.98
z=32	Benzoperylenes	1.32	321.43
z=34	Dibenzopyrenes	0.43	351.65
z=36	Coronenes	0.30	351.02
z=38	Naphthcoronenes	0.10	362.55
TOTAL		79.51	261.02

Aromatic Nitrogens

<u>z No.</u>	<u>Compound Types, C_nH_{2n-z}N*</u>		
z=13 ^N	Naphthenoquinolines	0.66	225.43
z=15 ^N	Carbazoles	0.64	259.58
z=17 ^N	Acridines	0.84	251.15
z=19 ^N	Naphthenobenzoquinolines	0.73	275.55
z=21 ^N	Benzocarbazoles	0.71	266.69
z=23 ^N	Benzacridines	0.41	291.38
z=25 ^N	Naphthenobenzacridines	0.28	288.64
z=27 ^N	Dibenzocarbazoles	0.10	323.17
z=29 ^N	Dibenzacridines	0.08	335.17
z=31 ^N	Acenaphthobenzindole	0.01	319.14
z=33 ^N	Tribenzocarbazoles	0.03	335.81
TOTAL		4.49	264.82

*Suggested Compound Types

Estimated Accuracy ± 10% Relative

Table 16(Cont.)

Aromatic Oxygens

<u>z No.</u>	<u>Compound Types, C_nH_{2n-2}O*</u>	<u>Sample, Wt-%</u>	<u>Avg. Mol. Wt.</u>
z=6 ^O	Phenols	1.02	156.43
z=8 ^O	Naphthenophenols	2.51	195.89
z=10 ^O	Benzofurans	1.87	221.03
z=12 ^O	Naphthols	1.11	204.92
z=14 ^O	Naphthenonaphthols	1.15	231.01
z=16 ^O	Dibenzofurans	3.00	239.35
z=18 ^O	Hydroxyanthracenes	1.54	267.88
z=20 ^O	Hydroxynaphthenoanthracenes	1.01	273.10
z=22 ^O	Hydroxypyrenes	1.20	280.56
z=24 ^O	Hydroxychrysenes	0.54	295.74
z=26 ^O	Hydroxycholanthrenes	0.53	322.73
z=28 ^O	Hydroxybenzopyrenes	0.19	308.66
z=30 ^O	Hydroxydibenzanthracenes	0.16	320.38
z=32 ^O	Hydroxybenzoperylene	<u>0.12</u>	<u>328.30</u>
TOTAL		15.95	237.04

Aromatic Sulfurs

<u>z No.</u>	<u>Compound Types, C_nH_{2n-2}S*</u>		
z=8 ^S	Dihydrothionaphthenes	0.02	164.07
z=12 ^S	Naphthenobenzothiophenes	0.02	188.06
z=24 ^S	Acephenanthrylenethiophenes	<u>0.01</u>	<u>274.08</u>
TOTAL		0.05	195.67

*Suggested Compound Types

Estimated Accuracy ± 10% Relative

Table 17

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 Hrs. Dynamic Test

Carbon Number Distribution, Wt. % of Aromatic Hydrocarbons

 C_nH_{2n-2}

C No.	z=6	z=8	z=10	z=12	z=14	z=16	z=18	z=20	z=22	z=24	z=26	z=28	z=30	z=32	z=34	z=36	z=38
C 10		0.08	0.04														
C 11		0.23	0.07	0.14													
C 12	0.02	0.34	0.10	0.31	0.05												
C 13	0.06	0.40	0.28	0.34	0.49	0.02											
C 14	0.13	0.58	0.64	0.40	1.23	0.21	0.12										
C 15	0.11	0.63	0.77	0.42	1.60	0.53	0.35	0.02									
C 16	0.19	0.49	0.84	0.58	1.37	1.19	0.45	0.19	0.46								
C 17	0.25	0.59	0.95	0.83	1.29	1.97	0.57	0.67	0.71								
C 18	0.32	0.59	0.89	0.84	1.23	2.01	0.75	0.92	0.76	0.18							
C 19	0.29	0.51	0.70	0.61	0.84	1.47	0.77	0.84	0.80	0.68	0.16						
C 20	0.23	0.37	0.51	0.51	0.64	1.14	0.75	0.70	0.88	0.78	0.29	0.17					
C 21	0.19	0.26	0.43	0.42	0.58	0.96	0.62	0.69	0.98	0.69	0.42	0.37	0.05				
C 22	0.14	0.36	0.24	0.31	0.40	0.69	0.52	0.49	0.70	0.58	0.57	0.42	0.12	0.17			
C 23	0.09	0.19	0.23	0.27	0.32	0.47	0.40	0.42	0.49	0.48	0.52	0.34	0.21	0.20			
C 24	0.06	0.28	0.22	0.21	0.23	0.35	0.28	0.36	0.41	0.44	0.39	0.30	0.20	0.20		0.02	
C 25	0.06	0.23	0.16	0.16	0.19	0.27	0.23	0.26	0.26	0.37	0.31	0.27	0.21	0.18	0.07	0.02	
C 26	0.12	0.13	0.05	0.12	0.15	0.15	0.19	0.17	0.22	0.31	0.21	0.21	0.17	0.18	0.09	0.07	0.01
C 27	0.09	0.09	0.11	0.08	0.11	0.13	0.13	0.14	0.16	0.19	0.21	0.16	0.15	0.15	0.07	0.05	0.02
C 28	0.08		0.08	0.06	0.09	0.06	0.05	0.12	0.12	0.07	0.18	0.12	0.12	0.10	0.05	0.04	0.02
C 29	0.05		0.10	0.05	0.08	0.05	0.06	0.06	0.07	0.05	0.11	0.10	0.08	0.08	0.07	0.04	0.02
C 30						0.05		0.07	0.05		0.04	0.04	0.06	0.04	0.05	0.02	0.01
C 31						0.04		0.02	0.04	0.04		0.04	0.03	0.02	0.03	0.02	0.02
C 32								0.04	0.02			0.03	0.02			0.02	
TOTAL	2.48	6.35	7.41	6.66	10.89	11.76	6.24	6.18	7.13	4.86	3.41	2.57	1.42	1.32	0.43	0.30	0.10

Σ=79.51

Table 17(Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 Hrs. Dynamic Test

Carbon Number Distribution, Wt. % of Aromatic Nitrogens
 $C_nH_{2n-2}N$

<u>C No.</u>	<u>z=13^N</u>	<u>z=15^N</u>	<u>z=17^N</u>	<u>z=19^N</u>	<u>z=21^N</u>	<u>z=23^N</u>	<u>z=25^N</u>	<u>z=27^N</u>	<u>z=29^N</u>	<u>z=31^N</u>	<u>z=33^N</u>
C 13	0.06										
C 14	0.13	0.05	0.04								
C 15	0.16	0.07	0.08		0.01						
C 16	0.10	0.11	0.16		0.05						
C 17	0.09	0.07	0.15	0.07	0.10						
C 18	0.07	0.08	0.12	0.13	0.12	0.04	0.02				
C 19	0.02	0.04	0.05	0.14	0.11	0.07	0.04				
C 20	0.01	0.09	0.08	0.14	0.11	0.06	0.06	0.01			
C 21	0.02	0.03	0.05	0.06	0.10	0.07	0.03	0.02			
C 22		0.04	0.03	0.04	0.09	0.09	0.04	0.02	0.02		
C 23		0.04	0.03	0.10	0.02	0.05	0.03	0.02	0.02		
C 24		0.01	0.03	0.03		0.03	0.03	0.02	0.02	0.01	0.01
C 25		0.01	0.02	0.02			0.03	0.01	0.01		0.01
C 26									0.01		
C 27											0.01
TOTAL	0.66	0.64	0.84	0.73	0.71	0.41	0.28	0.10	0.08	0.01	0.03

