

UNPRESENTED PAPERS

PROBLEMS ASSOCIATED WITH THE ANALYSIS OF SYNFUELS †
PRODUCT, PROCESS, AND WASTEWATER STREAMS

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ABSTRACT

Hittman Associates, Inc., as part of an environmental assessment of coal liquefaction technology sponsored by the U.S. Environmental Protection Agency (EPA), performed various analyses on samples from the Solvent Refined Coal II (SRC-II) plant in Ft. Lewis, Washington, and the Exxon Donor Solvent Plant (EDS) plant in Baytown, Texas. This paper describes several of the problems encountered in these analyses and methods taken to mitigate them. Recommendations are made on approaches for avoiding such problems.

INTRODUCTION

Though there exist standard methodologies for the analysis of pure organic extracts, the complex nature of the product and process streams from synfuels plants cause specific problems which are often complicated by the analytical requirements of the particular study. This paper addresses several problems encountered in the analyses of samples from the SRC-II pilot plant in Ft. Lewis, Washington, and the EDS facility in Baytown, Texas: (1) the analysis of products and effluents to determine process variability over a finite time period; (2) the analysis of volatile organic compounds from heavily loaded sample matrices; (3) the analysis of phenolics from heavily loaded phenolic streams; and (4) the analysis of sulfides, cyanide, thiosulfates, and thiocyanates from heavily loaded aqueous streams.

PROBLEM AREAS

REPRODUCIBILITY IN PROCESS VARIABILITY STUDIES

It is necessary to use a consistent approach in analyzing samples for process variability. For the Ft. Lewis effort, two sets of two samples of a heavy distillate stream were obtained over a 3-day period. This procedure provided a set of 12 discrete samples for studying process variability with built-in controls for sampling and analytical variability.

The heavy distillate stream is a very complex sample matrix containing several hundred discrete components which range in concentration from the parts-per-billion level to several percent of the overall mixture. Analytical options included: (1) several types of fractionation procedures such as the Level 1 Assessment protocol, which yields seven discrete fractions from a silicic acid column; (2) fractionation by Florisil chromatography followed by chemical and further column separation to achieve separations by class for sulfur, nitrogen, and polycyclic aromatic hydrocarbons; and (3) analysis of the gross mixture for major constituents. To eliminate variations which would be introduced by fractionation and concentration procedures, it was decided to analyze the gross mixtures and to use the data obtained to define process, sampling, and analytical variability.

With mixtures of this complexity, capillary gas chromatography provides the most effective separation. It can be coupled with mass spectrometry to obtain as much qualitative information as possible about the major constituents of the mixture. Figure 1 shows the region of the total ion reconstructed chromatogram of a typical sample from the heavy distillate series. The broad peaks are not a function of poor chromatography but, rather, indicate co-eluting components. Several of the major components are identified.

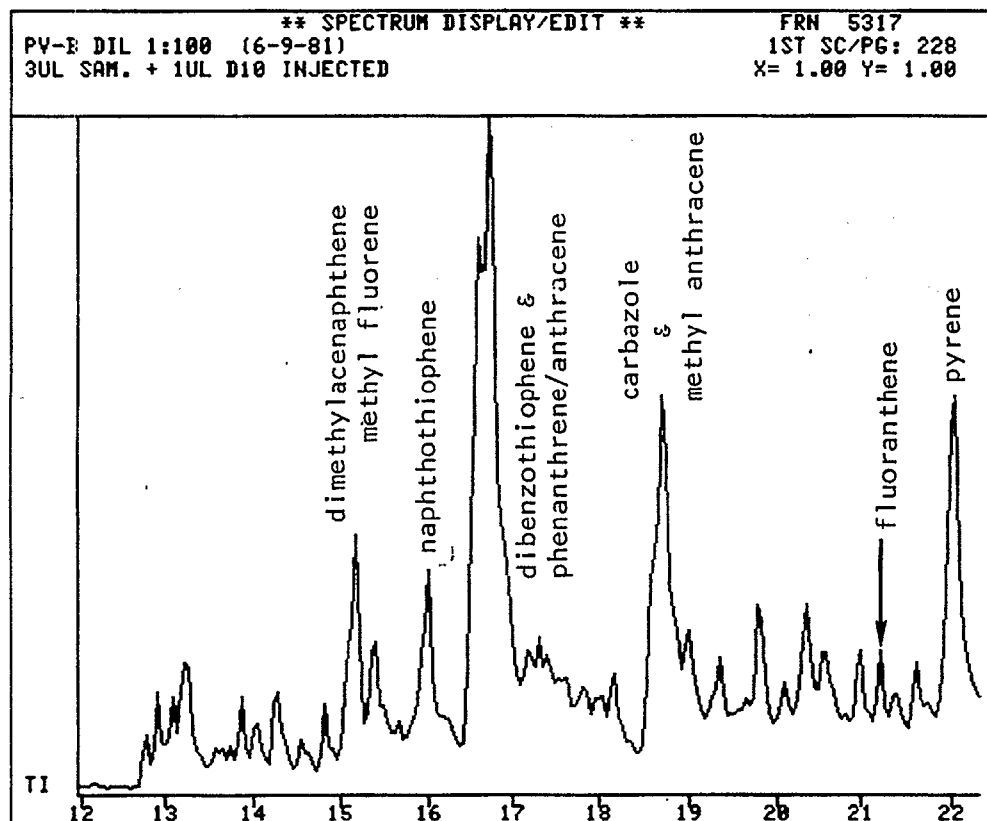


Figure 1. Total Ion Reconstructed Chromatogram of Heavy Distillate Sample from SRC Pilot Plant.

Figure 2 shows four separate total ion chromatograms of different samples taken the same day. The run-to-run reproducibility of these samples is good and can be used effectively for comparison purposes.

