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DOE/OR/03054-21
(DE84009919)

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# INTERNAL R&D TASK SUMMARY REPORT: ANALYTICAL METHODS DEVELOPMENT

By
F. K. Schweighardt

Work Performed Under Contract No. AC05-78OR03054

International Coal Refining Company
Allentown, Pennsylvania

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United States Department of Energy

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Distribution Category UC-89

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# ANALYTICAL METHODS DEVELOPMENT

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Prepared for the

UNITED STATES DEPARTMENT OF ENERGY
Office of Solvent-Refined Coal Products
under Contract DE-AC05-78-ORO-3054

Printed in the United States of America

Available from

National Technical Information Service

5285 Port Royal Road

Springfield, VA 22161

NTIS price codes

Printed copy: A04

Microfiche copy: A01

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## ABSTRACT

International Coal Refining Company (ICRC) conducted two research programs to develop analytical procedures for characterizing the feed, intermediates, and products of the proposed SRC-I Demonstration Plant. The major conclusion is that standard analytical methods must be defined and assigned statistical error limits of precision and reproducibility early in development. Comparing all SRC-I data or data from different processes is complex and expensive if common data correlation procedures are not followed. ICRC recommends that processes be audited analytically and statistical analyses generated as quickly as possible, in order to quantify process-dependent and -independent variables.

# EXECUTIVE SUMMARY

In developing the SRC-I process, ICRC realized the necessity for sound analytical characterization of the feed, intermediates, and products of the proposed demonstration plant in Newman, Kentucky. Two programs addressed this objective: Program 26.1--SRC-I Demonstration Plant Analytical Laboratory and Program 12.1.10--Development of SRC-I Product Analysis. This report summarizes goals and achievements of these two programs.

Under Program 26.1, expertise in analytical chemistry was developed by organizing historical knowledge and assimilating new knowledge as it became available from ICRC's contractors and the chemical literature. The data were then used to define samples, analytical methods, instrumentation, space, and staff needed to create a functional analytical laboratory.

Specific major accomplishments were: (1) definition of the performing areas, instruments, waste-solvent management, and staff qualifications for >4,300 $ft^2$ of laboratory space; (2) documentation of a sophisticated capillary gas chromatography/mass spectrometry method for identifying and quantifying coal-derived saturates, aromatics, phenols, and nitrogen bases; (3) development of a comprehensive X-ray method to profile and semiquantitatively measure major, minor, and trace elements in SRC, anode cokes, coal, and KMAC; and, most significantly, (4) a detailed Analytical Methods Manual listing over 70 laboratory procedures that can provide the most precise data base possible with current (1982) technology.

Program 12.1.10 was designed to define, quantify, document, and develop laboratory procedures that would correlate the analytical data bases (historical and future) from the Advanced Coal Liquefaction Facility in Wilsonville, Alabama with those from ICRC's 100-lb/day process development units in Trexlertown, Pennsylvania.

Major accomplishments were: (1) a comprehensive audit of Wilsonville's material-balance and daily sampling sites, along with the analytical procedures used in 1982; (2) a detailed precision/reproducibility study of five key analytical procedures to establish correlations between ICRC's and Wilsonville's data bases; (3) establishment of

interlaboratory round-robin testing that resulted in development of standard analytical methods for coal conversion [cresol solubility (precision, ±1%), solvent fractionation (±5%), vacuum distillation (±3%), simulated distillation by gas chromatography (±4%), and elemental analysis (±2%)]; (4) a comprehensive four-year historical review of the value of the microautoclave solvent-quality test (kinetic) used at Wilsonville and its impact on "next-day" process performance, which showed no direct correlation of the method as a predictive tool; (5) the finding that coal pyrite content (>1.2%) and its distribution had an impact on daily Wilsonville plant performance; (6) the finding that for each change of coal type at Wilsonville, more than 30 days is required in order for gas production and solvent molecular composition to reach a steady state; and (7) development of a solvent quality test that uses the "feed" coal with or without hydrogen as a measure of "next-day" performance.

Major observations deduced from Programs 12.1.10 and 26.1 are that standard analytical methods must be defined, documented, and assigned statistical error limits of precision and reproducibility as early as possible in the development of the coal liquefaction process. The value of each data point may change as design improvements are made, but the analytical procedures used should only change in the degree of precision attainable as time goes on. We have found that it is best to upgrade methods at points in the process development that can "stand alone" for data reduction and trend analysis. The comparison of all data or different data sets (other processes) is far too complex and costly if each group is allowed to follow its own procedures without regard to data correlation.

If a major coal process development program is to be initiated, we recommend that the sample analysis procedure be defined, quality control and quality assurance techniques be established and maintained, and empirical methods constantly refined to allow data-base correlation to process performance. Our reviews showed that far too many analyses were being made on ill-defined process streams, or the data generated did not add to the understanding of the process or characterization of feed, intermediate, or product streams. We suggest that an analytical audit be made of such processes and a statistical sensitivity analysis be generated as early in the program as possible to quantify process-dependent and -independent variables.

The following primary reports were produced from these programs and are published separately:

- Analytical Laboratory Methods Manual (Klusaritz et al., 1983)
- SRC-I Demonstration Plant Analytical Laboratory (Hamilton et al., 1983)
- Development of SRC-I Product Analysis (Schweighardt et al., 1983)

These reports contain further details and discussion of the topics covered in this report.

## INTRODUCTION

One of the objectives of International Coal Refining Company (ICRC) in the engineering and scientific development of clean-coal technology was to provide for sound analytical characterization of the feed for and products from the proposed SRC-I demonstration facility at Newman, Kentucky. To accomplish this objective, two major programs were defined:

1. Program Area 26.1--SRC-I Demonstration Plant Analytical Laboratory
2. Program Area 12.1.10--Development of SRC-I Product Analysis

Overall, Program Area 26.1 was designed to develop analytical expertise, organize the currently available analytical knowledge, and assimilate the knowledge provided by other contractors in order to define the sample sources, analytical methodology, equipment, space, and staff needed to create a functional analytical laboratory. This laboratory is needed to provide a full range of analytical support for the SRC-I plant operation.

Program 12.1.10 had as its objective the development of standard analytical work-up methods for products and intermediates. Standard methods were needed to relate the data base generated at the 6-ton-per-day Advanced Coal Liquefaction Facility in Wilsonville, Alabama to other pilot plant data. Furthermore, the methods could then be applied to the demonstration plant analytical laboratory.