Σ=4.49

Table 17(Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 hrs. Dynamic Test

Carbon Number Distribution, Wt. % of Aromatic Oxygens

C_nH_{2n-z}O

<u>C No.</u>	<u>z=6⁰</u>	<u>z=8⁰</u>	<u>z=10⁰</u>	<u>z=12⁰</u>	<u>z=14⁰</u>	<u>z=16⁰</u>	<u>z=18⁰</u>	<u>z=20⁰</u>	<u>z=22⁰</u>	<u>z=24⁰</u>	<u>z=26⁰</u>	<u>z=28⁰</u>	<u>z=30⁰</u>	<u>z=32⁰</u>
C 7	0.04													
C 8	0.11													
C 9	0.22	0.13												
C 10	0.21	0.30	0.01	0.05										
C 11	0.16	0.39	0.04	0.12										
C 12	0.11	0.35	0.09	0.19		0.01								
C 13	0.08	0.28	0.22	0.12	0.07	0.10								
C 14	0.05	0.27	0.32	0.13	0.20	0.30								
C 15	0.04	0.15	0.32	0.06	0.18	0.43	0.07							
C 16		0.23	0.31	0.10	0.21	0.50	0.16	0.04	0.02					
C 17		0.11	0.23	0.18	0.15	0.48	0.23	0.11	0.10					
C 18		0.09	0.20	0.16	0.14	0.39	0.21	0.17	0.16	0.02				
C 19		0.09	0.13		0.09	0.29	0.24	0.17	0.19					
C 20		0.07			0.06	0.21	0.14	0.18	0.19	0.13	0.04			
C 21		0.05			0.05	0.14	0.16	0.13	0.18	0.10	0.07	0.02		
C 22						0.09	0.12	0.08	0.12	0.12	0.09	0.05	0.04	0.01
C 23						0.06	0.10	0.06	0.09	0.12	0.08	0.07	0.04	0.03
C 24							0.06	0.04	0.06		0.06	0.03	0.03	0.02
C 25							0.03	0.03	0.03	0.05	0.06	0.02	0.02	0.02
C 26							0.02		0.06		0.05		0.02	0.02
C 27											0.03			0.02
C 28											0.02		0.01	
C 29											0.01			
C 30											0.02			
TOTAL	1.02	2.51	1.87	1.11	1.15	3.00	1.54	1.01	1.20	0.54	0.53	0.19	0.16	0.12

Σ=15.95

Table 17(Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 hrs. Dynamic Test

Carbon Number Distribution, Wt. % of Aromatic Sulfurs
 $C_nH_{2n-z}S$

<u>C No.</u>	<u>z=8S</u>	<u>z=12S</u>	<u>z=24S</u>
C 10	0.02		
C 12		0.02	
C 19			0.01
TOTAL	0.02	0.02	0.01
$\Sigma = 0.05$			

Table 18

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 Hrs. Static Test

Sample Analysis Summary

<u>Compound Types, C_nH_{2n-z}(X)</u>	<u>Avg. C No.</u>	<u>Avg. Mol. Wt.</u>	<u>Avg. z No.</u>	<u>Avg. Rings</u>	<u>Wt. %</u>
Saturated Hydrocarbons (P+N)			NOT DETERMINED		
Aromatic Hydrocarbons	20.04	263.07	17.73	2.81	78.33
Aromatic Nitrogens	19.55	268.49	19.45	3.61	5.36
Aromatic Oxygens	17.58	246.64	15.59	2.36	16.19
Aromatic Sulfurs	11.75	185.40	11.17	2.17	0.12
TOTAL	19.61	260.60	17.47	2.78	100.00
<u>Distribution of Aromatic Rings, C_nH_{2n-z}(X), Wt. %</u>	<u>C-H</u>	<u>C-H-N*</u>	<u>C-H-O*</u>	<u>C-H-S*</u>	<u>Total*</u>
1 Ring Aromatics	12.84	0.0	4.86	0.0	17.70
2 Ring Aromatics	28.11	0.68	5.42	0.11	34.32
3 Ring Aromatics	12.97	1.49	2.66	0.0	17.12
4 Ring Aromatics	13.33	2.31	1.91	0.01	17.56
5 Ring Aromatics	8.25	0.79	1.12	0.0	10.16
6 Ring Aromatics	2.40	0.08	0.22	0.0	2.70
7+ Ring Aromatics	0.43	0.01	0.0	0.0	0.44
					100.00

<u>Elemental Analysis</u>	<u>Wt. %</u>
Atomic Carbon	90.30
Atomic Hydrogen	8.35
Atomic Nitrogen	0.28
Atomic Oxygen	1.05
Atomic Sulfur	0.02
TOTAL	100.00

*Analysis is approximate

NOTE: Hundredths are shown for normalization purposes only.

(X) Indicates appropriate heteroatom.

Table 19

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 Hrs. Static Test

Hydrocarbon Types Analysis

Aromatic Hydrocarbons

<u>z No.</u>	<u>Compound Types, C_nH_{2n-z}*</u>	<u>Sample, Wt. %</u>	<u>Avg. Mol. Wt.</u>
z=6	Benzenes	1.31	250.83
z=8	Indans/Tetralins	5.08	234.51
z=10	Dinaphthene Benzenes	6.45	242.99
z=12	Naphthalenes	5.85	243.45
z=14	Acenaphthenes/Biphenyls	10.27	236.36
z=16	Fluorenes	11.99	253.70
z=18	Phenanthrenes/Anthracenes	6.67	265.87
z=20	Aceanthrenes	6.30	270.48
z=22	Pyrenes	8.13	272.05
z=24	Chrysenes	5.20	285.80
z=26	Cholanthrenes	3.71	297.06
z=28	Benzopyrenes	3.11	306.71
z=30	Dibenzanthracenes	1.43	320.74
z=32	Benzoperylene	1.68	320.98
z=34	Dibenzopyrenes	0.72	341.24
z=36	Coronenes	0.33	344.26
z=38	Naphthcoronenes	0.10	368.16
TOTAL		78.33	263.07

Aromatic Nitrogens

<u>z No.</u>	<u>Compound Types, C_nH_{2n-z}N*</u>		
z=13 ^N	Naphthenoquinolines	0.68	243.91
z=15 ^N	Carbazoles	0.65	231.55
z=17 ^N	Acridines	0.84	256.16
z=19 ^N	Naphthenobenzoquinolines	0.89	268.08
z=21 ^N	Benzocarbazoles	0.91	276.23
z=23 ^N	Benzacridines	0.51	308.51
z=25 ^N	Naphthenobenzacridines	0.45	296.22
z=27 ^N	Dibenzocarbazoles	0.19	289.23
z=29 ^N	Dibenzacridines	0.15	312.74
z=31 ^N	Acenaphthobenzindole	0.05	321.94
z=33 ^N	Tribenzocarbazoles	0.02	352.16
z=35 ^N	Benzonaphthacridines	0.01	343.14
z=37 ^N	Diindenoacridines	0.01	341.12
TOTAL		5.36	269.40

*Suggested Compound Types

Estimated Accuracy ± 10% Relative

Table 19 (Cont.)

