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## **Ecotoxicity of Materials from Integrated Two-Stage Liquefaction and Exxon Donor Solvent Processes**

**D. D. Dauble, Principal Investigator**

**A. J. Scott**

**E. W. Lusty**

**B. L. Thomas**

**R. W. Hanf, Jr.**

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ECOTOXICITY OF MATERIALS FROM INTEGRATED  
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Richland, Washington 99352

## SUMMARY

Coal-derived materials from two coal conversion processes were screened for potential ecological toxicity. We examined the toxicity of materials from different engineering or process options to an aquatic invertebrate and also related potential hazard to relative concentration, composition, and stability of water soluble components.

For materials tested from the Integrated Two-Stage Liquefaction (ITSL) process, only the LC finer (LCF) 650°F distillate was highly soluble in water at 20°C. The LCF feed and Total Liquid Product (TLP) were not in liquid state at 20°C and were relatively insoluble in water. Relative hazard to daphnids from ITSL materials was as follows: LCF 650°F distillate > LCF feed > TLP. For Exxon Donor Solvent (EDS) materials, process solvent produced in the bottoms recycle mode was more soluble in water than once-through process solvent and, hence, slightly more acutely toxic to daphnids. When compared to other coal liquids or petroleum products, the ITSL or EDS liquids were intermediate in toxicity; relative hazard ranged from 1/7 to 1/13 of the Solvent Refined Coal (SRC)-II distillate blend, but was several times greater than the relative hazard for No. 2 diesel fuel oil or Prudhoe Bay crude oil. Although compositional differences in water-soluble fractions (WSF) were noted among materials, phenolics were the major compound class in all WSFs and probably the primary contributor to acute toxicity.

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## INTRODUCTION

Recent studies on coal conversion technologies have focused on evaluating effects of process modifications or new design options to minimize the genotoxic potential of coal-derived materials (Pelroy and Wilson 1981; Wilson, Pelroy and Mahlum, in press; Wilson, Buhl and Moroni, in press). No comparable data base exists for comparing the ecological risk of different process options. Although chemical components of parent materials may indicate toxic activity or mutagenicity in biological systems, many compound classes in coal-derived materials are insoluble in water and pose little direct hazard to aquatic organisms. On the other hand, materials shown to have little biological activity in test systems may be hydrophylic, and thus available and potentially toxic to aquatic biota. For this reason, no single test or biological system can provide information indicative of the overall environmental hazard of a material.

The objective of our ecological toxicity screening studies was to help identify engineering strategies that may be used to reduce concentrations of toxic or bioactive agents in coal conversion materials. During initial stages of development, several process options may be available. Each process produces a material inherently different in chemical and toxicological properties. Our research focused on characterizing water-soluble components derived from materials produced by the Integrated Two-Stage Liquefaction (ITSL) and Exxon Donor Solvent (EDS) processes. Differences in chemistry were related to observed acute toxicity to the freshwater invertebrate Daphnia magna.

## MATERIALS AND METHODS

Six coal-derived materials were tested (Table 1). Three of the materials originated from the ITSL process: LC finer (LCF) feed, total liquid product (TLP), and LCF 650°F distillate. The ITSL materials were obtained from the C. E. Lummus Process Development Unit in New Brunswick, New Jersey. A description of the ITSL process conditions and the chemical characteristics of the parent material are summarized by Wilson, Pelroy and Mahlum (in press). The two EDS materials were recycle solvents produced under the bottoms recycle and once-through modes of operation. The EDS liquids were obtained from the 250 T/D pilot plant in Baytown, Texas (Wade et al. 1982). For purposes of comparison, tests were also conducted with a well-characterized coal liquid

TABLE 1. Materials Used for Ecotoxicity Testing and Comparison of Solubility Characteristics as Defined by Total Carbon Concentration in the Water-Soluble Fractions (WSF)

<u>Material</u>	<u>Coal Type</u>	<u>Physical State</u>	<u>Nominal Boiling Range (°F)</u>	<u>WSF Total Carbon (mg/L)(a,b)</u>
ITSL				
LCF feed	Illionis No. 6	solid	650-850 <sup>+</sup>	7
TLP	Illinois No. 6	variable	650-850	7
LCF 650°F distillate	Illinois No. 6	liquid	<650	124 ± 8
EDS process solvent				
once through	Illinois No. 6	liquid	400-800	103 ± 7
bottoms recycle	Illinois No. 6	liquid	400-900	163 ± 1
SRC-II				
distillate blend	Powhatan No. 5	liquid	150-850 <sup>+</sup>	1068 ± 33

(a) As measured by a carbon analyzer.