This report summarizes the approach, results, conclusions, and observations gathered during the contract period from January 1981 to September 1983. Included are references to papers published in ICRC's SRC-I Quarterly Technical Reports, which give full details on the experimental work.

## ANALYTICAL NEEDS OF THE DEMONSTRATION PLANT

In order for the demonstration plant to operate cost-effectively and within environmental guidelines, the plant process streams must be monitored and analyzed to provide data for use in process control, material-balance calculations, and product quality control. Support will be required in the area of feedstock, product, and environmental effluent analyses. The following are typical samples from each area:

Feedstocks

Coal
Recycle solvent
Hydrogen
Catalysts
Water

Products

Gases
Liquids
SRC solid
Coke
Ash concentrate
Carbon dioxide
Sulfur

Waste Streams

Ash concentrate
Evaporator solids
Untreated wastewater
Treated waste ash

## PROGRAM AREA 26.1--SRC-I DEMONSTRATION PLANT ANALYTICAL LABORATORY

A major part of ICRC's design for a plant demonstrating the SRC-I technology includes an analytical laboratory to support plant operation, commercial activity, and environmental monitoring. The objectives of program 26.1 were fivefold: (1) define the scope and physical plan of the analytical laboratory; (2) select methods to perform the required analyses; (3) modify existing analytical procedures as required to achieve the optimum accuracy and/or precision for coal-derived samples; (4) develop new methods when existing procedures are inadequate; and (5) prepare a manual defining all analytical procedures to be used in the SRC-I Demonstration Plant laboratory.

### Task 1: Identification of Required Analytical Tests and Review of Current Techniques

Objectives. Specific goals of Task 1 were as follows:

- Review current analytical methods
- Identify the test method or methods for each sample
- Prepare a preliminary list of analytical equipment for the laboratory

- Keep abreast of state-of-the-art coal-product analytical procedures through literature review, visits to coal conversion pilot plants, and conference attendance
- Define individual laboratory modules and their unique physical and spatial needs.

Method. We accomplished these objectives by developing comprehensive lists of analytical requirements defined by the area managers of engineering, manufacturing, wastewater treatment, environmental monitoring, and business/sales. The analyses were then grouped into specific laboratory modules that shared functionally related instrumentation. From personal experience and on-site visits to outside laboratories, we estimated floor space, major instruments, sample handling, organic/inorganic waste production, and storage. Also, a schedule for recruiting, hiring, and training all laboratory personnel was prepared.

Accomplishments. We determined that the analytical laboratory will require 4,320 square feet of space. Table 1 summarizes the distribution of floor space by area. The laboratory staff will comprise 22 technicians, 4 shift leaders, 2 chemists, and 1 quality control manager. Staffing the laboratory in a timely manner is critical to developing reliable on-site analysis. Special attention must be paid to anticipate analytical needs by building in flexibility of space and technical talent. Table 2 summarizes the qualifications of the staff members, and Table 3 outlines a labor timetable. We determined that 30% of the staff should be fully assembled 15 months prior to total plant operation, and 100% staffing is required 4 months before complete operation.

Capital equipment necessary to establish a full working laboratory (not including a computer system) would cost $560,000 in 1981 dollars. Table 4 lists the major equipment items by area need.

A major concern is chemical waste production from the laboratory, consisting primarily of nonhalogenated hydrocarbons and wastewaters containing 1-2% by weight of oils and trace amounts of priority pollutants. Table 5 summarizes the chemical waste anticipated from normal laboratory operation over a 1-week work period.

## Task 2: Methods Development

Objectives. Specific goals were to:

- Develop high-resolution gas chromatographic separations of coal liquids, including functional group subfractions, volatile subdistillates (naphtha), and heavy distillate
- Analyze SRC-I liquid products, using low-resolution mass spectrometry to identify components
- Define gas chromatographic reproducibility (integration area precision and retention times), to aid in routine peak assignments using a flame ionization detector and to allow sample-to-sample comparisons for qualitative differences
- Develop a capsule injection/gas chromatographic (GC) simulated distillation technique for determining the percent and boiling-range distribution of -850°F material in full-range SRC-I-derived samples containing nonboiling residue

Method. Task 2 was successfully conducted by using capillary column gas chromatography with a variety of detectors (flame ionization, nitrogen- and sulfur-selective, and mass spectrometric) to "fingerprint" and characterize a large variety of coal liquids, differing in origin, distillation range, and functionality. Four different commercial gas chromatographs were used to develop our understanding and optimize analysis parameters such as GC column selection, temperature programming rate, and injection techniques. This led to the development of state-of-the-art chromatographic precision and resolution for coal distillates. A selection of five stationary phases was used to optimize polar (hydroxy-) component resolution. In addition, chemical derivatization was used to characterize more than 100 individual components. Relative retention times and retention indices were studied to define the variability in retention times observed, and to minimize experimental problems as they impacted on reproducible peak assignments for all classes of compounds. Nitrogen- and sulfur-selective detectors were found necessary to detect heteroatomic species that are resistant to catalytic hydrotreatment.

A capsule injection/GC-simulated distillation procedure was developed to measure the quantity and boiling-range distribution of -850°F volatiles contained in full-range SRC-I materials. This procedure demonstrated satisfactory precision when performed by a research chemist in a methods development laboratory. However, use of this method as a routine analysis procedure showed a substantially larger standard deviation, which was unacceptable for ICRC's material-balance calculations. Since then, the quality assurance procedure has been significantly upgraded and other modifications have been made to the method to improve precision. Although we believe that the precision of the revised method is satisfactory, the present data base is not adequate to confirm this.

Accomplishments. A typical gas chromatogram of a Wilsonville process solvent is shown in Figure 1. We determined that the precision of measurement of the GC retention time for the commonly found polynuclear aromatic hydrocarbons in coal liquids was ±0.18 min, over the boiling range tetralin to chrysene. Table 6 gives the data base for retention indices determined under four sets of conditions. Table 7 compares the deviation of retention indices for the aromatics in three common solution matrixes. We can conclude that, within the error limits stated, peak assignments can be made by retention indices once the analyst is satisfied that the peak uniquely represents a single component. Precision studies on the same set of aromatics concluded that area measurement can be made to ±1.67% standard deviation over the range of 1-5 total area % per peak. Table 8 summarizes the findings under a series of chromatographic conditions.