Aromatic Oxygens

<u>z No.</u>	<u>Compound Types, C_nH_{2n-z}O*</u>	<u>Sample, Wt. %</u>	<u>Avg. Mol. Wt.</u>
z=6 ^O	Phenols	0.69	161.68
z=8 ^O	Naphthenophenols	2.00	201.70
z=10 ^O	Benzofurans	2.17	230.42
z=12 ^O	Naphthols	1.39	228.76
z=14 ^O	Naphthenonaphthols	0.96	227.30
z=16 ^O	Dibenzofurans	3.07	242.29
z=18 ^O	Hydroxyanthracenes	1.59	269.62
z=20 ^O	Hydroxynaphthenoanthracenes	1.07	278.64
z=22 ^O	Hydroxypyrenes	1.25	274.92
z=24 ^O	Hydroxychrysenes	0.66	301.21
z=26 ^O	Hydroxycholanthrenes	0.53	309.24
z=28 ^O	Hydroxybenzopyrenes	0.31	327.32
z=30 ^O	Hydroxydibenzanthracenes	0.28	323.14
z=32 ^O	Hydroxybenzoperylenes	<u>0.22</u>	<u>331.59</u>
TOTAL		16.19	246.64

Aromatic Sulfurs

<u>z No.</u>	<u>Compound Types, C_nH_{2n-z}S*</u>		
z=10 ^S	Benzothiophenes	0.09	179.18
z=12 ^S	Naphthenobenzothiophenes	0.02	188.07
z=20 ^S	Fluorenothiophenes	<u>0.01</u>	<u>236.07</u>
TOTAL		0.12	185.40

*Suggested Compounds

Estimated Accuracy ± 10% Relative

Table 20

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 Hrs. Static Test

Carbon Number Distribution, Wt.% of Aromatic Hydrocarbons
 C_nH_{2n-z}

C No.	z=6	z=8	z=10	z=12	z=14	z=16	z=18	z=20	z=22	z=24	z=26	z=28	z=30	z=32	z=34	z=36	z=38
C 10		0.06	0.02														
C 11		0.11	0.03	0.14													
C 12		0.22	0.05	0.21	0.03												
C 13	0.04	0.30	0.21	0.19	0.38	0.02											
C 14	0.07	0.44	0.55	0.31	1.04	0.16	0.09										
C 15	0.07	0.53	0.61	0.41	1.34	0.48	0.31										
C 16	0.11	0.56	0.70	0.49	1.31	1.10	0.41	0.17	0.46								
C 17	0.19	0.54	0.86	0.66	1.31	1.92	0.60	0.59	0.75								
C 18	0.23	0.45	0.92	0.82	1.24	2.07	0.77	0.98	0.77	0.20							
C 19	0.20	0.41	0.73	0.65	0.91	1.54	0.84	0.95	0.94	0.74	0.23						
C 20	0.17	0.42	0.47	0.49	0.70	1.29	0.80	0.78	1.12	0.85	0.40	0.25					
C 21	0.07	0.43	0.33	0.43	0.52	0.96	0.71	0.69	1.09	0.71	0.54	0.44	0.06				
C 22	0.06	0.21	0.24	0.37	0.51	0.72	0.55	0.56	0.81	0.65	0.63	0.49	0.14	0.19			
C 23	0.06	0.22	0.21	0.21	0.18	0.59	0.43	0.43	0.56	0.60	0.57	0.45	0.21	0.29			
C 24	0.04	0.18	0.19	0.22	0.23	0.39	0.33	0.34	0.44	0.44	0.34	0.36	0.28	0.26	0.09	0.02	
C 25			0.16	0.11	0.18	0.26	0.29	0.25	0.34	0.33	0.26	0.30	0.18	0.25	0.14	0.05	
C 26			0.09	0.14	0.19	0.19	0.22	0.16	0.23	0.26	0.30	0.23	0.16	0.23	0.14	0.05	
C 27			0.08		0.11	0.20	0.15	0.14	0.20	0.18	0.17	0.21	0.14	0.15	0.12	0.06	0.02
C 28					0.09	0.10	0.10	0.12	0.10	0.11	0.11	0.12	0.12	0.13	0.07	0.07	0.02
C 29							0.07	0.05	0.12	0.07	0.06	0.13	0.08	0.10	0.07	0.05	0.02
C 30								0.05	0.10	0.06	0.08	0.04	0.06	0.05	0.05	0.03	0.02
C 31								0.04	0.07		0.02	0.05		0.03	0.02		0.02
C 32									0.03			0.04			0.02		
TOTAL	1.31	5.08	6.45	5.85	10.27	11.99	6.67	6.30	8.13	5.20	3.71	3.11	1.43	1.68	0.72	0.33	0.10

 $\Sigma = 78.33$

Table 20 (Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 hrs. Static Test

Carbon Number Distribution, Wt-% of Aromatic Nitrogens
C_nH_{2n-z}N

<u>C No.</u>	<u>z=13^N</u>	<u>z=15^N</u>	<u>z=17^N</u>	<u>z=19^N</u>	<u>z=21^N</u>	<u>z=23^N</u>	<u>z=25^N</u>	<u>z=27^N</u>	<u>z=29^N</u>	<u>z=31^N</u>	<u>z=33^N</u>	<u>z=35^N</u>	<u>z=37^N</u>
C 12		0.02											
C 13		0.06											
C 14	0.07	0.07	0.02	0.02									
C 15	0.13	0.10	0.07	0.05	0.02								
C 16	0.11	0.12	0.11	0.07	0.04								
C 17	0.11	0.06	0.14	0.06	0.12	0.01							
C 18	0.06	0.06	0.09	0.14	0.14	0.03	0.02						
C 19	0.05	0.03	0.07	0.14	0.12	0.04	0.07	0.02					
C 20	0.05	0.07	0.09	0.08	0.09	0.06	0.05	0.04					
C 21	0.04	0.03	0.20	0.10	0.09	0.05	0.07	0.04	0.02				
C 22	0.03	0.02	0.03	0.07	0.08	0.05	0.05	0.04	0.03				
C 23	0.02	0.01	0.02	0.11	0.05	0.06	0.07	0.01	0.02	0.01			
C 24	0.01			0.03	0.05	0.06	0.04	0.03	0.04	0.02			
C 25				0.01	0.06	0.05	0.04	0.01	0.03	0.02			
C 26				0.01	0.03	0.05	0.04		0.01		0.01	0.01	0.01
C 27					0.02	0.03					0.01		
C 28						0.02							
TOTAL	0.68	0.65	0.84	0.89	0.91	0.51	0.45	0.19	0.15	0.05	0.02	0.01	0.01

Σ=5.36

Table 20 (Cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 hrs. Static Test

Carbon Number Distribution, Wt. % of Aromatic Oxygens
 $C_nH_{2n-z}O$

C No.	z=6 ⁰	z=8 ⁰	z=10 ⁰	z=12 ⁰	z=14 ⁰	z=16 ⁰	z=18 ⁰	z=20 ⁰	z=22 ⁰	z=24 ⁰	z=26 ⁰	z=28 ⁰	z=30 ⁰	z=32 ⁰
C 8	0.07													
C 9	0.10	0.05												
C 10	0.20	0.21		0.02										
C 11	0.09	0.28	0.02	0.10										
C 12	0.08	0.23	0.06	0.12		0.01								
C 13	0.07	0.22	0.19	0.11	0.06	0.08								
C 14	0.05	0.22	0.28	0.10	0.13	0.22								
C 15	0.03	0.15	0.40	0.11	0.18	0.38	0.05							
C 16		0.18	0.37	0.14	0.22	0.54	0.12	0.03	0.03					
C 17		0.26	0.32	0.19	0.15	0.52	0.23	0.09	0.12					
C 18		0.08	0.23	0.22	0.11	0.45	0.28	0.17	0.17	0.05				
C 19		0.07	0.14	0.10	0.11	0.33	0.20	0.18	0.24	0.05				
C 20		0.05	0.09	0.08		0.23	0.21	0.15	0.20	0.06	0.09	0.01		
C 21			0.07	0.05		0.15	0.15	0.13	0.18	0.10	0.07	0.01	0.02	
C 22				0.05		0.12	0.14	0.13	0.11	0.13	0.09	0.02	0.04	
C 23						0.04	0.10	0.09	0.10	0.09	0.08	0.05	0.05	0.04
C 24							0.07	0.06	0.07	0.08	0.08	0.08	0.06	0.06
C 25							0.04	0.04	0.03	0.05	0.07	0.07	0.05	0.05
C 26										0.03	0.03	0.05	0.03	0.04
C 27										0.02	0.02	0.02	0.02	0.03
C 28													0.01	
TOTAL	0.69	2.00	2.17	1.39	0.96	3.07	1.59	1.07	1.25	0.66	0.53	0.31	0.28	0.22

Σ=16.19

Table 20(Cont.)