(b) Values expressed as mean ± S.D.

derived from the Solvent Refined Coal (SRC)-II process (PNL 1980). This 2.9:1 blend of middle to heavy SRC-II distillate was obtained from a pilot plant in Fort Lewis, Washington.

The water-soluble fractions (WSF) were generated by adding 100 ml of coal liquid to 10 L of well water (1:100 ratio) in a 15-L glass carboy. The mixture was stirred at 90 rpm with a Teflon®-coated rod for 4 hr. Because two of the ITSL materials were not in liquid state, these materials were added on a weight-to-weight basis at the same mix ratio as the liquids. The LCF feed (solid) was ground into a fine powder with a blender before it was added to the carboy. The TLP (tar-like state) was spread over the bottom of the carboy before mixing. After a 1-hr settling period, 8 L of the respective WSFs were siphoned from the center of the carboy, chemically analyzed, and used for ecological testing. Exposure solutions for testing were made by diluting WSFs with well water. Dilutions were based on total carbon concentrations in the WSF (stock solution). New stock solutions were generated for replicate tests.

Total carbon (TC) of the WSFs was determined by direct aqueous injection into a Beckman 915B carbon analyzer. Detailed chemical analysis of the WSFs by gas chromatography was conducted to determine phenol and hydrocarbon composition (methodology outlined in Dauble et al. 1982). Total phenols in selected test exposure solutions were estimated by the direct photometric method (APHA 1976) at test initiation and again at 48 hr to monitor the relative stability of solubilized materials.

Acute toxicity of WSFs derived from the test materials was determined by static bioassays with the freshwater invertebrate, Daphnia magna. All tests were conducted at 20°C with a 16:8 hr, light:dark cycle. To initiate a test, five first-instar D. magna were placed in each of four 100-ml capacity glass jars at six treatment levels plus a control (4 x 7 matrix, 20 organisms per treatment level). For tests involving toxicological comparison of process conditions within general material classes (ITSL and EDS), paired tests were conducted concurrently. The 48-hr LC<sub>50</sub> determinations were made by the graphical method (APHA 1976). Toxicity thresholds were expressed as percent WSF and TC based on dilution of measured stock solution.

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CHEMICAL CHARACTERIZATION OF TEST MATERIALS

INTEGRATED TWO-STAGE LIQUEFACTION (ITSL) PROCESS MATERIALS

Water-soluble fractions (WSF) derived from the three ITSL materials consisted primarily of phenols. Total phenol concentrations of WSFs ranged from less than 1.0 mg/L in the LCF feed to 72.3 mg/L in the LCF 650°F distillate. Phenolic composition of LCF feed and LCF distillate WSFs was similar; C<sub>2</sub>-C<sub>6</sub> phenols comprised 70% to 80% of the total phenols in solution. In contrast, WSFs from the TLP contained more lighter molecular weight phenols, predominately cresols and C<sub>2</sub> and C<sub>3</sub> phenols (Table 2).

TABLE 2. Phenol and Aromatic Hydrocarbon Concentrations in Water-Soluble Fractions Derived from Three ITSL Materials. Concentrations expressed as means of duplicate samples.