In comparing specific analytical GC instrumental parameters between laboratories, we determined that the mean standard deviation varied from 1 to 12%. These findings directly impact the quantitative data comparisons between laboratories if different analytical methods are used. Table 8 summarizes our findings. Method 1 was selected as the most precise procedure because it was nearly independent of operator performance.

It was found that the range in GC response factor for individual compounds varied from 1 to 2.5 relative to naphthalene. We concluded that chromatographic response of the flame ionization detector (FID) to specific compound types must be considered if an absolute quantitative profile is to be constructed of changes occurring during a liquefaction run. Table 9 summarizes the range of response factors found for the major components found in SRC-I distillates.

## Task 3: Modification and Qualification of Methods

Task 3 was divided into three subtasks.

Subtask 1. This part of task 3 was designed to:

- Evaluate the analytical methods available for determining trace amounts of N, S, and Cl in coal liquids
- Compare and contrast American Petroleum Institute (API) hydrometer specific gravity to laboratory-defined specific gravity of narrow boiling-point (50°F) fractions of coal-derived liquids

Methods used in general laboratory practice to analyze N, S, and Cl were identified, and analytical results compared. Laboratory procedures were develeoped to quantify hot filtration sediment, pentane insolubles, filterable insolubles, and adherent insolubles. The final acceptable methods are included in the Methods Manual, which was assembled under Task 4 of Program 26.1. Specific gravities from pycnometer measurements were compared to API hydrometer gravities for distillates taken at 50°F intervals.

We determined that the microcoulometric procedures for Cl and S are directly applicable to coal-derived naphtha liquids. However, nitrogen levels were too low to be adequately detected by microcoulometry. A chemiluminesence method incorporated in the Antek nitrogen analyzer provides the lowest level of nitrogen detection, ppm, at the highest precision, ±5% at 100 ppm.

Analytical methods were developed or modified from American Society for Testing and Materials (ASTM) procedures to quantify hot filtration sediment, pentane insolubles, filterable insolubles, and adherent insolubles. These procedures are also listed in the Analytical Methods Manual of Task 4.

We found that specific gravities calculated from the API tables are within experimental error of the specific gravities determined by pycnometer for 50°F boiling fractions of hydrotreated, coal-derived distillates over the range $C_5$ to 850°F.

Subtask 2--Microanalytical Techniques. This part of the program was intended to:

- Evaluate state-of-the-art instrumentation for precise and accurate determination of C, H, O, N, S, and trace N in SRC-I solids and liquids
- Validate the analytical procedures for ultimate use in the demonstration plant analytical laboratory

Accomplishments of Subtask 2 can be summarized as follows:

- The Perkin-Elmer Model 240[1] elemental analyzer was evaluated for the simultaneous determination of carbon, hydrogen, and nitrogen in SRC-I related products. A modified procedure was developed that achieves an accuracy of ±0.1% for analysis of hydrogen, which is below the manufacturer's specification of ±0.3% absolute.
- Evaluation of the performance of a LECO SC-132 sulfur analyzer showed it is capable of excellent analytical precision and can be applied to analyze SRC-I related products with a standard deviation of less than ±0.04%, in the range 0.2 to 5 wt %.
- We evaluated the accuracy and precision of the Coulometrics oxygen analyzer. Our modified procedure can be used to directly determine organic oxygen in SRC-I materials with an accuracy of ±0.3% absolute.
- We evaluated the Dohrman microcoulometric titration system for trace nitrogen analysis in hydrotreated naphtha and found the results to be unreliable. A chemiluminescent analysis with an Antek instrument was also evaluated and found to provide an acceptable precision, ±0.5% absolute at 0.05 to 2% by weight.

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[1] Manufacturers' names are used as reference only and do not imply an endorsement.

Subtask 3--Development of Metals Analysis. The purpose of subtask 3 was twofold:

- To identify and develop X-ray methods to quantify major, minor, and trace metal concentrations in SRC solids, anode cokes, coal, and Kerr-McGee ash concentrate (KMAC)
- To develop a method for determining trace boron levels in SRC and LC-Finer solids

Two methods were used:

- A 100-mg micropellet energy-dispersive X-ray fluorescence (EDXRF) method was developed and evaluated for analyzing metals in dry ash residues of SRC and two-stage liquefaction (TSL) solids, SRC heavy oils, anode cokes, coal, and KMAC. The nondestructive EDXRF will rapidly and quantitatively measure major elements (Al, Si, K, Ca, Ti, and Fe) and semiquantitatively analyze elements with an atomic number of 11 or above.
- A graphite furnace atomic absorption procedure was developed for analysis of trace boron in SRC solid.

The metals analysis method is documented under Task 4, Analytical Methods Manual. The EDXRF procedure was tested for precision on standards supplied by the National Bureau of Standards (NBS) and on SRC and KMAC materials from the SRC-I Advanced Coal Liquefaction Facility in Alabama. Tables 10 and 11 summarize the analytical precision attainable on standard pellets and 12 standard coal samples, respectively. The results obtained by the EDXRF method were tested in a round-robin study with Alcoa, Galbraith Labs, and the Institute of Mining and Minerals Research of the University of Kentucky. Table 12 compares results of the round-robin analysis of SRC and KMAC samples.

### Task 4: Methods Manual

Objectives of Task 4 were to:

- Prepare a Methods Manual for use in the demonstration plant analytical laboratory.
- Develop a precision and accuracy statement for each method as it relates to SRC-I material.
- Develop a quality assurance and specific safety statements for each method.

The most significant accomplishment of Program 26.1 was compilation of the Analytical Laboratory Methods Manual (Klusaritz et al., 1983), which completely documents over 70 analytical procedures. This manual collates all analytical procedures required to operate an SRC-I plant and is a primary tool for establishment and operation of the laboratory. Its purpose is to provide sufficient information on each analytical technique to direct the acquisition of laboratory hardware and reagents and to facilitate their setup. Because the manual contains detailed information on the performance of each analysis, it can be used for both technician training and reference. In conjunction with proper laboratory supervision, this manual should provide the basis for safe, precise, and efficient performance of all laboratory techniques.