Analysis of High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid After 625°F 100 hrs. Static Test

Carbon Number Distribution, Wt.-% of Aromatic Sulfurs
 $C_nH_{2n-z}S$

<u>C No.</u>	<u>$z=10^S$</u>	<u>$z=12^S$</u>	<u>$z=20^S$</u>
C 10	0.01		
C 11	0.05		
C 12	0.03	0.02	
C 16			0.01
TOTAL	0.09	0.02	0.01

$\Sigma=0.12$

Table 21

Comparison of Sample Summaries for the V-178 Coal-Derived Liquid
Original and After Static and Dynamic Tests

Sample Analysis Summary

Compound Types, $C_nH_{2n-2}(X)$	Coal Liquids Wt. %		
	Original	After Static	After Dynamic
Saturated Hydrocarbons (P+N)		NOT DETERMINED	
Aromatic Hydrocarbons	87.8	78.3	79.5
Aromatic Nitrogens	3.0	5.4	4.5
Aromatic Oxygens	9.2	16.2	16.0
Aromatic Sulfurs	T	0.1	T

Elemental Analysis by MS in Wt. %

Atomic Carbon	90.89	90.30	90.13
Atomic Hydrogen	8.31	8.35	8.55
Atomic Nitrogen	0.18	0.28	0.24
Atomic Oxygen	0.61	1.05	1.07
Atomic Sulfur	0.01	0.02	0.01

T ≤ .05 wt. %

Table 22

Results of Chemiluminescence Study at 100°C of V-178 Coal-Derived
Liquid Samples from Corrosion Tests at 625°F (329°C)

<u>Time, mins</u>	<u>Photon Counts/sec</u>		
	<u>Original Sample</u>	<u>After Static Test</u>	<u>After Dynamic Test</u>
0	12	3	10
1	24	27	32
2	63	42	47
5	125	85	108
10	228	192	232
15	349	285	330

Table 23

Results of Chemiluminescence Study at 150°C of Coal-Derived
Liquid Samples from Corrosion Tests at 625°F (329°C)

<u>Time, mins</u>	<u>Photon Counts/sec</u>		
	<u>Original Sample</u>	<u>After Static Test</u>	<u>After Dynamic Test</u>
0	0	0	0
1	23	25	22
5	124	107	110
10	242	230	240
15	360	340	350

Table 24

Chemiluminescence Response of Original V-178
Coal-Derived Liquid at 150°C Under Nitrogen

<u>Time, mins</u>	<u>Photon Count/sec</u>
0	0
1	24
5	131
10	245
15	340

Table 25

ESR of Various V-178 Coal-Derived Liquid

<u>Sample</u>	<u>Peak Height</u>	<u>g-Value</u>
Original	89.6	2.00334
After 625°F Static Test	28.5	2.00295
After 625°F Dynamic Test	41.0	2.00300
Original	86.8	2.00309
After 625°F Dynamic Test	19.6	2.00275

Table 26

Corrosion Rate Data for 1010, 5 Cr-0.5 Mo,
Type 304L and Type 316L in a V-178 Coal-Derived Liquid

Metal	Corrosion Rate, mpy					
	550°F		625°F		700°F	
	Static	Dynamic	Static	Dynamic	Static	Dynamic
1010	18.3	31.9	30.2	27.7	-	-
5 Cr-0.5 Mo	18.8	29.2	14.2	26.1	-	-
304L	-	-	6.3	4.1	4.5	4.1
316L	-	-	1.5	0.7	3.2	3.0

Table 27

Stability Test Products Analysis
of V-178 Coal-Derived Liquid Before and After
Stability Test Storage Temperature 110°F (43.3°C)

<u>Analysis</u>	<u>Before Aging</u>	<u>After Aging for 30 Days</u>
API° @ 60°F	6.8	6.9
Specific Gravity 60/60°F	1.023	1.022
Kinematic Viscosity, cSt		
100°F	76.4	87.8
210°F	5.6	5.8
Heptane-Insolubles, wt.%	2.54	*
Toluene-Insolubles, wt.%	0.02	0.11
Acid number, mg KOH/g	0.15	0.17
Dissolved O ₂ , ppm	<1.0	<1.0
Particulates, wt.%	0.132	0.310

* Heptane-insolubles for this sample could not be accurately measured because of the gummy nature of the insolubles.

Table 28

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Before Aging

Sample Analysis Summary

<u>Compound Types, C_nH_{2n-z}(X)</u>	<u>Avg. C No.</u>	<u>Avg. Mol. Wt.</u>	<u>Avg. z No.</u>	<u>Avg. Rings</u>	<u>Wt. %</u>
Saturated Hydrocarbons (P+N)			NOT DETERMINED		
Aromatic Hydrocarbons	19.69	259.55	16.32	2.52	77.49
Aromatic Nitrogens	18.76	258.96	18.13	3.39	4.78
Aromatic Oxygens	17.20	241.97	15.01	2.26	17.63
Aromatic Sulfurs	<u>13.33</u>	<u>186.47</u>	<u>14.04</u>	<u>2.40</u>	<u>0.10</u>
TOTAL	19.20	256.33	16.18	2.52	100.00
<u>Distribution of Aromatic Rings, C_nH_{2n-z}(X), wt. %</u>	<u>C-H</u>	<u>C-H-N*</u>	<u>C-H-O*</u>	<u>C-H-S*</u>	<u>Total*</u>
1 Ring Aromatics	17.32	0.0	5.79	0.01	23.12
2 Ring Aromatics	29.40	0.88	5.97	0.07	36.32
3 Ring Aromatics	12.29	1.57	2.66	0.00	16.52
4 Ring Aromatics	11.39	1.84	2.03	0.02	15.28
5 Ring Aromatics	5.74	0.46	1.09	0.0	7.29
6 Ring Aromatics	1.29	0.03	0.09	0.0	1.41
7+ Ring Aromatics	0.06	0.0	0.0	0.0	0.06
<u>Elemental Analysis</u>	<u>Wt. %</u>				
Atomic Carbon	89.89				
Atomic Hydrogen	8.67				
Atomic Nitrogen	0.26				
Atomic Oxygen	1.16				
Atomic Sulfur	<u>0.02</u>				
TOTAL	100.00				

*Analysis is approximate

NOTE: Hundredths are shown for normalization purposes only.

(X) Indicates appropriate heteroatom.