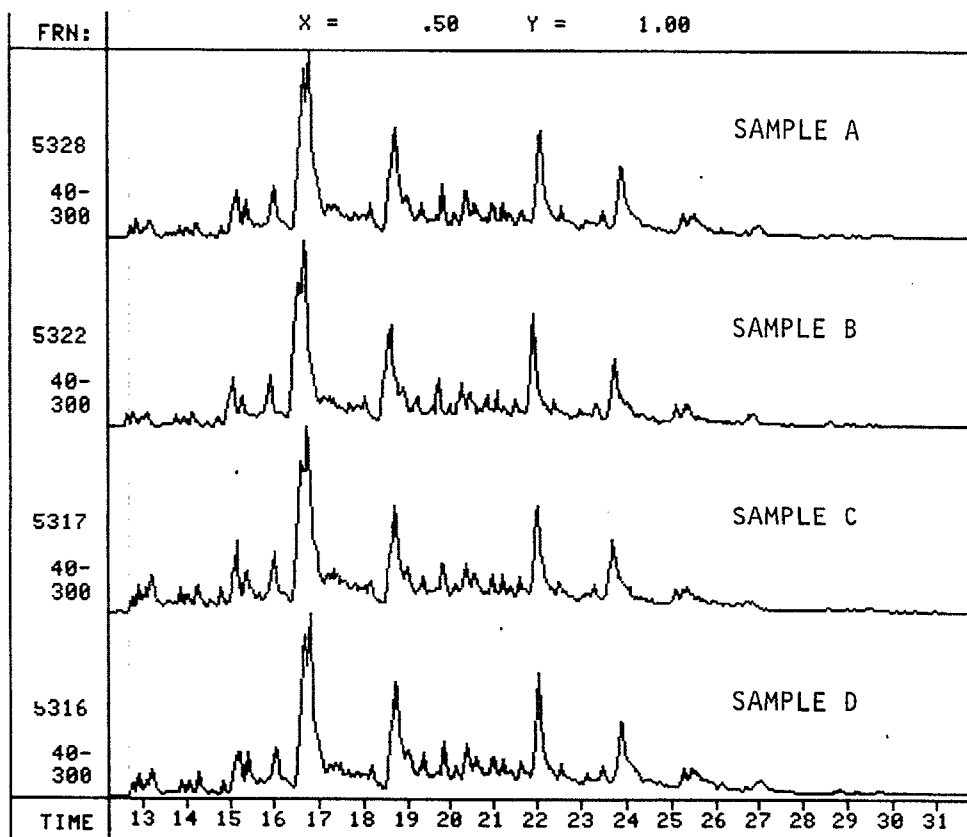


Figure 2. Comparison of Process Variability Samples.

Figure 3 shows selected extracted ion chromatographic profiles (EICP) of the molecular ions for naphthothiophene, dibenzothiophene, phenanthrene/anthracene, and a series of C_2 biphenyls and acenaphthenes. Using EICP, it is possible to discriminate between species in the mixture, while components co-elute when gas chromatography is used alone.

Figure 4 shows another example of co-eluting species. In this case, pyrene and fluoranthene co-elute with substituted phenanthrene/anthracene components and with a series of C_2 carbazoles.