The procedures were either developed under Program Areas 26.1 (Tasks 1-3) and 12.1.10 (Tasks 1 and 2), or are standard Air Products and Chemicals, Inc. procedures, developed exclusively for SRC-I products and materials. The procedures include equipment, reagents, and, where possible, precision statements. Applicable standard ASTM methods are listed. However, for each ASTM method, and for each APCI method, modifications for handling coal-derived products are included, as well as general and specific safety precautions that must be adhered to strictly. Other analyses were obtained with permission from the Wilsonville Advanced Coal Liquefaction Facility, Conoco, Kerr-McGee, and the Bartlesville Energy Technology Center.

## PROGRAM 12.1.10--DEVELOPMENT OF SRC-I PRODUCT ANALYSIS

Two areas of critical need were identified for orderly design, design verification, and operation of the SRC-I Demonstration Plant:

- Development of standard analytical work-up methods for products and intermediates that can be used to relate Wilsonville data to other pilot plant data bases, and can then be applied to the demonstration plant
- Development and correlation of the results of a method for measuring recycle solvent quality for plant operation and control

Both areas related to analytical services--those required to develop a reliable data base, and those that could be correlated to the historical data base generated at the Wilsonville Advanced Coal Liquefaction Facility. This data base was necessary to ensure operational control during plant start-up and operation. The results of this work facilitated selection of the analytical methods for the SRC-I Demonstration Plant laboratory that are documented in the Methods Manual described under Task 4 of Program Area 26.1. Independent topical reports were prepared to address the correlation of ICRC and Wilsonville data bases.

### Task 1: Develop Standard Product Work-Up Procedures

Objective. The objective of Task 1 was to provide common work-up procedures for DOE contractor participants in the ICRC Phase I program. The analytically precise procedures were then used to correlate performance of the ICRC coal process development unit (CPDU) and the Wilsonville coal facility.

Method. The approach taken was to critically review analytical procedures generally used in coal liquefaction to define a complete material-balance data base, in particular those used by the Wilsonville Advanced Coal Liquefaction Facility. Then we selected those procedures that should be documented and reproduced in the ICRC laboratory. Those methods that were found to be least precise, that required extended analytical time, or that could not be correlated to Wilsonville results were investigated in depth. The resultant modified methods were then considered for automation and/or standardization for inclusion in the Methods Manual. To quantify analytical reproducibility under actual field conditions, six different coal liquefaction laboratories conducted a round-robin analysis.

Accomplishments. During FY 1981, a comprehensive audit of the Wilsonville plant sampling sites and material-balance procedures was made for the SRC-I first stage. More than 35 different sample streams were identified, 26 of which were analyzed during material-balance periods and 16 during routine daily operation. Over the past 4 years, 61 analytical procedures have been used at Wilsonville; of these, 27 were still in practice as of January 1983.

During a typical material-balance period, 153 individual analyses are performed, of which only 67 contribute directly to material-balance calculation or daily operation. To generate the data needed for a material balance, 13 unique analytical methods are used. Of these, four key methods were identified: cresol solubility, solvent fractionation, simulated distillation by gas chromatography (SDGC), and vacuum distillation. For daily operation, another procedure should be added--the solvent quality test by microautoclave coal conversion (which is discussed under Task 2).

A comprehensive summary report of all sampling sites and documentation of the analytical procedures were the major end results of this task. By observing the actual methods in practice at Wilsonville, we were able to document the four key material-balance procedures. Each method was then established at ICRC's laboratories in order to quantify analytical precision.

Cresol Solubility. The cresol solubility procedure (Wilsonville no. 34550-21), used to quantify coal conversion, was found to have a reproducibility of ±0.7 wt % (2 standard deviations) at the 95% confidence level.

Solvent Fractionation. We compared the solvent fractionation procedures used by five subcontracted laboratories involved in SRC-I liquefaction (Kingsley and Schweighardt, 1982). A description of the solvent-extracted fractions is provided in Table 13 and the results are in Table 14. An important observation was that, for any one of the procedures, the sum of weight percent yields of oils and asphaltenes was significantly more precise than the yield of either fraction alone (Table 15). This result tells us that the separation of oils and asphaltenes is greatly affected by sample composition and the method of analysis.

Solvent fractionation by Soxhlet extraction (Wilsonville method no. 34550-3) is one of the most important analytical procedures for providing input data to material-balance calculations. We found the Soxhlet procedure the least precise and most difficult to reproduce between different operators on identical samples. Subsequent to Soxhlet extraction, the benzene solubles are treated with n-pentane to isolate oils and asphaltenes. The error associated with this separation by precipitation was >±8% absolute over the range of 10-50% by weight of oils. A major experimental error was the addition of 2-10 mL of tetrahydrofuran (THF) as a wash solvent to wash the benzene solubles into the n-pentane. This small amount of THF caused an uncontrollable cosolubility effect. Such differences directly impact material balance and yield calculations. Because different labs throughout the country use different alkane ($C_5$, $C_6$, $C_7$) solvents to define oils, we compared the solubility of an SRC-I solid in n-pentane, common hexanes, and n-heptane. The results are illustrated in Figure 2. Pentane, although the least active solvent, was found to give an approximate correlation to vacuum distillate overhead (~850°F), as shown in Table 16 and confirmed by a modified simulated distillation procedure.

*Vacuum Distillation and Simulated Distillation*. Distillation of material-balance sample streams to quantify a specific overhead, e.g., 450-850°F, had been considered routine because the ASTM D1160, D86, and D2887 procedures are well-established for petroleum materials. However, an investigation of the commonly practiced distillation and gas chromatography (GC) simulated distillation procedures pointed out a major problem that existed in comparing distillate data bases.

We found that Wilsonville did not use the ASTM D2887 procedure as written to determine boiling-point distribution from vacuum and atmospheric laboratory distillations. Wilsonville's procedure incorporates a blend of aromatics, alkanes, and polar components to relate retention times of specific peaks to absolute boiling points. Many other procedural differences were found that are outlined in the topical report (Kingsley and Schweighardt, 1981). Figure 3 summarizes how these differences impact the reported boiling-point distribution. We found that response factors must be used to relate area percent to weight percent when aromatic retention time standards are employed.

It is possible to reproduce the Wilsonville simulated distillation procedures for the entire range of material-balance sample distillates. Figure 4 gives the comparison between two laboratories. An important finding was that both the integration mode and integrator had to be matched before an acceptable fit was obtained.