Table 29

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Before and After Aging

Hydrocarbon Types Analysis

Aromatic Hydrocarbons

z No.	Compound Types, $C_nH_{2n-z}^*$	Original		After Aging	
		Sample, Wt. %	Avg. Mol. Wt.	Sample, Wt. %	Avg. Mol. Wt.
z=6	Benzenes	2.57	254.80	2.73	266.15
z=8	Indans/Tetralins	7.03	242.28	7.32	258.25
z=10	Dinaphthene Benzenes	7.72	242.78	7.56	246.99
z=12	Naphthalenes	6.71	243.78	6.48	249.34
z=14	Acenaphthenes/Biphenyls	10.86	235.55	10.29	238.21
z=16	Fluorenes	11.83	253.56	11.69	256.43
z=18	Phenanthrenes/Anthracenes	6.31	266.01	6.27	265.70
z=20	Aceanthrenes	5.98	270.77	6.05	271.09
z=22	Pyrenes	6.77	270.50	6.91	270.01
z=24	Chrysenes	4.62	288.86	4.79	291.59
z=26	Cholanthrenes	2.71	306.34	3.00	305.78
z=28	Benzopyrenes	2.07	306.53	2.26	310.08
z=30	Dibenzanthracenes	0.96	328.62	1.18	334.29
z=32	Benzoperylene	0.96	322.96	1.12	325.40
z=34	Dibenzopyrenes	0.33	341.61	0.35	344.96
z=36	Coronenes	0.06	337.47	0.16	357.03
TOTAL		77.49	259.55	78.16	264.49

Aromatic Nitrogens

z No.	Compound Types, $C_nH_{2n-z}^N$				
z=13 ^N	Naphthenoquinolines	0.88	231.32	0.51	239.17
z=15 ^N	Carbazoles	0.75	239.21	0.69	234.92
z=17 ^N	Acridines	0.87	251.20	0.82	254.11
z=19 ^N	Naphthenobenzoquinolines	0.79	269.49	0.64	268.38
z=21 ^N	Benzocarbazoles	0.72	265.95	0.65	280.53
z=23 ^N	Benzacridines	0.33	290.69	0.35	292.82
z=25 ^N	Naphthenobenzacridines	0.26	296.52	0.29	307.92
z=27 ^N	Dibenzocarbazoles	0.17	311.10	0.17	301.73
z=29 ^N	Dibenzacridines	0.03	314.14	0.09	324.53
z=31 ^N	Acenaphthobenzindole	0.03	305.12	0.03	335.31
TOTAL		4.78	258.96	4.24	266.25

*Suggested Compound Types

Estimated Accuracy $\pm$ 10% Relative

Table 29(Cont.)

Aromatic Oxygens

z No.	Compound Types, $C_nH_{2n-z}O^*$	Original		After Aging	
		Sample, Wt. %	Avg. Mol. Wt.	Sample, Wt. %	Avg. Mol. Wt.
z=6 ^O	Phenols	1.08	165.55	0.97	163.54
z=8 ^O	Naphthenophenols	2.36	188.23	2.16	191.11
z=10 ^O	Benzofurans	2.35	226.71	2.42	236.19
z=12 ^O	Naphthols	1.56	223.66	1.53	231.17
z=14 ^O	Naphthenonaphthols	1.40	244.46	1.32	243.76
z=16 ^O	Dibenzofurans	3.01	240.56	3.05	243.33
z=18 ^O	Hydroxyanthracenes	1.53	265.89	1.66	268.12
z=20 ^O	Hydroxynaphthenoanthracenes	1.13	282.10	1.18	282.09
z=22 ^O	Hydroxypyrenes	1.24	286.79	1.26	281.25
z=24 ^O	Hydroxychrysenes	0.79	301.04	0.73	303.41
z=26 ^O	Hydroxycholanthrenes	0.48	314.78	0.50	314.95
z=28 ^O	Hydroxybenzopyrenes	0.37	321.12	0.34	328.69
z=30 ^O	Hydroxydibenzanthracenes	0.24	318.05	0.29	330.83
z=32 ^O	Hydroxybenzoperylene	0.08	330.63	0.12	344.65
z=34 ^O	Hydroxybenzopyrenes	0.01	318.10		
TOTAL		17.63	241.97	17.53	246.49

Aromatic Sulfurs

z No.	Compound Types, $C_nH_{2n-z}S^*$				
z=8 ^S	Dihydrothionaphthenes	0.01	164.07		
z=10 ^S	Benzo thiophenes	0.05	170.46	0.04	172.56
z=12 ^S	Naphthenobenzothiophenes	0.0	188.07	0.03	188.07
z=18 ^S	Acenaphthenothiophenes	0.00	224.07		
z=20 ^S	Fluoreno thiophenes	0.02	236.07	0.00	236.07
z=22 ^S	Tribenzothiophenes	0.00	234.05		
TOTAL		0.10	186.47	0.07	179.20

*Suggested Compound Types

Estimated Accuracy $\pm$ 10% Relative

Table 30

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Before Aging

Carbon Number Distribution, Wt-% Aromatic Hydrocarbons
 C_nH_{2n-z}

C No.	Z=6	Z=8	Z=10	Z=12	Z=14	Z=16	Z=18	Z=20	Z=22	Z=24	Z=26	Z=28	Z=30	Z=32	Z=34	Z=36
C 11	0.06	0.24	0.08	0.14												
C 12	0.05	0.33	0.11	0.30	0.06											
C 13	0.10	0.42	0.30	0.33	0.51	0.03										
C 14	0.13	0.56	0.69	0.38	1.21	0.19	0.13									
C 15	0.13	0.70	0.79	0.45	1.57	0.56	0.32	0.02								
C 16	0.22	0.68	0.89	0.55	1.34	1.26	0.40	0.19	0.46							
C 17	0.29	0.64	1.07	0.84	1.31	1.92	0.55	0.59	0.68							
C 18	0.37	0.64	0.93	0.85	1.25	1.99	0.71	0.94	0.74	0.17						
C 19	0.29	0.55	0.69	0.64	0.84	1.42	0.73	0.85	0.74	0.68	0.09					
C 20	0.18	0.45	0.50	0.52	0.58	1.17	0.77	0.68	0.87	0.71	0.19	0.20				
C 21	0.20	0.43	0.45	0.42	0.53	0.90	0.63	0.64	0.76	0.63	0.31	0.32				
C 22	0.15	0.37	0.30	0.30	0.40	0.67	0.56	0.51	0.63	0.50	0.46	0.27	0.08	0.15		
C 23	0.14	0.28	0.21	0.28	0.34	0.49	0.44	0.39	0.49	0.50	0.44	0.26	0.14	0.14		
C 24	0.08	0.22	0.13	0.23	0.24	0.32	0.30	0.33	0.40	0.41	0.33	0.23	0.16	0.15	0.05	
C 25	0.07	0.17	0.18	0.18	0.16	0.28	0.27	0.24	0.30	0.29	0.24	0.22	0.14	0.10	0.06	0.00
C 26	0.08	0.20	0.11	0.09	0.17	0.17	0.16	0.19	0.20	0.22	0.18	0.18	0.11	0.10	0.06	0.02
C 27	0.03	0.10	0.15	0.09	0.11	0.14	0.11	0.17	0.16	0.19	0.15	0.14	0.10	0.11	0.03	0.01
C 28		0.05	0.08	0.07	0.09	0.09	0.08	0.10	0.13	0.13	0.12	0.09	0.09	0.08	0.05	0.03
C 29			0.06	0.05	0.08	0.10	0.05	0.07	0.07	0.07	0.07	0.06	0.07	0.06	0.04	0.00
C 30					0.07	0.08	0.05	0.03	0.08	0.06	0.06	0.05	0.03	0.03	0.02	0.00
C 31						0.03	0.03	0.02	0.03	0.05	0.04	0.04	0.03	0.02	0.02	
C 32						0.02	0.02	0.02	0.03	0.01	0.03	0.01	0.01	0.02		
TOTAL	2.57	7.03	7.72	6.71	10.86	11.83	6.31	5.98	6.77	4.62	2.71	2.07	0.96	0.96	0.33	0.06