Phenols (mg/L)				Aromatic Hydrocarbons (mg/L)			
Phenols (mg/L)	LCF 650 F Distillate	LCF Feed	TLP		LCF 650 F Distillate	LCP Feed	TLP
Phenol	0.53	0.01	0.32	C <sub>2</sub> benzenes	231.40	0.34	87.85
Cresols	4.94	0.05	0.78	C <sub>3</sub> benzenes	83.10	0.97	25.27
C <sub>2</sub> phenols	29.23	0.05	0.82	C <sub>4</sub> benzenes	112.70	11.54	53.88
C <sub>3</sub> phenols	22.38	0.14	0.67	Naphthalene	60.00	37.05	15.77
C <sub>4</sub> phenols	7.30	0.09	0.21	Benzothiofene	1.50	0.28	1.32
C <sub>5</sub> /C <sub>6</sub> phenols	4.54	0.11	0.20	C <sub>1</sub> naphthalene	49.60	7.90	14.64
Indanols	2.28	0.05	0.19	B <sub>1</sub> phenyl	nd	1.70	4.69
C <sub>1</sub> Indanols	1.20	0.04	0.13	C <sub>2</sub> naphthalenes	26.50	3.45	6.96
C <sub>2</sub> Indanols	nd	0.02	0.04	C <sub>3</sub> naphthalenes and C <sub>1</sub> , C <sub>2</sub> fluorines	66.80	43.55	25.72
Total phenols	72.40	0.56	3.36	Dibenzothiofene	6.60	8.45	9.24
				Phenanthrene	9.00	24.81	4.89
				Anthracene	0.70	3.73	0.24
				C <sub>1</sub> phenanthrene	2.50	9.55	2.40
				Fluoranthene	1.10	7.69	1.22
				Pyrene	7.00	18.44	9.65
				Total aromatic hydrocarbons	658.50	179.45	263.74

nd = not detectable.

Aromatic hydrocarbon content was low in all WSFs and comprised less than 3% of the TC in solution. The ratio of aromatic hydrocarbons to total phenols was highest in WSFs derived from the LCF feed process material (Table 2). Composition of soluble aromatic hydrocarbons was similar for TLP and LCF distillate process materials. The major hydrocarbon constituents in WSFs derived from both materials were C<sub>2</sub>-C<sub>4</sub> benzenes, which comprised about 65% of the total aromatics. Naphthalene and C<sub>3</sub> naphthalene were the major hydrocarbon constituents in the LCF feed WSF. Phenanthrene and pyrene were also present and collectively comprised 24% of the total aromatic hydrocarbons in the WSF (Table 2).

#### EXXON DONOR SOLVENT (EDS) PROCESS MATERIALS

Although compositional differences were noted, phenols were the major water-soluble components in the two EDS process materials. Both WSFs contained primarily low molecular weight compounds; however, phenol and cresols comprised a higher percentage of the total phenols in WSFs derived from the recycle solvent produced in the once-through compared to the bottoms recycle mode of operation (Table 3).

Total aromatic hydrocarbon concentrations were approximately four times greater in WSFs derived from EDS recycle solvent under bottoms recycle than under once-through operations. Naphthalene and C<sub>1</sub> naphthalene were major aromatic hydrocarbon constituents of both WSFs, comprising about 40% of the total. The C<sub>2</sub>-C<sub>4</sub> benzenes comprised about 40% of the aromatic hydrocarbons in WSFs of once-through materials, compared to only 9% of the total in WSFs derived from the bottoms recycle material.

Overall, it appears that WSFs derived from EDS recycle solvents produced in the once-through mode are less concentrated and contain a higher percentage of low molecular weight components than WSFs derived from the bottoms recycle materials (Table 3).

#### REFERENCE MATERIAL: SOLVENT REFINED COAL (SRC-II) LIQUID

Data on the composition of WSFs from the SRC-II liquid are provided in Table 4 to facilitate comparison with ITSL and EDS materials. Phenols were the

TABLE 3. Phenol and Aromatic Hydrocarbon Concentrations in Water-Soluble Fractions (WSF) Derived from Two EDS Recycle Solvents. Concentrations expressed as means of duplicate samples.