While investigating the simulated distillation procedures, we found that the initial vacuum distillation method was more difficult to reproduce between laboratories than the simulated distillation. A round-robin study was conducted to compare the individual distillation procedures of four laboratories (Kingsley et al., 1983). All analyzed one common sample, a Wilsonville vacuum tower bottoms (T102), which is the feed to the critical solvent deasher. The results are given in Table 17. Each laboratory could reproduce its own work, but could not compare its results for end-point determination based only on temperature at a specific pressure for a cut point of 850°F. However, a correction was possible with a relative error of ±8%. A recently developed encapsulated GC simulated distillation procedure was used for each overhead sample and the data were corrected to 850°F as the end point, using the standard ASTM D2887 procedure. Table 18 shows the reproducibility results between four laboratories.

Our major observations from the distillation round-robin analyses were that we needed to define one specific vacuum distillation set-up, and to control the heat-up of the pot and head to within 25°F, with the head trailing the pot. Therefore, a second distillation round-robin was conducted based on those guidelines. Six laboratories participated, each receiving identical distillation glassware set-ups, custom-designed for controlling vacuum and temperature. Analytical results for the T102 bottoms sample were very precise (±2%), and reproducibility between laboratories was good, 6.5 ± 2.5 wt % overhead. These results were only achieved when the glassware was identical. Subsequent encapsulated GC simulated distillation showed the distillate bottoms to be nearly void of -850°F material and the interlaboratory yield of 450-850°F material was 33 ± 3 wt %.

Elemental Analysis. Elemental balances of oxygen, sulfur, and nitrogen are of particular importance in defining the efficiency of catalytic hydrotreatment of SRC or heavy distillates. However, when we analyzed organic nitrogen in the six major process streams of the Wilsonville H-OIL hydrotreater, results were random. Consequently, a round-robin analysis for elemental nitrogen was conducted (Schweighardt, 1983). A subset of the participants also conducted full-range elemental analysis (C, H, O, N, S). Table 19 lists the results of the nitrogen analysis. We concluded that Wilsonville's instrumental method suffered from a serious analytical sample-handling problem, which could be corrected by modifying the CHN procedure by encapsulating the test material, to retain light ends. We also concluded that trace nitrogen (<0.3 wt %) could be best analyzed by a more sensitive procedure such as the Antek chemiluminescence system. In general, elemental analysis procedures that included direct oxygen analysis provided the most precise elemental balances. The overall error analysis for two standard deviations was ±0.39 wt %. Table 20 summarizes the statistical comparisons.

## Task 2: Recycle Solvent Composition and Solvent Quality

"Solvent quality" is a contrived solvent property used in coal liquefaction. It is assumed to be an important guide for expressing how well a recycle solvent will convert coal to products soluble in tetrahydrofuran (THF) or pyridine. At the Wilsonville Advanced Coal Liquefaction Facility, solvent quality is measured by a kinetic microautoclave test. This test, originally developed by the Conoco Coal Development Company, has been used to quantify solvent quality at Wilsonville since 1978. The method defines solvent quality as the weight percent tetrahydrofuran solubles generated, based on weight percent moisture- and ash-free (MAF) coal.

This study identified important independent and dependent variables associated with the solvent refining of coal by relating plant operation to coal feedstock and product slate, and by correlating the impact to changes in the results from the microautoclave solvent quality test.

We have concluded that in SRC-I liquefaction, an effective process solvent should be (a) coal-derived to permit continuous plant operation; (b) a distillate with a nominal boiling range of 450-900°F; (c) able to sustain a 10-40 wt % coal slurry through feed pumps; (d) able to rapidly accept coal dissolution products in solution or suspension; (e) able to act as a hydrogen donor or shuttling agent for hydrogen transfer under a predominantly free-radical mechanism; and (f) capable of carrying the liquefaction product stream through solid/liquid separation processes and ultimately of being recycled to continue the process.

A typical first-stage recycle solvent generated under SRC-I process conditions exhibits a complex molecular composition. The solvent is composed of a mixture of alkyl ($C_1$-$C_4$)-substituted polynuclear hetero- and hydroaromatic compounds. Gas chromatography/mass spectrometry (GC/MS) data have revealed that 40 individual components compose almost 60 wt % of the solvent. The remaining 40 wt % may include hundreds to thousands of individual compounds. The major molecular species are substituted naphthalenes (5-30%) and phenanthrenes (5-10%).

During SRC-I liquefaction, each time the solvent/coal mixture passes through the reactor, 0-30 wt % new solvent material is generated, on an MAF feed coal basis. Therefore, we have found that the molecular composition constantly changes and shifts in response to processing conditions and feed slurry composition.

Figures 5, 6, and 7 graph weight percent distributions of 100°F distillate fractions, functional groups, and aromatic ring sizes, respectively, of a representative Wilsonville recycle solvent. We concluded from this study that the nominal molecular composition of Wilsonville recycle solvent does not reach a steady state in less than 30 days of coal processing. We have observed that, during the first 30 days of processing a new coal type with a lined-out solvent from the previous feed coal type, the total gas ($C_1$-$C_4$ and heteroatom) production (MAF coal) maximizes before establishing a coal-type dependent gas production rate nearly one-half of that observed during the first 30 days of operation. Figure 8 shows the relationship at Wilsonville over four years.

Solvent Quality. The concept of quantifying solvent quality by a microautoclave test does have merit if the test is run with the same coal type used for coal liquefaction. Correlation of the solvent quality result to preheater chemistry (rapid coal dissolution with minimum hydrogen shuttling) and reactor chemistry (rapid and sustaining hydrogen donation/shuttling) is apparently possible. As used at Wilsonville during runs 133-234, the microautoclave SQ result was of tangential significance for absolute day-to-day pilot plant operation.

From our SQ study of Wilsonville, it is our opinion that bench-scale batch experiments comparing different coals with solvents of questionable parentage for single-pass conversion are, at best, of limited relative value, and result in conflicting interpretations from lab to lab. Such experiments do define the kind of solvent composition that provides specific results. If such results or such solvents can be produced in situ is another question. Laboratory liquefaction experiments that may be the most meaningful (although the most time-consuming and expensive) are full recycle of the solvent in order to attain apparent steady-state operation. We estimate 5-12 solvent passes are required for line-out depending on process conditions, and the coal composition.

We have proposed a solvent quality test that takes into account the daily changes in feed coal, the impact of solvent action on primary liquefaction/coal dissolution, and sustaining conversion of large compounds to distillate in the presence of a donor solvent/hydrogen gas mixture. Figure 9 outlines the solvent quality test matrix.