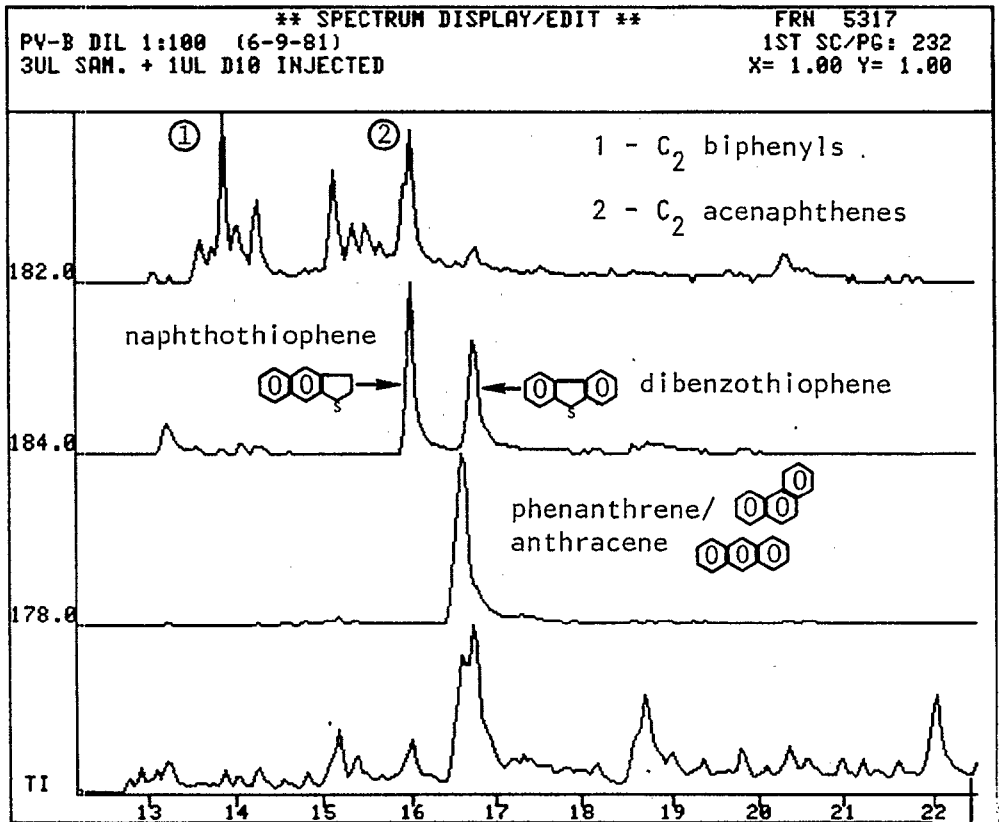


Figure 3. Co-eluting Components in the Anthracene Region

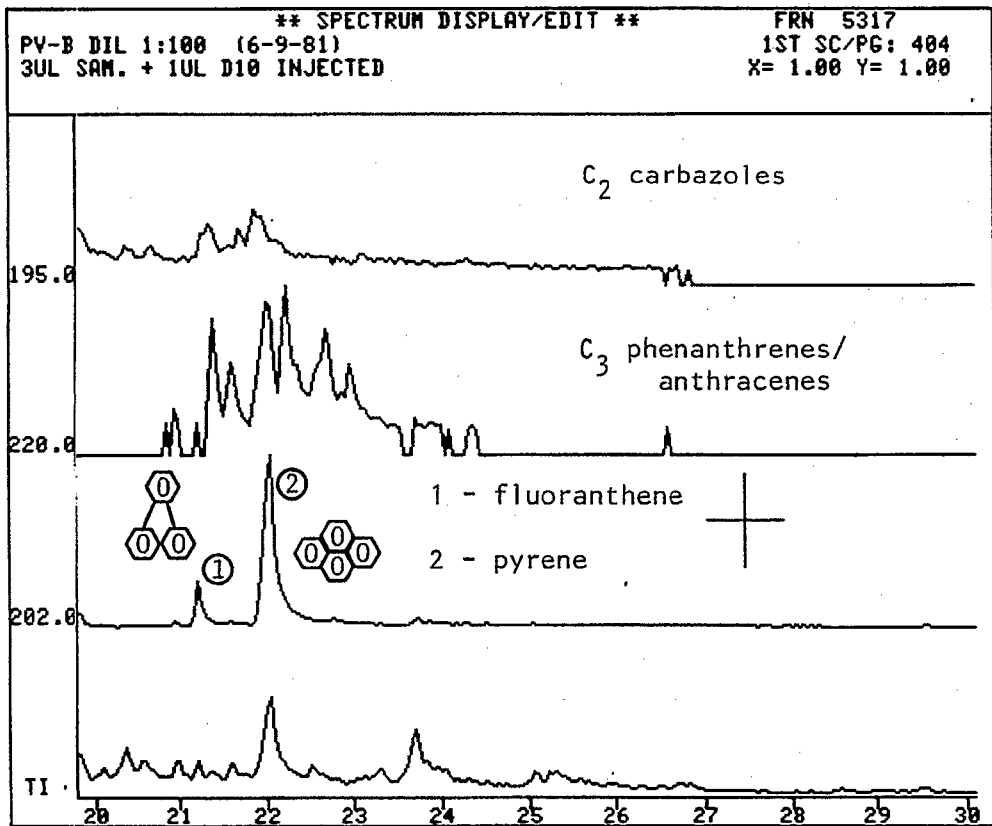


Figure 4. Co-eluting components in the Pyrene Region

By using the extracted ion chromatographic profiles for various components and analyzing the mass spectral data, it is possible to identify a discrete set of components. The areas of peaks from the extracted ion chromatographic profiles can be integrated to generate quantitative data for specific components from the gross sample. The most commonly used calculation involves obtaining integrated peak areas from known amounts of standards and comparing these peak areas to those of a known amount of internal standard. From these data, it is possible to calculate the relative response factor (RRF). The amount of specific components present can then be determined using the equation below. For gas chromatography, this is an excellent and extremely reproducible calculation because the detectors used are very stable.

$$\text{RRF} = \frac{\text{amount } d_{10} \times \text{Area Std.}}{\text{amount Std.} \times \text{Area } d_{10}}$$

$$\text{Amount X} = \frac{\text{Area}_x}{\text{Area } d_{10}} \times 1/\text{RRF} \times D$$

Where: Std. = compound of interest

d_{10} = internal standard

D = dilution factor

When used in conjunction with mass spectrometry, this method yields acceptable reproducibility. However, mass spectrometry is not as stable a detector and is more sensitive to changes in relative concentration of the sample and internal standard than is gas chromatography. This means that RRF values must be calculated very frequently if acceptable quantitative data are to be obtained for repetitive studies. Additionally, the overall sensitivity of the mass spectrometer can change dramatically over short periods of time.

The RRF method was used to generate the data in Table 1. Several representative concentrations for components found in the heavy distillates are shown. Samples A and B are one pair and C and D are a second pair. A and B were taken at the same time, C and D at different times, during the same day. These data were obtained as part of the process variability study and are, we feel, representative in light of the complexity of the sample matrix.

The values in Table 2 were derived by using the data obtained for standards normally used in the generation of RRF values. In this case, these data were used to calculate a working standard curve by least squares linear regression (LSLR) analysis. A second set of least squares linear regression lines were calculated using the data but normalizing the output of the internal standard to a set figure and adjusting the areas of the known compounds to reflect this normalization. All of the lines used had a correlation coefficient exceeding the 95 percent confidence level.

Generally, the values obtained for analytical pairs are in better agreement for the normalized lines.

TABLE 1. COMPARISON OF VALUES OBTAINED BY RRF CALCULATION (g/kg)

Component	Sample		Sample	
	A	B	C	D
Fluorene	2.17	2.36	2.12	1.50
Carbazole	5.67	6.67	7.55	5.68
Dibenzothiophene	8.46	8.96	9.20	8.36
Phenanthrene/Anthracene	20.02	22.64	24.46	20.88
Pyrene	11.66	13.68	13.28	11.37
Chrysene	1.09	1.57	1.73	1.21
Benzo(a)Pyrene/Benzo(e)Pyrene [B(a)P + B(e)P]	.83	.56	.85	.38

TABLE 2. COMPARISON OF SAMPLES BY LSLR AND NORMALIZED LSLR

Component	LSLR		LSLR-N		LSLR		LSLR-N	
	A	B	A	B	C	D	C	D
Fluorene	2.04	1.81	1.74	1.89	1.23	1.07	1.44	1.21
Carbazole	4.95	4.68	4.48	5.27	4.47	3.40	5.59	4.48
Dibenzothicphene	7.27	6.20	6.67	7.06	5.42	4.90	7.25	6.59
Phenanthrene/ Anthracene	16.99	15.41	15.27	17.27	13.15	11.96	16.56	15.93
Pyrene	13.27	12.47	14.8	17.39	9.58	8.69	15.08	14.42
Chrysene	1.32	1.49	1.09	1.58	1.28	1.04	1.44	1.22
B(a)P + B(e)P	.40	.49	.31	.47	.52	.34	.48	.34

Table 3 shows a comparison of the three methods used for quantitation of these selected compounds. The values shown represent a percentage variation from an average value for the paired samples. Using the LSLR

data does little to alter the measurements obtained by RRF values when the data are very close, as in the A-B pair for fluorene and in the dibenzothiophene pairs. However, for data which show large variations by the RRF method, such as chrysene and B(a)P+B(e)P, the data obtained by LSLR analysis are more precise. Overall, for the full set of reported components (40-50 in all), the normalized LSLR data sets proved to be more precise. It is our experience that the use of normalized least square linear regression analysis for measurements requiring higher precision than a normal screening technique is a useful alternative to standard procedures. The data from screening analyses could also be calculated by this type of analysis to afford more consistent results.