 $\Sigma = 77.49$

Table 30 (cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Before Aging

Carbon Number Distribution, Wt.% Aromatic Nitrogens
C_nH_{2n-z}N

<u>C No</u>	<u>Z=13^N</u>	<u>Z=15^N</u>	<u>Z=17^N</u>	<u>Z=19^N</u>	<u>Z=21^N</u>	<u>Z=23^N</u>	<u>Z=25^N</u>	<u>Z=27^N</u>	<u>Z=29^N</u>	<u>Z=31^N</u>
C 12	0.01	0.02								
C 13	0.06	0.04	0.0							
C 14	0.15	0.11	0.02	0.01						
C 15	0.18	0.09	0.08	0.04	0.01					
C 16	0.12	0.11	0.13	0.07	0.05					
C 17	0.11	0.10	0.16	0.08	0.09	0.00	0.00			
C 18	0.08	0.08	0.13	0.11	0.11	0.01	0.02			
C 19	0.07	0.04	0.10	0.12	0.13	0.07	0.02	0.01		
C 20	0.04	0.04	0.08	0.04	0.14	0.06	0.04	0.02		
C 21	0.03	0.02	0.03	0.08	0.06	0.05	0.04	0.03		
C 22	0.02	0.03	0.02	0.05	0.05	0.04	0.03	0.03	0.01	0.01
C 23	0.01	0.03	0.03	0.15	0.03	0.04	0.03	0.02	0.01	0.01
C 24	0.00	0.02	0.01	0.03	0.01	0.04	0.03	0.03	0.01	0.01
C 25		0.02	0.02	0.01	0.03	0.02	0.02	0.02		
C 26			0.01		0.01		0.02	0.00		
C 27							0.01	0.01		
TOTAL	0.88	0.75	0.82	0.79	0.72	0.33	0.26	0.17	0.03	0.03

Σ = 4.78

Table 30 (cont.)

Analysis by High Resolution Mass SpectrometryV-178 Coal-Derived Liquid Before Aging

Carbon Number Distribution, Wt.% Aromatic Oxygens

C No.	z=6 ⁰	z=8 ⁰	z=10 ⁰	z=12 ⁰	z=14 ⁰	z=16 ⁰	z=18 ⁰	z=20 ⁰	z=22 ⁰	z=24 ⁰	z=26 ⁰	z=28 ⁰	z=30 ⁰	z=32 ⁰	z=34 ⁰
C 7	0.04														
C 8	0.10	0.02													
C 9	0.19	0.14													
C 10	0.20	0.32	0.01	0.06											
C 11	0.15	0.37	0.04	0.13											
C 12	0.11	0.32	0.13	0.18	0.01	0.03									
C 13	0.08	0.32	0.21	0.14	0.08	0.12									
C 14	0.07	0.28	0.34	0.11	0.13	0.32	0.03	0.02							
C 15	0.06	0.19	0.38	0.11	0.22	0.35	0.05	0.03							
C 16	0.05	0.14	0.40	0.14	0.17	0.54	0.17	0.04	0.04						
C 17	0.03	0.12	0.30	0.19	0.16	0.43	0.21	0.09	0.09						
C 18		0.07	0.21	0.16	0.14	0.40	0.25	0.14	0.13	0.05					
C 19		0.04	0.21	0.12	0.11	0.26	0.21	0.17	0.17	0.08	0.03				
C 20		0.03	0.06	0.09	0.09	0.22	0.20	0.14	0.18	0.13	0.04	0.02	0.01		
C 21			0.06	0.06	0.07	0.14	0.13	0.12	0.19	0.11	0.05	0.04	0.03		
C 22				0.04	0.05	0.08	0.10	0.09	0.10	0.11	0.07	0.06	0.04	0.01	
C 23				0.03	0.05	0.06	0.06	0.08	0.11	0.09	0.07	0.06	0.04	0.01	
C 24					0.04	0.03	0.05	0.08	0.08	0.08	0.07	0.06	0.04	0.01	0.01
C 25					0.02	0.03	0.03	0.05	0.06	0.03	0.06	0.04	0.03	0.02	
C 26							0.03	0.05	0.03	0.05	0.05	0.03	0.02	0.02	
C 27							0.01	0.02	0.03	0.04	0.04	0.04	0.01	0.01	
C 28								0.01	0.02	0.02		0.02	0.02		
C 29									0.01						
TOTAL	1.08	2.36	2.35	1.56	1.40	3.01	1.53	1.13	1.24	0.79	0.48	0.37	0.24	0.08	0.01

 $\Sigma = 17.63$

Table 30 (cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Before Aging

Carbon Number Distribution, Wt.% Aromatic Sulfurs
 $C_nH_{2n-z}S$

<u>C No.</u>	<u>Z=8^S</u>	<u>Z=10^S</u>	<u>Z=12^S</u>	<u>Z=18^S</u>	<u>Z=20^S</u>	<u>Z=22^S</u>
C 10	0.01	0.02				
C 11		0.03				
C 12			0.02			
C 15				0.00		
C 16					0.02	0.00
TOTAL	0.01	0.05	0.02	0.00	0.02	0.00

$\Sigma = 0.10$

Table 31

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Aged 30 Days

Sample Analysis Summary

Compound Types, $C_nH_{2n-z}(X)$	Avg. C No.	Avg. Mol. Wt.	Avg. z No.	Avg. Rings	Wt-%
Saturated Hydrocarbons (P+N)			Not Determined		
Aromatic Hydrocarbons	20.06	264.49	16.51	2.56	78.16
Aromatic Nitrogens	19.36	266.25	18.91	3.55	4.24
Aromatic Oxygens	17.54	246.49	15.22	2.29	17.53
Aromatic Sulfurs	<u>12.17</u>	<u>179.20</u>	<u>11.97</u>	<u>2.11</u>	<u>0.07</u>
TOTAL	19.58	261.34	16.38	2.56	100.0
Distribution of Aromatic Rings, $C_nH_{2n-z}(X)$, Wt.%	<u>C-H</u>	<u>C-H-N*</u>	<u>C-H-O*</u>	<u>C-H-S*</u>	<u>Total*</u>
1 Ring Aromatics	17.61	0.0	5.55	0.0	23.16
2 Ring Aromatics	28.46	0.51	5.90	0.07	34.94
3 Ring Aromatics	12.32	1.51	2.84	0.0	16.67
4 Ring Aromatics	11.70	1.64	1.99	0.00	15.33
5 Ring Aromatics	6.44	0.55	1.13	0.0	8.12
6 Ring Aromatics	1.47	0.03	0.12	0.0	1.62
7+ Ring Aromatics	0.16	0.0	0.0	0.0	0.16
<u>Elemental Analysis</u>	<u>Wt.%</u>				
Atomic Carbon	89.91				
Atomic Hydrogen	8.72				
Atomic Nitrogen	0.22				
Atomic Oxygen	1.14				
Atomic Sulfur	0.01				
TOTAL	100.0				

*Analysis is approximate

NOTE: Hundredths are shown for normalization purposes only.

(X) Indicates appropriate heteroatom.