Correlation of Coal Pyrite Content to SRC-I Liquefaction. Under program 12.1.10, we have observed that the pyrite content of the feed coal does impact primary coal dissolution. If the feed coal contains less than 1.2 wt % pyrite that is finely dispersed throughout the coal matrix, the results of a microautoclave solvent quality test average (n = 32) less than 70% tetrahydrofuran (THF) solubility. However, if the coal contains more than 1.2 wt % pyrite on average (n = 39), the THF solubility is >70% and the processing system produces the greatest distillate yields. Most important is that if there is >1.2% pyrite, the effect is not pyrite dependent but coal-type dependent. Figure 10 shows the relationship of pyrite content to microautoclave solvent quality (average of >70 points).

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## Table 1

Summary of Space and Labor Requirements for Newman Laboratory

| Area | Space ($ft^2$) | | Labor (hr/week) |
|---|---|---|---|
| Clean areas | | | |
| Environmental | 24 x 20 (480) | | 153.5 |
| Instrumental areas | | | |
| Microanalytical | 24 x 20 (480) | | 83.8-88.0 |
| Instrumental | 24 x 20 (480) | | 95.3 |
| GC | 12 x 20 (240) | | 77.3 |
| GC/LC | 12 x 20 (240) | | 17.9 |
| Dirty areas | | | |
| Distillation | 12 x 20 (240) | | 101.0 |
| Wet lab/ASTM | 24 x 20 (480) | | 210.4-233.5 |
| Solvent quality activity test | 12 x 20 (240) | | 55.5 |
| Utility areas | | | |
| Sample preparation/analysis | 12 x 20 (240) | | 18.3 |
| Cold storage | 24 x 20 (480) | | -- |
| Room temperature storage | 12 x 20 (240) | | -- |
| Chemical storage | 12 x 20 (240) | | -- |
| Sample receiving/stockroom | 12 x 20 (240) | | -- |
| Total | 4,320[a] | Total | 861.1-888.4[b] |

[a] 3,840 $ft^2$ if cold storage is not included in lab space estimate.
[b] Assuming 8-hr day and 100% lab efficiency.

### Table 2

### Qualifications of Personnel at the Demonstration Plant Laboratory

| Position | Qualifications |
|---|---|
| Quality Control Manager | Requires a B.S. Chemist with 20 years experience, including 10 years in a supervisory position; or possibly an M.S. Chemist with 10 years experience, including 5 years in a supervisory capacity. |
| Chemist 1 | A B.S./M.S. Chemist with 5 years experience; preferably major experience in instrumentation and separations, i.e., GC, LC |
| Chemist 2 | A B.S./M.S. Chemist with 5 years experience; some knowledge of wet chemical analysis, including AA/ICP |
| Technicians/shift leaders | Technicians should be hired with varying degrees of education and experience. Approximately one-third of the technicians should have either an A.A. degree and one year experience or 3-5 years experience; one-third could have an A.A. degree with no experience; the final one-third might not have an A.A. degree, but should have 1-3 years experience |

## Table 3

### Analytical Laboratory Labor Timetable[a]

| | Month/year | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 9/84 | | 1/85 | | 4/85 | | 8/85 | | 10/85 | | 12/85 |
| Area start-up | Coal receiving and wastewater treatment | | Gasifier | | SRC unit | | LC-Firer | | Critical solvent deashing unit | | Coker/calciner |
| Analytical areas (man-hr/wk) | Coal analysis (inc. particle size) | 18 | GC (30%) | 24 | Dist. (50%) | 50 | Wet | 210 | GC (40%) | 32 | |
| | Sample prep. | 18 | Environ. (40%) | 80 | Microanal. (50%) | 35 | SQAT | 55 | X-Ray (50%) | 20 | |
| | Environmental (60%) | 120 | | | GC (30%) | 24 | Dist. (50%) | 50 | Microanal. (50%) | 35 | |
| | LC | 18 | | | AA (50%) | 7 | | | | | |
| | AA & X-ray (50%) | 27 | | | | | | | | | |
| | Industrial hygiene | 10 | | | | | | | | | |
| Total man-hr/wk | | 211 | | 104 | | 116 | | 315 | | 37 | |
| 50% contingency for start-up | | 300 | | 150 | | 175 | | 472 | | 130 | |
| % total staff (22 technicians) | | 30 | | 15 | | 17 | | Remaining 38 | | | |

### Analytical Laboratory Staffing Phase-In Program[a]

| | Month/year | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 6/84[b] | 7/84 | 8/84 | 9/84 | 12/84 | 1/85 | 3/85 | 4/85 | 6/85 | 7/85 | 8/85 |
| % total staff on board (technicians) | | | | 30 | | 45 | | 62 | | | 100 |
| Number to be hired on specified date | | 4 | 3 | | 3 | | 3 | | 5 | 4 | |

[a] From Wood et al. (1981).

[b] The Quality Control Manager and two chemists should be hired on or before June 1984.

Table 4

Major Equipment Requirements for Demonstration Plant Analytical Laboratory[a]