TABLE 3. COMPARISON OF METHODS BY PERCENTAGE DEVIATION FROM AVERAGE

Component	<u>RRF</u>		<u>LSLR</u>		<u>LSLR-N</u>	
	AB	CD	AB	CD	AB	CD
Fluorene	4.4	17.1	5.6	7.0	3.8	8.3
Carbazole	8.1	14.0	2.9	13.0	8.1	11.0
Dibenzothiophene	2.8	4.7	7.8	5.0	2.9	4.77
Phenanthrene/ Anthracene	6.13	7.9	5.1	4.8	6.1	1.9
Pyrene	8	7.78	3.1	4.9	8.0	2.2
Chrysene	18.0	17.6	5.6	10.0	6.4	10
B(a)P + B(e)P	24	39	11	20	9.5	17

INTERVAL BETWEEN COMPONENTS

	<u>RRF</u>	<u>Absolute Differ- ence</u>	<u>LSLR</u>	<u>Absolute Differ- ence</u>	<u>LSLR-N</u>	<u>Absolute Differ- ence</u>
	Fluorene	1.50 - 2.36	.86	1.07 - 2.04	.97	1.21 - 1.84
Carbazole	5.67 - 7.55	1.88	3.40 - 4.95	1.55	4.48 - 5.77	1.29
Dibenzo- thiophene	8.36 - 9.20	.84	4.90 - 7.27	2.37	6.59 - 7.06	.47
Phenan- threne/ Anthracene	20.02 -24.46	4.44	11.96 - 16.99	5.03	15.93 - 17.44	1.51
Pyrene	11.37 - 13.68	2.31	8.69 - 13.27	4.58	14.42 - 17.39	2.97
Chrysene	1.09 - 1.73	.65	1.04 - 1.49	.45	1.09 - 1.59	.50
B(a)P + B(e)P	.38 - .85	.47	.34 - .56	.22	.34 - .59	.25

PURGE AND TRAP ANALYSIS

The next area of difficulty is the analysis of heavily loaded process streams for volatile organics. The major problem associated with these analyses is the limited capacity of the adsorbing material to efficiently adsorb the levels of organic materials present in process streams. The adsorbent used in our synfuels studies was Tenax resin with a capacity to adsorb 1 to 2 percent of the weight of Tenax in the trap. The actual amount of Tenax is generally 0.5 gm or less. A 0.5 to 1.0 percent loading of this material would be equivalent to 2.5 to 5.0 mg capacity. Above this level, the Tenax will hold more material but with a dramatic decrease in trapping efficiency. Table 4 shows data obtained from a heavily loaded process stream from the EDS plant at Baytown, Texas. Samples B₁ through B₅ are the same sample purged at different levels of concentration. Samples A through F are different samples from the same stream.

TABLE 4. COMPARISON OF PURGE AND TRAP DATA
BY VOLUME PURGED

Sample	Quantity Purged (mls)	Quantity Calculated (mg/l)	Observed Load on Tenax/(mg)	Actual Load on System (mg)
B ₁	0.25	2890	.7	.7
B ₂	1.00	2769	2.7	2.7
B ₃	2.50	1700	4.3	6.8
B ₄	5.00	997	4.5	13.5
B ₅	10.00	460	4.6	27.0
A	1.00	2216	2.6	2.6
B	1.00	2805	2.8	2.8
C	1.00	2642	2.6	2.6
D	5.00	1255	6.3	13.0
E	10.00	523	5.2	26.0
F	10.00	508	5.1	26.0

As indicated in Table 4, adsorbing capacity has no real effect for the samples run at 1.0 mls. Above this level, however, the data are affected significantly. It is important to note that although the overall content of purgeable materials is extremely high, they are not readily amenable to analysis by direct aqueous injection because it does not effectively separate the many components present in these samples. Using the mass spectrometer as a detector and injecting even several μ l of

aqueous material, there are nanogram to low microgram quantities of many of the components. At levels of 2 g/l, only 2 µg/µl are being analyzed by direct injection.

Table 5 shows the results obtained from a naphtha sample, which should contain a very high percentage of purgeable materials. This sample was run as a pure organic. The samples were diluted 1:10 with methanol. Two samples were run with 100 µl of the diluate injected into 10 mls of H₂O. Two samples were run with 50 µl of the diluate injected into a 10 ml aqueous matrix.

TABLE 5. COMPARISON OF PURGE VALUES FOR NAPHTHA

Sample	Analyzed (µl)	Observed Concentration (g/l)	Observed Loading Tenax (mg/l)	Actual Tenax Loading (mg/l)
Naphtha A	10	382	3.82	5.80
Naphtha B	10	297	2.97	5.80
Naphtha C	5	584	2.92	2.92
Naphtha D	5	576	2.86	2.86

The values at a 50 µl injection of a 1:10 aliquot are reasonable for this type of stream, indicating that approximately 75 percent of the components in the naphtha streams are purgeables.

We recommend that internal process streams be run at a volume of 0.25 to 0.50 mls of sample for purge and trap analysis except for those streams for which there is little or no chance that purgeables are present. A second run can then be made after calculation of an effective column loading. Also, we recommend that volatile organic materials such as naphtha streams be run at levels of no more than 50 µl of a 1:10 diluate. Analytical parameters for streams with higher boiling ranges are based on volatile content. Industry literature can be used as a guide for estimating the quantity of samples to be analyzed. For process streams, we have found that sour water streams, including streams from pumps and drums as well as gas scrubber streams, should be regarded as heavily loaded process streams for volatiles.

PHENOLICS ANALYSIS

A third problem in analyzing process streams is the high level of phenolic materials present in sour water streams. Toxic gases evolve during acidification of the aqueous samples. Also, the high levels of phenolics present in extracts tend to create problems when concentrated for analysis by gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS). Very low recovery rates (20 to 40 percent) have been reported in many cases. The more volatile phenols are extremely difficult to recover quantitatively. For this reason, EPA has recommended a colorimetric method for the analysis

of steam-distillable phenol. The method is fairly specific for phenol; however, there are other major phenolics present in these streams, such as cresols, xylenols, and trimethyl phenols. These phenols are not quantitatively measured by this method. We have analyzed these streams by direct aqueous injections as well as colorimetric and GC/MS methods.