	Phenols (mg/L)			Aromatic Hydrocarbons (µg/L)	
	Once-Through Recycle Mode	Bottoms Recycle Mode		Once-Through Recycle Mode	Bottoms Recycle Mode
Phenol	7.36	1.00	C <sub>2</sub> benzenes	13.2	48.5
Cresols	16.89	8.51	C <sub>3</sub> benzenes	20.8	25.9
C <sub>2</sub> phenols	17.38	40.40	C <sub>4</sub> benzenes	285.1	196.4
C <sub>3</sub> phenols	12.83	45.10	Naphthalene	170.9	767.2
C <sub>4</sub> phenols	3.11	13.26	Benzothiophene	11.9	20.9
C <sub>5</sub> phenols	4.28	9.98	C <sub>1</sub> naphthalene	158.9	506.8
Indanols	3.78	9.01	C <sub>2</sub> naphthalene	70.2	699.6
C <sub>1</sub> indanols	<u>2.49</u>	<u>3.86</u>	C <sub>3</sub> naphthalenes and C <sub>1</sub> , C <sub>2</sub> fluorenes	63.7	608.9
Total Phenols	68.12	131.12	Dibenzothiophene	<0.8	45.6
			Phenanthrene	<0.2	62.0
			Anthracene	<0.2	<0.8
			C <sub>1</sub> phenanthrene	5.3	19.4
			Fluoranthene	<1.3	<0.7
			Pyrene	<u>&lt;3.4</u>	<u>8.0</u>
			Total aromatic hydrocarbons	800.0	3009.2

**TABLE 4.** Concentrations of Phenols and Aromatic Hydrocarbons in WSF Derived from an SRC-II Liquid. Concentrations expressed as means of duplicate samples.

Phenols			Aromatic Hydrocarbons		
Compound	Concentration (mg/L)	% of Total	Compound	Concentration (mg/L)	% of Total
Phenol	172.08	16.7	C <sub>2</sub> benzenes	1106.6	12.8
Cresols	353.51	34.3	C <sub>3</sub> benzenes	615.7	7.1
C <sub>2</sub> phenols	255.79	24.8	C <sub>4</sub> benzenes	1078.8	12.5
C <sub>3</sub> phenols	129.44	12.5	Naphthalene	2901.9	33.5
C <sub>4</sub> phenols	24.05	2.3	Benzothiofophene	81.3	0.9
C <sub>5</sub> phenols	18.02	1.7	C <sub>1</sub> naphthalenes	1292.2	14.9
Indanols	62.45	6.1	C <sub>2</sub> naphthalenes	1146.8	13.2
C <sub>1</sub> indanols	<u>16.19</u>	<u>1.6</u>	C <sub>3</sub> naphthalenes and C1, C2 fluorines	306.0	3.5
Total phenols	1031.53	100.0	Dibenzothiofophene	<4.3	<0.1
			Phenanthrene	128.0	1.5
			Anthracene	<1.2	<0.1
			C <sub>1</sub> phenanthrene	<1.3	<0.1
			Fluoranthene	<1.1	<0.1
			Pyrene	<u>&lt;1.1</u>	<u>&lt;0.1</u>
			Total aromatic hydrocarbons	8657.3	100.0

major chemical class present in the SRC-II WSF. Concentrations of total phenols were about 100 times greater than total aromatic hydrocarbon concentrations. The WSF was predominated by cresols and C<sub>2</sub> phenols, which comprised almost 60% of the total phenols. Aromatic hydrocarbons were dominated by various naphthalenes (approximately 60% of the total) and C<sub>2</sub>-C<sub>4</sub> benzenes.

Concentrations of total phenols and total aromatic hydrocarbons in the SRC-II WSF were higher than those observed for WSFs derived from ITSL and EDS process materials. Table 5 compares the concentration of total carbon and the composition by major chemical classes for WSFs from the six materials analyzed. All WSFs were predominated by phenolics; aromatic hydrocarbons were minor components, with the exception of WSFs derived from the solid ITSL material. When the TC determined by GC (Table 5) is compared to that obtained by the carbon analyzer (Table 1), it appears that all carbon cannot be accounted for in the phenolic and saturate/aromatic hydrocarbon classes. Additional carbon may be present as heteroatom groups, such as nitrogen-containing compounds.