| Laboratory | Equipment | Approximate cost (1981 dollars) |
|---|---|---|
| Environmental | Autotitrator | 10,000 |
| | Total organic carbon | 15,000 |
| | Ion chromatograph | 25,000 |
| | pH meter | 1,000 |
| | Incubator | 1,000 |
| | Oven (2) | 2,000 |
| | Five-place balance (2) | 7,000 |
| | Recorder | 1,500 |
| | Total | 62,500 |
| Microanalytical | Microtrac particle-size analyzer | 35,000 |
| | Dohrman low S&N analyzer | 15,000 |
| | O analyzer | 15,000 |
| | C, H, N, and S analyzer | 35,000 |
| | Microbalance and table | 5,500 |
| | Five-place balance | 3,500 |
| | Adiabatic calorimeter | 7,500 |
| | Total | 116,500 |
| Sample preparation/ analysis | Hardgrove grindability | 3,000 |
| Instrumentation | ICP/graphite furnace/autosampler | 90,000 |
| | Recorder | 1,500 |
| | Hg cold vapor/hydride | 6,000 |
| | Energy-dispersive X-ray | 72,500 |
| | Sampler changer | 12,500 |
| | Total | 182,500 |
| Wet lab/ASTM | Fisher coal analyzer | 12,000 |
| | Oven and muffle furnace | 2,200 |
| | Five-place balance (2) | 7,000 |
| | Karl-Fischer titrator | 5,000 |
| | Closed flash tester | 1,000 |
| | Reid vapor-pressure bombs (3) | 1,500 |
| | Copper strip bombs (3) | 2,000 |
| | Accelerated gum bombs (3) | 2,500 |
| | Kinematic viscosity bath | 1,500 |
| | Total | 34,700 |
| Gas chromatography/ liquid chromatography | LC pumping system and microprocessor | 20,000 |
| | Ultraviolet/visible and fluorescence detectors | 20,000 |
| | Integrator (2) | 8,000 |
| | Recorder (3) | 4,500 |
| | Simulated distillation unit | 20,000 |
| | Capillary-column GC (2) | 25,000 |
| | Packed-column GC | 15,000 |
| | Total | 112,500 |
| Solvent quality activity test | Shaker arrangement, microautoclave, and sand bath system | 30,000 |
| | Millipore filters and accessories (3) | 6,000 |
| | Ultrasonic probe and accessories | 2,500 |
| | Rotary evaporator (2) | 4,000 |
| | Total | 42,500 |
| Distillation | Product work-up still (4) | 4,000 |

[a] Klusaritz and Znaimer (1981).

## Table 5

### Summary of Chemical Waste from Analytical Laboratory for 1-Week Period

| Type of waste | Amount |
|---|---|
| SRC liquid[a] | 14 gal |
| SRC solid and coal[a] | 18 lb |
| Nonhalogenated solvents | 28 gal |
| Halogenated solvents | 4 gal |
| Wastewater[b] | 11 gal |
| Benzene | 1 gal |

[a] Assume most will be recycled through process.

[b] Assume will be recycled through wastewater treatment section.

### Table 6
### Retention Indices[a]
### Calculated from Standard Solutions
### Using Different Chromatographs
### and Different Columns

| Compound | Lee et al. (1979) | PE 3920[d] | Varian 4600[d] | PE Sigma[d] |
|---|---|---|---|---|
| Tetralin[b] | 197.04 ± 0.5 | 196.58 ± 0.04[e] | 196.60 | 196.15 |
| Naphthalene | 200 | 200 | 200 | 200 |
| Quinoline[c] | 209.70 | 209.25 ± 0.09 | 208.70 | 208.30 |
| 1-Methylnaphthalene | 221.04 ± 0.25 | 222.48 ± 0.46 | 221.77 | 221.30 |
| Biphenyl | 233.96 ± 0.24 | 235.53 ± 0.36 | 234.22 | 233.81 |
| 3-Methylindole[c] | 236.66 | 236.85 ± 0.14 | 235.69 | 235.86 |
| 2,6-Dimethylnaphthalene | 237.58 ± 0.17 | 239.41 ± 0.33 | 237.87 | 237.30 |
| Acenaphthene | 251.29 ± 0.14 | 253.06 ± 0.23 | 251.63 | 250.90 |
| Dibenzofuran[c] | 257.17 ± 0.05 | 257.81 ± 0.13 | 256.59 | 256.92 |
| Fluorene | 268.17 ± 0.15 | 269.50 ± 0.26 | 268.30 | 267.87 |
| Xanthene | 280.48 ± 0.22 | 280.92 ± 0.17 | 279.60 | 279.30 |
| 9,10-Dihydroanthracene[c] | 284.89 ± 0.19 | 284.87 ± 0.21 | 283.40 | 284.54 |
| Dibenzothiophene | 295.81 ± 0.03 | 295.67 ± 0.21 | 294.85 | 294.71 |
| Phenanthrene | 300 | 300 | 300 | 300 |
| Phenanthridine | 308.97 ± 0.28 | 307.32 ± 0.32 | 306.57 | 306.36 |
| Carbazole[c] | 312.13 | 311.13 ± 0.03 | 310.09 | 311.48 |
| 1-Methylphenanthrene | 323.90 ± 0.08 | 323.78 ± 0.28 | 322.92 | 322.23 |
| Pyrene | 351.22 ± 0.08 | 351.94 ± 0.12 | 351.88 | 351.21 |
| Chrysene | 400 | 400 | 400 | 400 |

[a] The method for calculating retention indices is defined in the text.
[b] Calculated by assuming that the retention time difference between benzene and naphthalene is equal to the difference between naphthalene and phenanthrene.
[c] These are heterospecies which behave differently on different columns; see Parees and Kamzelski (1982).
[d] See Table 3 of Parees and Kamzelski (1982) for Varian 4600 GC column and conditions; see Table 4 for PE 3920 and Sigma GC columns and conditions.
[e] These values are the average of three runs with the standard deviation included.

Table 7
Retention Indices[a]
Calculated from Different Solutions
Analyzed on the Same GC[b]

| Compound | Standard solution | Coal tar | Process solvent |
|---|---|---|---|
| Tetralin | 196.58 | --- | 196.43 |
| Naphthalene | 200 | 200 | 200 |
| 2-Methylnaphthalene | --- | 218.90 | 219.90 |
| 1-Methylnaphthalene | 222.48 | --- | 222.90 |
| Biphenyl | 235.53 | 234.55 | 235.46 |
| Acenaphthene | 253.06 | --- | 252.45 |
| Dibenzofuran | 257.81 | 257.52 | 258.16 |
| Fluorene | 269.50 | 268.71 | 269.05 |
| Dibenzothiophene | 295.67 | --- | 295.00 |
| Phenanthrene | 300 | 300 | 300 |
| Fluoranthene | --- | 344.78 | 344.37 |
| Pyrene | 351.94 | 351.97 | 351.60 |
| Chrysene | 400 | 400 | 400 |

[a] Calculation of retention indices is defined in Parees and Kamzelski (1982).

[b] These analyses were all done on the PE-3920 gas chromatograph with the glass SE-52 column; see Table 4 of Parees and Kamzelski for specific conditions.