Table 6 shows a comparison of the results obtained by GC/MS, colorimetric, and direct injection analysis for two streams heavily loaded with phenolics. In both cases, the GC/MS gave lower values than the direct injection GC method. For heavily loaded samples, the GC method has the advantage of allowing analysis in situ with a minimum of sample handling. The only possible alteration to this method would be to carefully acidify to a slightly acid pH (6.0 to 7.0) any samples which were very basic to assure that all phenolics have been analyzed.

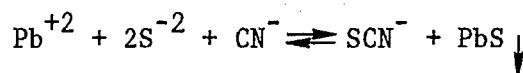
TABLE 6. PHENOLICS BY COMPARATIVE METHODS
(mg/l)

Sample	METHOD		
	GC/MS	Colorimetric	Direct Injection
Sample A	3500	2439	11321
Sample B	4300	16024	13131

The method we used was a slight modification of Standard Method 510E from the 14th edition of Standard Methods for the Examination of Water and Wastewater.¹

ANALYSIS OF THIOCYANATES AND RELATED SPECIES

There are also problems associated with the analysis of sulfide, cyanide, thiosulfate, and thiocyanate (SCN^-) in streams which have a high sulfide and/or H_2S content. These samples were analyzed in accordance with procedures defined in Manual of Methods: Preservation and Analysis of Coal Gasification Wastewater.² We found that the precipitation of sulfide from heavily loaded streams as lead sulfide is not easily accomplished and that the precipitations required several days. In addition, cyanide can easily be lost by occlusion during the precipitation. The amounts of lead sulfide were so great that this occlusion is a very real problem. Dilution of the original samples prior to precipitation was not possible because the lower limits of the required analytical range preclude dilution. Another consideration in the cyanide analysis is the equilibrium shown below. It appears that the equilibrium is being forced to the left as sulfur is removed from the system.



Theoretically, the analysis of SCN^- should then become an important parameter. However, the same problems occur in this analysis since sulfides must initially be precipitated and there are high levels of lead sulfide present. To date, no adequate solution to this problem has been developed.

CONCLUSION

There is currently no all-encompassing methodology available for the analysis of process, product, and wastewater streams from synthetic fuels plants. Each type of stream and each individual process stream must be handled under conditions which will optimize the value and validity of the data obtained. In our current studies, we are attempting to modify existing procedures, as appropriate, to provide the most effective analytical approach. In particular, we are correlating GC/MS and GC data by utilizing the qualitative data obtained from GC/MS as a guide, then using capillary gas chromatographic data as the eventual quantitative tool. By incorporating the specificity of the mass spectral data, we are better able to quantitate unresolved gas chromatographic peaks. We are also assessing alternatives to existing methodologies of volatile organics analysis to obtain a more consistent approach to the problem of heavily loaded process streams. Finally, we are attempting to modify the precipitation procedures for sulfides to adapt a method which is viable for heavily loaded process streams.

REFERENCES

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2. Luthy, R.G. Manual of Methods: Preservation and Analysis for Coal Gasification Wastewaters. Prepared for the U.S. Department of Energy, by the Environmental Studies Institute, Carnegie, Mellon University, Pittsburgh, PA, July 1978.

SOLVENT EXTRACTION PROCESSING
FOR COAL CONVERSION WASTEWATERS

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ABSTRACT

This paper outlines experimental and modeling techniques that are being used to evaluate solvent extraction processing of coal conversion wastewaters. The project includes characterization of organic contaminants in slagging fixed-bed gasification process wastewater, as well as screening studies to evaluate removal efficiencies for these contaminants. Experiments are also in progress to measure distribution coefficients for several solvent types with phenol and representative base- and neutral-fraction aromatic solutes. These experiments are being performed with both clean water and wastewater systems. Results from these experiments are being evaluated in light of three techniques for estimating distribution coefficients: Modified regular solution theory as used in chemical engineering processing, expanded solubility parameter approach as used in liquid-liquid chromatography, and estimation of octanol-water partition coefficient as employed in environmental science. This paper reviews results obtained to date and explains direction for work during the coming year.

INTRODUCTION

Solvent extraction is a candidate treatment process for reducing organic contaminants from coal conversion wastewater. Solvent extraction is especially attractive for treatment of highly contaminated streams where the cost of treatment may be compensated, in whole or in part, by the value of recovered material and by reduction of loadings on downstream wastewater processing units. Solvent extraction may also eliminate the need for additional physicochemical wastewater treatment steps.

Experimental work now in progress is aimed at defining solvent extraction

processing characteristics of a slagging fixed-bed coal gasification wastewater. This work includes tests with both wastewater and clean water systems as well as theoretical considerations aimed at development of a model to aid prediction of the fate of organic contaminants during solvent extraction treatment.

SOLVENT EXTRACTION PROCESSING

A typical solvent extraction system is illustrated in Figure 1 (Earhart, et al., 1977), where it is shown that the process is comprised of three basic unit operations: (1) an extractor where wastewater and solvent are mixed and separated, (2) a solvent regenerator where solvent is separated from extracted solutes for reuse, and (3) a solvent recovery step where residual solvent is removed from the treated wastewater.

Solvent extractors may be classified as either stagewise contactors, such as mixer-settlers, or as differential contact extractors, such as a packed column or rotary disk contactor. Solvent regeneration is usually accomplished by distillation, and solvent recovery may be achieved by either stripping or secondary solvent extraction.

Numerous solvents are available for use in solvent extraction systems. Solvents which have been employed for processing phenolic streams include: light aromatic oil mixtures, tricresyl phosphate, n-butyl acetate (NBA), diisopropylether (DIPE), and methylisobutyl ketone (MIBK). When choosing an extraction solvent, two of the most important considerations are high solute distribution coefficient and low aqueous solubility. While aqueous solubility data are available for most solvents, solvent-solute distribution coefficient data are available for relatively few compounds, notably phenol and its derivatives. It is especially noteworthy that there is essentially no solute distribution coefficient data for the variety of base- and neutral-fraction solutes which may exist in a coal conversion wastewater.