TABLE 5. Composition of Major Chemical Classes in WSFs Derived from Materials Used for Ecotoxicity Testing

<u>Material</u>	<u>Total Carbon (mg/L)</u>	<u>% Phenols</u>	<u>% Hydrocarbons</u>	
			<u>Saturates</u>	<u>Aromatics</u>
ITSL				
LCF feed	0.8	71.3	<0.1	22.5
Total liquid product	3.7	91.8	<0.1	7.1
LCF 650°F distillate	73.0	99.0	<0.1	1.0
EDS process solvent				
Once through	68.9	98.8	<0.1	1.2
Bottoms recycle	134.5	97.5	0.2	2.3
SRC-II				
Distillate blend	1040.4	99.1	<0.1	0.8

## PHENOLIC DEGRADATION IN TEST SOLUTIONS

Concentrations of total phenols in all test solutions declined rapidly. At initial test concentrations of less than 5 mg/L TC, only about 32% to 79% of the phenols remained in solution after 48 hr (Figure 1). Phenol concentrations in dilutions of EDS WSFs were approximately 50% of day 0 values after 48 hr. Test solutions from soluble ITSL material components exhibited varying stability. Phenols solubilized from the LCF feed, although present in relatively low concentrations, appeared more stable than phenols solubilized from other ITSL materials. Fewer of the original phenolics were present in test solutions derived from the SRC-II material than those from EDS or ITSL materials.

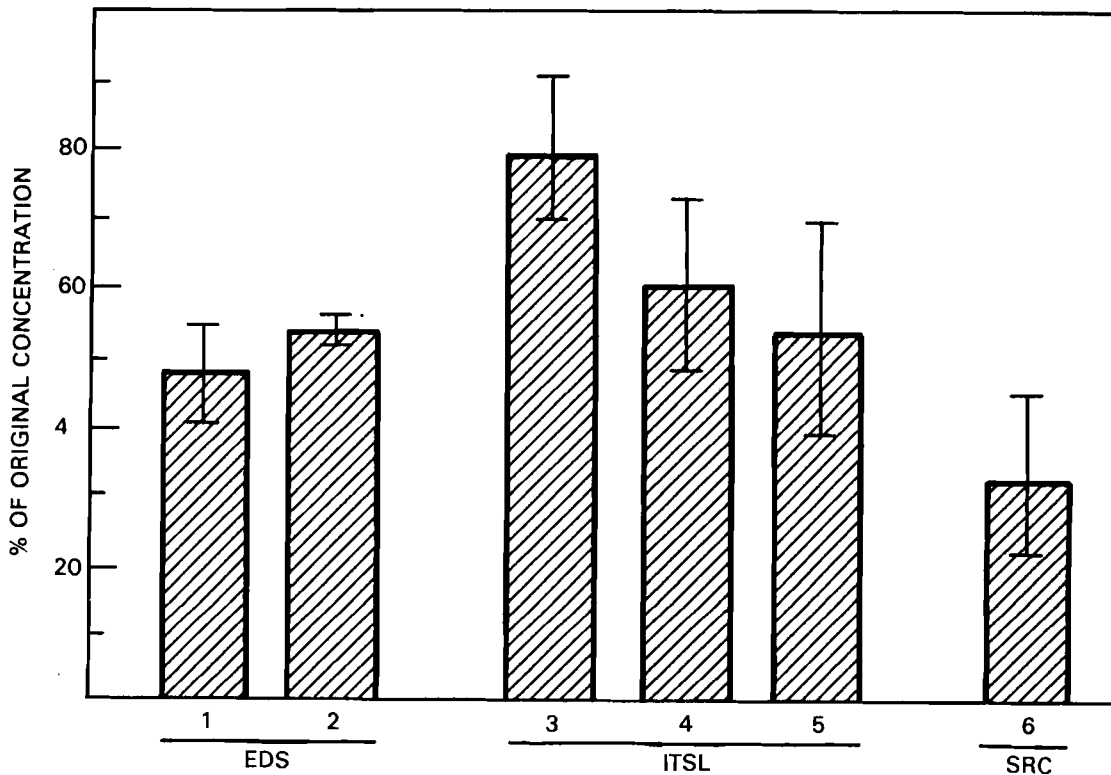


FIGURE 1. Loss of Total Phenolic Concentrations in Static WSF Test Solutions after 48 Hr as Measured by Dye Photometry. Key: 1 = once-through mode, 2 = bottoms recycle mode, 3 = LCF feed, 4 = TLP, 5 = LCF 650°F distillate, 6 = SRC-II distillate blend. Vertical lines represent  $\pm 1$  standard deviation of the mean.