Table 32

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Aged 30 Days

Carbon Number Distribution, Wt.% Aromatic Hydrocarbons

C No.	Z=6	Z=8	Z=10	Z=12	Z=14	Z=16	Z=18	Z=20	Z=22	Z=24	Z=26	Z=28	Z=30	Z=32	Z=34	Z=36
C 11	0.04	0.19	0.07	0.12												
C 12	0.05	0.26	0.08	0.25	0.04											
C 13	0.06	0.32	0.27	0.27	0.42	0.03										
C 14	0.11	0.53	0.66	0.33	1.13	0.18	0.11									
C 15	0.13	0.63	0.76	0.38	1.37	0.50	0.31	0.02								
C 16	0.19	0.61	0.76	0.51	1.27	1.15	0.40	0.18	0.41							
C 17	0.28	0.65	0.91	0.75	1.28	1.79	0.55	0.62	0.68							
C 18	0.38	0.62	0.96	0.79	1.15	1.91	0.70	0.90	0.66	0.14						
C 19	0.29	0.55	0.68	0.66	0.79	1.43	0.75	0.85	0.78	0.64	0.12					
C 20	0.23	0.46	0.63	0.53	0.58	1.18	0.73	0.72	0.95	0.72	0.22	0.13				
C 21	0.25	0.39	0.42	0.42	0.52	0.95	0.67	0.59	0.94	0.61	0.33	0.33				
C 22	0.17	0.36	0.28	0.39	0.40	0.65	0.53	0.55	0.70	0.59	0.48	0.32	0.08	0.13		
C 23	0.11	0.23	0.26	0.32	0.36	0.45	0.44	0.44	0.51	0.53	0.44	0.28	0.16	0.19		
C 24	0.10	0.35	0.19	0.24	0.28	0.39	0.36	0.36	0.33	0.42	0.38	0.28	0.18	0.16		
C 25	0.09	0.35	0.16	0.15	0.20	0.32	0.23	0.25	0.31	0.32	0.30	0.30	0.19	0.14	0.07	0.01
C 26	0.09	0.22	0.17	0.12	0.15	0.26	0.19	0.19	0.23	0.26	0.24	0.17	0.13	0.14	0.09	0.03
C 27	0.07	0.20	0.13	0.09	0.11	0.15	0.13	0.14	0.12	0.20	0.15	0.13	0.11	0.11	0.07	0.03
C 28	0.05	0.15	0.07	0.06	0.09	0.14	0.07	0.09	0.11	0.12	0.14	0.10	0.11	0.08	0.04	0.02
C 29	0.04	0.14	0.06	0.05	0.08	0.10	0.06	0.06	0.08	0.09	0.10	0.09	0.06	0.05	0.04	0.03
C 30		0.11	0.04	0.05	0.05	0.06	0.04	0.04	0.05	0.05	0.08	0.07	0.05	0.05	0.03	0.02
C 31					0.02	0.05		0.03	0.03	0.05	0.02	0.04	0.05	0.04	0.01	0.02
C 32								0.02	0.02	0.05	0.00	0.02	0.03	0.02		
C 33													0.02	0.01		
C 34													0.01			
TOTAL	2.73	7.32	7.56	6.48	10.29	11.69	6.27	6.05	6.91	4.79	3.00	2.26	1.18	1.12	0.35	0.16

 $\Sigma = 78.16$

Table 32 (cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Aged 30 Days

Carbon Number Distribution, Wt.% Aromatic Nitrogens

<u>C No.</u>	<u>z=13^N</u>	<u>z=15^N</u>	<u>z=17^N</u>	<u>z=19^N</u>	<u>z=21^N</u>	<u>z=23^N</u>	<u>z=25^N</u>	<u>z=27^N</u>	<u>z=29^N</u>	<u>z=31^N</u>
C 12		0.02								
C 13	0.07	0.05								
C 14	0.07	0.08	0.02	0.02						
C 15	0.03	0.10	0.06	0.02	0.00					
C 16	0.07	0.09	0.11	0.02	0.02					
C 17	0.06	0.10	0.17	0.08	0.04	0.00				
C 18	0.05	0.07	0.11	0.10	0.11	0.02	0.00			
C 19	0.06	0.05	0.09	0.07	0.10	0.05	0.03			
C 20	0.03	0.05	0.11	0.12	0.08	0.05	0.04	0.03		
C 21	0.02	0.03	0.07	0.06	0.09	0.06	0.05	0.04	0.02	
C 22	0.02	0.02	0.03	0.07	0.07	0.06	0.04	0.03	0.02	
C 23	0.03	0.03	0.01	0.03	0.06	0.05	0.02	0.02	0.01	0.01
C 24			0.03	0.04	0.02	0.05	0.04	0.02	0.01	0.01
C 25			0.01	0.01	0.02	0.01	0.03	0.01	0.01	0.01
C 26					0.03		0.03	0.01	0.02	0.00
C 27					0.01		0.01	0.01	0.00	
TOTAL	0.51	0.69	0.82	0.64	0.65	0.35	0.29	0.17	0.09	0.03

$\Sigma = 4.24$

Table 32 (cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Aged 30 Days

Carbon Number Distribution, Wt.% Aromatic Oxygens

C No.	z=6 ⁰	z=8 ⁰	z=10 ⁰	z=12 ⁰	z=14 ⁰	z=16 ⁰	z=18 ⁰	z=20 ⁰	z=22 ⁰	z=24 ⁰	z=26 ⁰	z=28 ⁰	z=30 ⁰	z=32 ⁰
C 7	0.03													
C 8	0.10													
C 9	0.19	0.12	0.01											
C 10	0.19	0.27	0.01	0.03										
C 11	0.11	0.33	0.02	0.12										
C 12	0.11	0.31	0.10	0.14	0.01	0.01								
C 13	0.06	0.23	0.22	0.14	0.07	0.11								
C 14	0.08	0.22	0.36	0.11	0.14	0.27	0.03	0.02						
C 15	0.05	0.22	0.31	0.12	0.18	0.40	0.06	0.02						
C 16	0.02	0.21	0.31	0.14	0.21	0.47	0.15	0.04	0.04					
C 17	0.03	0.13	0.24	0.12	0.17	0.51	0.21	0.10	0.16					
C 18		0.12	0.23	0.18	0.12	0.40	0.28	0.16	0.17	0.05				
C 19			0.17	0.17	0.11	0.26	0.26	0.15	0.18	0.06	0.03			
C 20			0.17	0.08	0.10	0.24	0.19	0.15	0.18	0.09	0.04		0.01	
C 21			0.15	0.06	0.09	0.13	0.13	0.14	0.15	0.10	0.04	0.03	0.02	
C 22			0.12	0.05	0.08	0.09	0.12	0.11	0.09	0.12	0.09	0.06	0.03	
C 23				0.04	0.02	0.07	0.08	0.10	0.08	0.09	0.08	0.06	0.05	0.01
C 24				0.03	0.02	0.05	0.07	0.07	0.07	0.08	0.08	0.05	0.05	0.02
C 25						0.04	0.04	0.05	0.04	0.06	0.06	0.04	0.03	0.03
C 26							0.03	0.04	0.03	0.03	0.04	0.03	0.03	0.02
C 27							0.01	0.02	0.02	0.03	0.02	0.03	0.02	0.02
C 28								0.01	0.03	0.01	0.01	0.02	0.02	0.01
C 29									0.01	0.01	0.01	0.01	0.02	0.01
C 30									0.01			0.01	0.01	
TOTAL	0.97	2.16	2.42	1.53	1.32	3.05	1.66	1.18	1.26	0.73	0.50	0.34	0.29	0.12
														Σ = 17.53

Table 32 (cont.)

Analysis by High Resolution Mass Spectrometry

V-178 Coal-Derived Liquid Aged 30 Days

Carbon Number Distribution, Wt.% Aromatic Sulfurs

$C_nH_{2n-z}S$						
<u>C. No.</u>	<u>z=4^S</u>	<u>z=6^S</u>	<u>z=8^S</u>	<u>z=10^S</u>	<u>z=12^S</u>	<u>z=20^S</u>
C 10				0.01		
C 11				0.03		
C 12					0.03	
C 16						0.00
TOTAL				0.04	0.03	0.03
$\Sigma = .07$						

Table 33

Results of Chemiluminescence Study at 100°C (212°F)
of V-178 Coal-Derived Liquid Before and After Aging

<u>Time, Mins.</u>	<u>Photon Counts/second</u>	
	<u>Before Aging</u>	<u>After Storage,</u>
		<u>30 Days, 110°F</u>
0	-	-
0.5	8	15
1.0	19	28
2.0	36	48
3.0	61	59
5.0	100	101

Table 34

Results of Chemiluminescence Study at 150°C (302°F)
of V-178 Coal-Derived Liquid Before and After Aging

<u>Time, Mins.</u>	<u>Photon Counts/second</u>	
	<u>Before Aging</u>	<u>After Storage,</u> <u>30 day, 110°F.</u>
0	-	-
0.5	12	11
1.0	28	20
2.0	44	38
3.0	77	60
5.0	114	102
10.0	221	212
15.0	328	339