The solute distribution coefficient may be defined as either the equilibrium ratio of solute mass concentration (C , mg/l) in solvent and water phases (K_D'), or as the ratio of solute mole fraction activity coefficients (γ) in each phase (K_D);

$$K_D' = C_s/C_w$$

$$K_D = \gamma_w/\gamma_s$$

where the subscript s refers to the solvent phase and w to the water phase. Mass concentration and mole fraction activity distribution coefficients are related by the ratio of water and solvent molar volumes;

$$K_D' = \frac{V_w}{V_s} K_D$$

Usually solute distribution coefficient data must be determined from laboratory testing, and this may be a costly and time consuming task. However,

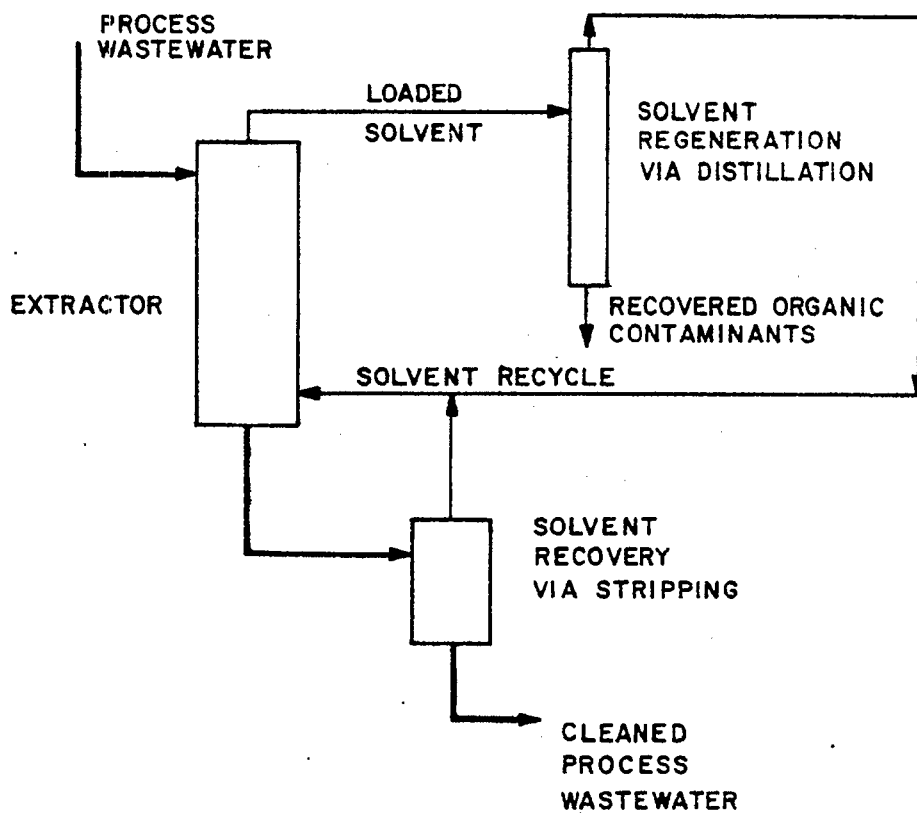


FIG.1 TYPICAL SOLVENT EXTRACTION PROCESS WITH A VOLATILE SOLVENT

time and expense can be saved by using thermodynamic models to estimate solute mole fraction activity distribution coefficients (K_D). Evaluation of the accuracy and applicability of thermodynamic models is included in the theoretical aspects of this project.

PROJECT OBJECTIVES

Work now in progress entails both experimental and theoretical aspects of modeling the fate of organic compounds during wastewater treatment, with emphasis on solvent extraction. Specific objectives of the project are outlined below:

- 1) Characterize organic contaminants in slagging fixed-bed gasification process wastewater before and after several steps of bench-scale treatment. This work will include screening studies with several solvents to evaluate the efficiency of solvent extraction for removal of phenolics, as well as for removal of base and neutral fraction aromatics. In addition, data from these tests will be used to observe organic specie removal during ammonia stripping and biological oxidation.
- 2) Review the literature for assessment of models to predict distribution coefficients and compile a listing of the available experimental data on distribution coefficients for wastewater contaminants.
- 3) Perform experiments to measure distribution coefficients for phenol and representative base and neutral fraction aromatic compounds in both clean water and wastewater systems. The effect of solute concentration on the value of the distribution coefficient will also be evaluated.
- 4) Assess the applicability and accuracy of distribution coefficient models by comparing model results with experimental data found in this study and experimental data reported in the literature.

RESULTS TO DATE

Several study objectives have been completed or are in the process of being completed. Screening studies have been performed with several solvents to assess their suitability for extraction of phenolic solutes from raw slagging fixed-bed coal gasification wastewater. This wastewater was generated from gasification of a lignite coal.

As a result of these screening studies, MIBK was selected for execution of bench-scale treatment tests incorporating solvent extraction, ammonia stripping and biological oxidation. As a part of this study, organic contaminants were characterized in collaboration with Argonne National Laboratory to assess removal of acid, base and neutral fraction solutes following solvent extraction-

ammonia stripping and biological oxidation. Results of this work were presented at the 54th Annual Conference of the Water Pollution Control Federation (Luthy, et al., 1981). A summary of this work is provided in Tables 1 and 2.

Table I shows average solvent extraction treatment characteristics for MIBK extracted wastewater. MIBK is particularly effective for removal of phenolics ($K_D \sim 100$), and the data show that in the process of reducing phenolics to about 5 mg/l there is also substantial reduction of TOC, COD and BOD. Biological oxidation was evaluated by both activated sludge (AS) and powdered activated carbon-activated sludge (PAC-AS) treatment with 5000 mg/l PAC. Both AS and PAC-AS showed good removal of the pollutants shown in Table 1. The biological oxidation studies showed that solvent extracted wastewater was easier to treat via AS in comparison with wastewater not pretreated for reduction of phenolics. Solvent extraction eliminated the requirement for dilution prior to AS and also reduced wastewater foaming during biological treatment. Solvent extraction also resulted in lower mass loading of residual organic material (eg. color, TOC, and COD) in the biological reactor effluent.