## ECOTOXICITY TESTS

### INTEGRATED TWO-STAGE LIQUEFACTION PROCESS MATERIALS

Based on percent dilution of the WSF, the LCF 650°F distillate was more toxic than the other ITSL materials tested (Table 6). The acute LC<sub>50</sub> for Daphnia magna ranged from 6.1% to 9.0% of the WSF for the LCF distillate, compared to 71.6% and 85.0% of the WSF for LCF feed and TLP, respectively. When toxicity comparisons were made relative to TC in solution, WSFs generated from the LCF feed material appeared slightly more toxic than other ITSL materials (Table 6). The acutely toxic range for ITSL materials was fairly narrow and ranged from 4.9 to 11.8 mg/L TC.

TABLE 6. Acute Toxicity of WSF ITSL Process Materials to Daphnia magna. (Values given as single test comparisons or as range of replicate tests.)

<u>Material</u>	<u>48-hr LC<sub>50</sub></u>	
	<u>% WSF</u>	<u>Total Carbon (mg/L)</u>
LCF feed	71.6	4.9
Total liquid product	85.0	7.4
LCF 650°F distillate	6.1-9.0	7.1-11.8

### EXXON DONOR SOLVENT MATERIALS

No significant difference in acute toxicity to daphnids was observed for WSFs of the two EDS materials. Based on percent dilution of the WSF, LC<sub>50</sub> values were similar, ranging from 2.2% to 7.2% of the WSF (Table 7). Acute toxicity thresholds based on TC in solution were also similar for both EDS materials.

TABLE 7. Acute Toxicity of WSF or EDS Process Materials to Daphnia magna. (Values given as range of duplicate tests.)

<u>Process Mode</u>	<u>48-hr LC<sub>50</sub></u>	
	<u>% WSF</u>	<u>Total Carbon (mg/L)</u>
Once through	2.2-7.2	2.4-7.8
Bottoms recycle	2.2-5.4	3.6-8.8

REFERENCE MATERIAL

Based on percent dilution of the WSF, the SRC-II material was more toxic to daphnids than WSFs of ITSL or EDS materials. The acute LC<sub>50</sub> ranged from 0.4% to 0.7% of the SRC-II WSF. Based on TC, however, similar toxicities were noted for daphnids exposed to WSFs of all six test materials. Overlapping LC<sub>50</sub> values were observed for the replicate test series.



## DISCUSSION

All materials evaluated in these studies were treated identically, thus each had equal opportunity to enter solution. Toxicity of WSFs of each material was, therefore, highly dependent on the solubility of the parent material and the toxic properties of individual soluble components. Within material (process) types, the three ITSL materials showed the widest range of physical states. These physical properties directly influenced solubility in water and acute toxicity to daphnids. Low solubility of the LCF feed and TLP was reflected in low TC concentrations in WSFs. Readily soluble phenolic constituents were present in WSFs of all materials; however, hydrocarbon contribution was greatest in WSFs of the LCF feed. Because of higher concentrations in LCF 650°F distillate WSFs, acute mortality to daphnids was noted at greater dilutions of the WSF than for other ITSL materials.

The EDS process solvent produced in the bottoms recycle mode contained more material that could be extracted in water than did the EDS process solvent produced in the once-through mode. This was reflected by higher TC concentrations in similarly derived WSFs. Potential ecological hazard of the two materials was similar, and the range of toxicity values overlapped. However, the bottoms recycle material can be expected to be more persistent in the aquatic environment because it contains relatively higher concentrations of aromatic hydrocarbons and higher molecular weight phenols.