Table 35

ESR Study of

V-178 Coal-Derived Liquid Before and After Aging

	<u>Peak Height</u>	<u>g-Value</u>
Fresh Coal Liquid	132	2.00300
Aged Coal-Derived Liquid, 30 days, 110°F	1166	2.00325

FLOW DIAGRAM OF WILSONVILLE ADVANCED COAL LIQUEFACTION FACILITY

Fig. 2 CORROSION TEST APPARATUS

Figure 3

Autoclave for Dynamic Corrosion Tests

Fig. 4 Machineable Ceramic Autoclave Inserts

Fig. 5 Corrosion Deposit on Type 304L
Coupon After Cleaning With Brass
Wool, Toluene: Alcohol and 10%
Nitric Acid Etch. Mag = 1000X

Fig. 6 IR Spectra of 5 Cr-0.5 Mo
Corrosion Deposit

Fig. 7 Photoacoustic Record of
5 Cr-0.5 Mo Corrosion Deposit

EG&G PRINCETON APPLIED RESEARCH

Fig. 8 Synchronous Fluorescence Spectrum
for After 700°F Test Coal-Derived
Liquid n-C₅ Insolubles

Fig. 9 Synchronous Fluorescence Spectrum for DMF-Toluene
Extract of 5 Cr-0.5 Mo Steel Corrosion Product (625°F)

Fig. 10 Synchronous Fluorescence Spectrum of DMF-Toluene Extract of 1010 Steel Corrosion Product (550°F)

Fig. 11 Synchronous Fluorescence Spectrum of DMF-Toluene
Extract of Corrosion Product from Type 316L Steel (625°F)

Fig. 12 Synchronous Fluorescence Spectrum of DMF-Toluene
Extract of Corrosion Product from Type 316L Steel (700°F)

Cross-Section

Sulfur DOT Pattern

Iron DOT Pattern

Chromium DOT Pattern

Fig. 13 EDX Spot Sulfur, Iron and Chromium
Pattern of Cross-Section of 5 Cr-0.5 Mo
Coupon Exposed in V-178 Coal-Derived
Liquid at 550°F for 100 hours. Mag = 1000X

Fig. 14 Total Ionization (TI) Plots of Corrosion Film, H_2O and CO_2

Fig. 15 Typical Mass Spectrum of 5 Cr-0.5 Mo Corrosion Product

FRN 12068

Fig. 16 X-ray Diffraction Pattern of 5 Cr-0.5 Mo Corrosion Product

Static

Dynamic

1000X

1000X

Fig. 17 SEM Photograph and Metallographic Cross-Section Views of 1010 Steel After Exposure in V-178 Coal-Derived Liquid at 550°F for 100 hours.

Static

Dynamic

1000X

1000X

500X
ETCHED

Fig. 18 SEM Photograph and Metallographic Cross-Section Views of 1010 Steel after Exposure in V-178 Coal-Derived Liquid at 625°F for 100 hours.

Static

1000X

500X
ETCHED

Dynamic

1000X

Fig. 19 SEM Photograph and Metallographic Cross-Section Views of 5 Cr-0.5 Mo after Exposure in V-178 Coal-Derived Liquid at 550°F for 100 hours

Static

Dynamic

1000X

Fig. 20 SEM Photographs of 5 Cr-0.5 Mo Steel after Exposure in V-178 Coal-Derived Liquid at 625°F for 100 Hours

Static

Dynamic

1000X

1000X

Fig. 21 SEM Photographs and Metallographic Cross-Section Views of Type 304L After Exposure in V-178 Coal-Derived Liquid at 625°F for 100 Hours.

Static

1000X

Dynamic

1000X

500X
ETCHED

Fig. 22 SEM Photographs and Metallographic Cross-Section Views of Type 304L After Exposure in V-178 Coal-Derived Liquid at 700°F, for 100 Hours.

Fig. 23 SEM Photograph and Metallographic Cross-Section Views of Type 304L After Exposure in V-178 Coal-Derived Liquid at 750°F for 100 Hours.

Static

Dynamic

1000X

Fig. 24 SEM Photographs of Type 316L After
Exposure in V-178 Coal-Derived Liquid
at 625°F for 100 Hours.

Static

1000X

1000X

500X
ETCHED

Dynamic

Fig. 25 SEM Photographs and Metallographic Cross-Section Views of Type 316L After Exposure in V-178 Coal-Derived Liquid at 700°F for 100 Hours.

1000X

1000X

500X
ETCHED

Type 304L

Type 316L

Fig. 26 SEM Photographs and Metallographic Cross-Section Views of Type 304L and Type 316L After Exposure in V-178 Coal-Derived Liquid at 625°F for 400 hours. Static Only.

Fig. 27 DTA-DSC of Original V-178 Coal-Derived Liquid

Fig. 28 DTA-DSC of V-178 Coal-Derived Liquid
After Static Test at 625°F and 100 Hours.

Fig. 29 DTA-DSC of V-178 Coal-Derived Liquid
After Dynamic Test at 625°F and 100 Hours

Fig. 30

Block Diagram of Chemiluminescence Apparatus

Fig. 31 DTA-DSC of V-178 Coal-Derived Liquid
Before Aging

Fig. 32 DTA-DSC of V-178 Coal-Derived Liquid After Aging
at 110°F for 30 Days

IX. APPENDIX

Corrosion Data Assessment

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Oak Ridge National Laboratory

The results were compared with some of the earlier tests done by Southwest Research under MPC auspices and some laboratory tests done by ORNL. The objectives of these comparisons were to compare the data from this report with earlier tests in process solvent and note any differences in liquid analyses where available.

An in-depth comparison of liquid chemistry would require a literature search. However, some data from the Southwest Research activities and the UOP V-178 analysis are presented here.

Table A-1

Analysis	Sample ID ^a			
	V-178	A 511	B 312	F 216
Acid number (mg KOH/g)	0.11	0.1	0.11	0.11
Elemental Analysis				
Sulfur (wt %)	0.27	0.45	0.42	0.12
Nitrogen (wt %)	0.27	0.76	1.02	1.03
Chlorine (ppm)	71	95	325	14
Phenol, Total oxygenates (wt %)	0.8			
Phenol, % as p-cresol		7.1	6.7	4.04

^aSample ID

V-178, Wilsonville, Tray 8, T-102, two-stage; Ill. No. 6

A 511, Wilsonville, T-105 bottoms, single-stage, Ky. No. 9

B 312, Ft. Lewis, Wash Solv. Col. bottoms, Ky. No 9/11 mix

F 216, Catlettsburg, fractionation bottoms, Ill. No. 6

In addition to the obvious differences in chlorine and sulfur contents which can be related to the feed coal, there are significant differences in the nitrogen and apparently the phenol contents of these liquids. This could be very significant in that the Southwest program utilized a factorial experiment with synthetic solutions to show that corrosion rates of carbon steel are affected by total nitrogen (amines), chloride, and phenol contents. These synthetic mix static corrosion tests also indicated the "the corrosion rate of carbon steel in solutions with similar amine hydrochloride concentrations were found to be essentially independent of temperature between 275 and 500°F". This is significant in evaluating the current tests which involved a liquid derived from hydrotreated solvent with a resultant lower nitrogen content in the coal-derived oil tested than that found in the pilot plant process solvents. Since the pilot plant liquids were derived by recycling unhydrotreated solvent the resulting total nitrogen content was higher. Since the process solvent for the Demonstration Plant Process is derived from nonhydrotreated oil fractionations, it can be expected to contain a higher total nitrogen. Higher amine contents may also increase the quantity of chloride retained in the oil streams. Further analysis of available data and probably additional testing will be required to assess this possibility.

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