GC/MS analysis of acid, base, and neutral fraction organics were performed on raw condensate, solvent extracted-ammonia stripped wastewater, and AS and PAC-AS effluents. HPLC analysis were performed after each treatment step for detection of eleven polycyclic aromatic hydrocarbons. Table 2, which provides a summary of the analytical results, shows that no organic contaminants could be detected in the acid, base, and neutral fraction suspended phase samples of MIBK extracted wastewater. Analysis of solvent extracted-ammonia stripped aqueous phase acid fraction samples showed the presence of residual phenol, cresols, and other acid fraction compounds, while base and neutral fraction species showed mainly low levels of relatively few compounds. Analysis of AS and PAC-AS treated water showed excellent reduction of those few organic compounds which remained after extraction and stripping. These results showed that solvent extraction for reduction of phenolics offers several wastewater processing advantages for treatment of coal conversion condensates.

This work was followed by a preliminary investigation of thermodynamic models for the prediction of solute distribution coefficients between water and an organic solvent for phenol and other aromatic solutes. The results of this work were presented at the Symposium on Water Management and Pollution Control for Coal Gasification and Liquefaction, sponsored by the Division of Environmental Chemistry at the 182nd ACS National Meeting in August (Campbell and Luthy, 1981). This work showed that most of the experimental solvent extraction studies reported in the literature have focused on phenolic compounds. No distribution coefficient data were found for base and neutral fraction solutes with solvents normally used for phenol recovery. Furthermore, no distribution coefficient data was found for tests using actual coal refinery wastewaters.

A review of the chemical engineering and liquid-liquid chromatography literature revealed that solvent extraction models which are used in these disciplines are based on developments evolving from regular solution theory. These concepts have been applied to several solute-solvent systems, and it was found that some empiricism is necessary for estimation of certain thermodynamic

TABLE 1. SLAGGING FIXED-BED WASTEWATER TREATMENT CHARACTERISTICS¹

Parameter mg/l unless noted	Raw Wastewater RA-52	Solvent Extracted MIBK	Ammonia Stripped	Activated Sludge	PAC/AS
TOC	11.100	1.950	1.380	580	385
COD	32.000	3.900	2.980	1,340	640
BOD	26.000	2.900	1.820	32	30
Phenolics	5.500	5	3	0.1	<0.02
Org-N	115	51	33	10	4
NH ₃ -N	6.300	4.400	30	84	20
NO ₃ ⁻ -N	<5	<5	<5	40	100
SCN ₂ ⁻	120	110	105	4	<0.5
CN ⁻	1.8	1.5	1.5	1.4	1.3
CN ^{tot}	0.1	--	0.1	0.1	<0.1
Freon Ext	410	--	10	<5	<5
Alk (as CaCO ₃)	20,700	16,300	850	175	72
Cond (µmho/cm)	20,000	18,600	1,490	2,230	2,200
Color (Pt-Co units)	500	500	700	500	<20

¹Reference: Luthy, Stamoudis, Campbell, and Harrison, 1981. Analyses of solvent extracted samples for TOC, COD and BOD were performed after gentle heating to expel residual MIBK.

Table 2

Concentration (µg/l) of Representative Organic Compounds Identified in Slagging Fixed Bed Quench Water at Various Stages of Treatment

Peak Number	Compound Name	Raw Water		MIBK-Stripped		MIBK-AS		MIBK-AS/PAC	
		SS	FW	SS	FW	FW	FW	FW	FW
<u>Acid Fractions</u>									
20	Phenol	15,100	3,080,000	NT	500	10	3		
50	Methylphenol	6,300	427,000	NT	100	1	1		
80	Methylphenol	16,400	494,000	NT	80	1	1		
140	C ₂ -Phenol	8,890	155,000	NT	50	NT	NT		
260	Hydroxindan	340	3,820	NT	40	NT	NT		
370	1-Naphthol	250	1,150	NT	40	NT	NT		
<u>Base Fraction</u>									
10	Pyridine	3.9	14,530	NT	NT	NT	0.1		
15	Toluene	NT	77	NT	NT	NT	NT		
20	Methylpyridine	12	7,120	NT	1.0	NT	NT		
140	Aniline	27	6,500	NT	NT	NT	NT		
390	Azaphthalene	450	500	NT	NT	NT	NT		
730	Azafluorene	20	15	NT	NT	NT	NT		
<u>Neutral Fraction</u>									
32	Methylcyclohexane, or C ₂ -cyclopentene	ND	22,750	NT	4230	NT	NT		
35	Denzonitrile	ND	2,900	NT	NT	NT	NT		
70	Indene	2300	910	NT	NT	NT	NT		
125	C ₃ -Thiazole	ND	ND	NT	31	1.8	NT		
180	Naphthalene	16,300	26,600	NT	NT	NT	NT		
330	Indole	150	5,000	NT	NT	NT	NT		
380	Biphenyl	1100	ND	NT	NT	NT	NT		
460	Acenaphthene	2480	ND	NT	NT	NT	NT		
710	Phenanthrene	4680	ND	NT	NT	NT	NT		
790	Fluoranthene	1510	ND	NT	NT	NT	NT		
810	Pyrene	830	ND	NT	NT	NT	NT		

SS, Suspended Solids; FW, Filtered Water; ND, Not Determinable; NT, Not Detected.

parameters. Despite this limitation, our analysis has shown that for MIBK and water, neutral fraction aromatic solutes are predicted to have distribution coefficients substantially greater than that for phenol. Thus, in the process of reducing phenolics from relatively high concentrations to comparatively low concentrations, it is expected that neutral fraction solutes would be reduced by even greater proportions.

CURRENT AND FUTURE WORK

Recent experimental work has been directed towards measuring distribution coefficients in both clean water and wastewater systems. This work has examined three solutes: phenol as a representative acid fraction solute, and aniline and pyridine as representative base fraction solutes. These compounds comprise the predominate parent chemical species for compounds previously identified in each of these fractions. It is planned to measure distribution coefficients for benzene with several solvents, as benzene is the parent specie for neutral fraction solutes.