Potential ecological risk is also influenced by the stability of soluble components of these materials. For example, components that are easily degraded by bacteria or through photolysis would pose little long-term hazard after discharge to water. Since all materials tested exhibited some loss of phenols (the major water soluble constituents) in only 48 hr, potential toxicity can be expected to decrease with time. Previous studies at PNL (Dauble et al. 1982) showed that degradation of various phenol classes was related to molecular weight; lower molecular weight compounds (i.e., phenol, cresol) exhibited greatest degradation over time. Thus, some materials with potential for high acute toxicity may pose little long-term risk. However, materials containing higher substituted and heavier molecular weight compounds can be

expected to be more persistent in the environment and contribute to chronic effects associated with long-term exposures.

Toxicity of EDS and ITSL WSFs can be compared with other fossil-derived materials or selected chemicals. Giddings and Washington (1981) examined a wide range of petroleum and coal liquefaction materials and found that, based on percent dilution of the WSF, petroleum products were less toxic to algae than were bituminous materials. Gray et al. (1982) and States et al. (1982) reported similar results in chronic exposure studies with algae and daphnids, respectively, and noted that inherent toxicity was mainly related to the presence of highly soluble toxic phenolic constituents in WSFs of coal liquids. The three liquids tested here (i.e., LCF 650°F distillate, EDS process solvents) were less soluble in water and less toxic than either a shale oil (personal communication, C. D. Becker, PNL) or a crude oil (Wong, Engelhardt and Strickler 1981). Observed differences in toxicity may be a result of compositional differences in WSFs.

Since all WSFs tested were complex mixtures of several organic compound classes, toxicity cannot be attributable to any one chemical class. Instead, biological responses are a result of exposure to interacting toxic components. Although lower molecular weight phenolics comprised a relatively high percentage of the TC in solution for all WSFs, WSFs generated from ITSL and EDS materials were more toxic to daphnids than were phenol or various cresols (DeGraeve et al. 1980).

Results of toxicity studies with a variety of materials such as shale oil, crude and refined petroleum, and coal liquids are summarized in Table 8. Data indicate that the ITSL and EDS liquids were less toxic than SRC-II liquids, but more toxic than refined petroleum products. The non-liquid ITSL materials were fairly insoluble and exhibited less toxicity than all but the refined petroleum products. Future studies will be used to evaluate materials from various two-stage liquifaction and other processes for potential acute ecotoxicity.

Our experiments were conducted under standard conditions to allow comparison of results. Other factors known to influence toxicity of complex organic materials to daphnids include mixing regimes (Bean et al. 1982), water temperature (Ullrich and Millemann 1983) and water quality (Becker et al. 1983).

TABLE 8. Comparative Toxicity to Daphnids of ITSL, EDS and Several Reference Materials. Relative solubility indicated by total carbon (TC) in WSF generated under similar conditions. LC<sub>50</sub> and WSF value given as mean of reported values.

Material	Concentration of WSF (mg/L)	48-hr LC <sub>50</sub>		RH <sup>(a)</sup>	Reference
		% Dilution	TC (mg/L)		
SRC-II distillate blend	1068	0.6	6.3	1	Our data
ITSL - LCF feed	7	71.6	4.9	1/121	Our data
ITSL - TLP	9	85.0	7.4	1/141	Our data
ITSL - LCF 650°F distillate	124	7.6	9.5	1/13	Our data
EDS process solvent once through	103	4.7	5.1	1/8	Our data
EDS process solvent bottoms recycle	161	3.8	6.2	1/7	Our data
Shale oil	65	10.4	16.0	1/42	Becker <sup>(b)</sup>
No. 2 diesel fuel	9	None at 100% WSF	--	<1/169	States et al. (1981)
No. 6 bunker feed stock	5	≈47.0	≈2.4	1/80	States et al. (1981)
Prudhoe Bay crude oil	3	None at 100% WSF	--	<1/169	Strand and Vaughan (1981)

(a) RH = relative hazard as compared to SRC-II distillate blend (after States et al. 1981).

(b) Personal communication, C. D. Becker, PNL.

Therefore, while these investigations are adequate for indicating relative hazard to aquatic life, more thorough studies with other organisms, or chronic exposure studies would be useful for assessing long-term environmental risk.

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