Five solvents have been incorporated in these tests, methylisobutyl ketone (MIBK), di-isopropylether (DIPE), n-butyl acetate (NBA), toluene, and tetradecane. These compounds are representative of major classes of organic solvents. MIBK is reportedly used in an extraction process licensed by the Chem-Pro Equipment Corp. (Greminger, et al., 1980), while DIPE is employed in the Lurgi Phenol-solvan process. NBA shows a relatively high distribution coefficient for phenol, and it has been proposed for use in dual-solvent extraction systems (Earheart, et al., 1977). Toluene is a component of coal-derived light oil, which was widely used at one time for extraction of phenolics from coke plant ammonia liquor. Tetradecane was included in this study for comparison purposes because it is an alkane, and because crude oil or related compounds are sometimes involved in petroleum refining operations for extraction of phenol from water or for washing phenol from refinery products.

Solvent extraction tests were performed with these solvents and solutes, in single and multiple solute clean water systems as well as in actual wastewater, to investigate potential synergistic/antagonistic effects. The effect of solute concentration was also investigated. The results of this work are being summarized in the form of a technical paper.

It is planned to execute another treatment study using slagging fixed-bed wastewater generated from gasification of another type of coal. Results obtained from this work would be used to verify the previous results, as well as to assess processing differences for a different water-solvent system. The tests would also include detailed wastewater characterization at different levels of phenol removal. These analyses would provide information on whether various contaminants are removed concomitantly in proportion to their respective distribution coefficients.

The latter tests are important from an economic point of view. Goldstein (1981) notes that single stage extraction is less costly than multiple-stage extraction, and that partial phenol recovery may be economically attractive. An 80 percent recovery of 5,500 mg/l phenolics is reported as supplying enough energy in the recovered material to run the extraction process. Goldstein also recommends solvent extraction if phenolic levels are high and if BOD concentrations are greater than 4,000 to 6,000 mg/l.

ADVANTAGES OF SOLVENT EXTRACTION

The economic issues regarding cost of solvent extraction versus reduced cost of additional wastewater treatment are not easily evaluated. However, it is clear that solvent extraction of phenolic condensates is advantageous for numerous reasons. Some of these reasons are outlined below.

- (a) Solvent extraction removes most base and neutral fraction solutes. This is significant because many of the toxic or hazardous organic contaminants in coal conversion wastewaters are found in these fractions.
- (b) Recovered material may be combusted for heat value, and this heat may be used to drive the extraction process. Properly designed combusters would destroy hazardous organic compounds.
- (c) Solvent extraction would remove hazardous organics, and this would reduce or eliminate problems with disposal of hazardous organic sludges formed as a result of wastewater treatment and reuse.
- (d) Solvent extraction would reduce or eliminate problems with carry over of volatile aromatic hydrocarbons during sour water treatment.
- (e) Extraction removes creosotes, and thus it is likely that most "tar acids" would be removed. This may be particularly important in treatment of coal liquefaction wastewater, where it is believed that acid treatment is required prior to biological oxidation to precipitate tar acids (Drummond, et al., 1981). Solvent extraction may eliminate the need for this step.
- (f) Solvent extraction can eliminate the need for dilution prior to biological oxidation. It has been found in various studies that dilution is required when biological oxidation is employed for treatment of heavily contaminated gasification or liquefaction process condensates (Luthy, 1981; Drummond et al., 1981). Our recent work (Luthy, et al., 1981) has shown that dilution was not necessary when treating solvent extracted coal gasification condensate.
- (g) Pretreatment by solvent extraction results in lower mass loading of residual organic material (i.e. TOC, COD, and color) in biological reactor effluent. Also, foaming was not a problem when solvent extracted wastewater was subjected to activated sludge treatment.
- (h) Since solvent extraction pretreatment can eliminate the need for dilution water as well as result in lower loadings of residual organics, it should benefit any additional treatment required prior to wastewater reuse.

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APPENDIX

Attendees

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Thomson	Sidney	3333 Michelson Drive	Irvine	CA	92730	Fluor Engineers & Constructors, Inc.
Thornton	Richard	1660 S. Albion	Denver	CO	80222	Camp Dresser & McKee, Inc.
Trask	Jeff	576 Standard Avenue	Richmond	CA	94572	Chevron Research Company
Troscinski	Ed	2901 Butterfield Road	Oak Brook	IL	60521	Malco Chemical Co.
Tucker	J. D.	5790 Old Hillington Road	Memphis	TN	38127	W. R. Grace & Co.
Tucker	W. Gene	IERL, MD-63	Research Triangle Park	NC	27711	U.S. EPA
Vans	Elizabeth	401 M Street, S. W. (PH-208)	Washington	DC	20460	U.S. EPA
Voerman	Garrett	1860 Lincoln Street	Denver	CO	80295	U.S. EPA, Region VIII
Wallace	Anna W.	P. O. Box 12194	Research Triangle Park	NC	27709	Research Triangle Institute
Walline	Rob	1860 Lincoln Street	Denver	CO	80295	U.S. EPA, Region VIII
Weiner	Larry	9725 E. Hampden	Denver	CO	80231	U.S. EPA, Region VIII
Weinreich	Gary	One Woodward Avenue	Detroit	MI	48226	ANG Coal Gasification Co.
Wetherold	Robert G.	8501 Mo-Pac Blvd., P. O. Box 9948	Austin	TX	78766	Radian Corporation
Wetzel	Rolf	Molkestrasse 29	D-4300 Essen 1	W GERMANY		Gesellschaft fur Kohle-Technologie mbH
Whipple	Gary	P. O. Box 8361	South Charleston	WV	25303	Union Carbide Corp.
White	David	11455 West 48th Avenue	Wheatridge	CO	80033	Camp Dresser & McKee, Inc.
Wilder	Russell	300 Bear Hill Road	Waltham	MA	02154	EG&G Environmental Consultants
Wilks	Walter H.	1500 Market Street	Philadelphia	PA	19102	Catalytic, Inc.
Wilson	Weslon	1860 Lincoln Street	Denver	CO	80295	U.S. EPA, Region VIII
Witmer	Fred E.	EP-33, Office of Env. Programs	Washington	DC	20545	U.S. Department of Energy
Yee	William C.	913 Hamilton Bank Building	Knoxville	TN	37902	Tennessee Valley Authority
Yeh	Rick F.	P. O. Box 27125, 8303 S. W. Freeway	Houston	TX	77027	Black, Sivalis & Bryson, Inc.
Yen	Alan F.	P. O. Box 2752	Allentown	PA	18001	International Coal Refining Co.
Yu	Wen C.	7929 Westpark Drive	McLean	VA	22102	UOP/SDC

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