Table 8

Precision Studies: Comparison of Analytical Parameters

| Method[a] | Column | Area percentage MSD[b] | Comments |
|---|---|---|---|
| 1. Perkin-Elmer 3920 GC<br>Spectra-Physics 4100 integrator<br>Split injection 55:1<br>Hamilton syringe<br>Operator #1 | Glass SE-52 | 1.27 | 350-850°F process solvent distillate. Nine compounds chosen with areas of 1.2-5.5%. |
| 2. Perkin-Elmer 3920 GC<br>Spectra-Physics 4100 integrator<br>Split injection 14:1<br>Diluted sample<br>Hamilton syringe<br>Operator #1 | Glass SE-52 | 12.3 | Same sample and components as method 1. Areas of 1.5-5.3% |
| 3. Perkin-Elmer Sigma 1 GC and integrator<br>Splitless injection<br>Diluted sample<br>Hamilton syringe<br>Operator #1 | Fused silica SE-54 | 4.38 | Same sample as method 1. Ten compounds chosen with areas of 0.9-6.0%. |
| 4. Varian Vista GC and integrator<br>Split injection 48:1<br>Hamilton syringe<br>Operator #1 | Fused silica SE-54 | 3.98 | Same sample as method 1. Same compounds as method 3. Areas of 1.2-5.7% |
| 5. Finnigan 9610 GC<br>Finnigan 4000 MS<br>Finnigan INCOS integrator<br>Split injection 50:1<br>Hamilton syringe<br>Operator #1 | Glass SE-52 | 5.24 | 400-650°F process solvent distillate. Seven compounds chosen with areas of 1.5-10.1% (limited number of peaks included in quantitation, causing percentage assigned to each to be high). |
| 6. Perkin-Elmer 3920 GC<br>Spectra-Physics 4100 integrator<br>Split injection 55:1<br>Hamilton syringe<br>Operator #2 | Glass SE-52 | 1.86 | Same sample as method 5. Twelve compounds chosen with areas of 1.0-4.5%. The A % MSD drops to 1.07 if a value of ±10% on a 2.2 A % peak is excluded. |
| 7. Perkin-Elmer 3920 GC<br>Spectra-Physics 4100 integrator<br>Split injection 55:1<br>S.G.E. syringe<br>Operator #2 | Glass SE-52 | 3.97 | Same sample as method 5. Thirteen compounds chosen with areas of 0.5-5.2%. The A % MSD drops to 3.32 if a value of ±12% on a 0.60 A % peak is excluded. |
| 8. Perkin-Elmer Sigma 1 GC<br>Spectra-Physics 4100 integrator<br>Split injection 42:1<br>Hamilton syringe<br>Operator #1 | Fused silica SE-54 | 5.69 | Same sample as method 5. Fourteen compounds chosen with areas of 0.45-6.6%. The A % MSD drops to 1.8 if four bad peaks with areas of 1.8-6.1% are excluded. |

[a] The Hamilton syringe was 1.0-μL positive displacement type with a Chaney adapter for volume precision. The Scientific Glass Engineering (S.G.E.) syringe was 1.0-μL positive displacement type. The injection volume was 0.2 μL in all cases, except 0.9 μL was injected in case 3. In cases 2 and 3, the sample was diluted appropriately to hold the amount of sample entering the column approximately equal to the other analyses. The glass SE-52 column used in case 5 is nominally the same as that used in cases 1, 2, 6, and 7; however, it is not identical. The fused-silica column used in case 4 is very similar but not identical to the column in cases 3 and 8 (same manufacturer and nominal specifications, but slightly different internal diameter). The standard deviations are the average result of triplicate analyses. The standard deviations for individual peaks did not correlate with peak area. That is, the average standard deviations for 0.5 A % components were similar to 1-2 A % components and 4-6 A % components.

[b] MSD, mean standard deviation.

Table 9

Response Factors for the Larger Group of Selected Standard Compounds Determined on the Varian Vista GC[a]

| Compound | Response factor[b] | Weight percent[c] | µg/µL[d] | ng[e] |
|---|---|---|---|---|
| Phenol | 1.407 ± 0.016 | 1.11 | 10.10 | 38.8 |
| Indan | 1.046 ± 0.006 | 1.10 | 10.04 | 38.6 |
| m-Cresol | 1.297 ± 0.008 | 1.29 | 11.83 | 45.5 |
| Ethylaniline | 1.297 ± 0.008 | 0.56 | 5.10 | 19.6 |
| Naphthalene | 1.000 | 2.00 | 17.64 | 67.8 |
| Dimethylphenol | 1.409 ± 0.012 | 0.54 | 4.94 | 19.0 |
| Dodecane | 1.042 ± 0.053 | 0.65 | 5.96 | 22.4 |
| 1-Methylnaphthalene | 1.028 ± 0.006 | 2.77 | 25.34 | 97.5 |
| Tetrahydroquinoline | 1.186 ± 0.029 | 0.65 | 5.97 | 23.0 |
| Biphenyl | 1.995 ± 0.010 | 1.39 | 12.73 | 49.0 |
| Dimethylnaphthalene | 1.006 ± 0.013 | 0.75 | 6.83 | 26.3 |
| Diphenyl ether | 1.195 ± 0.021 | 2.84 | 25.96 | 99.8 |
| Acenaphthene | 1.005 ± 0.021 | 1.73 | 15.79 | 60.7 |
| 1-Naphthol | 1.472 ± 0.113 | 0.66 | 6.04 | 23.2 |
| Fluorene | 1.036 ± 0.068 | 1.50 | 13.71 | 52.7 |
| Xanthene | 1.100 ± 0.137 | 0.62 | 5.69 | 21.9 |
| Dibenzothiophene | 1.134 ± 0.091 | 1.14 | 10.44 | 40.2 |
| Phenanthrene | 1.030 ± 0.039 | 2.99 | 27.29 | 105.0 |
| Phenanthridine | 1.092 ± 0.212 | 0.76 | 6.95 | 26.7 |
| 1-Methylphenanthrene | 1.115 ± 0.178 | 0.40 | 3.61 | 13.9 |
| Nonadecane | 1.184 ± 0.080 | 0.31 | 2.86 | 11.0 |
| Pyrene | 1.064 ± 0.048 | 1.30 | 11.93 | 45.9 |
| Triphenylene | 1.164 ± 0.086 | 0.24 | 2.21 | 8.5 |
| Hexacosane | 1.489 ± 0.105 | 0.24 | 2.22 | 8.5 |
| Perylene | 1.558 ± 0.245 | 0.23 | 2.10 | 8.1 |
| Dibenzoanthracene | 2.483 ± 0.721 | 0.10 | 0.92 | 3.5 |
| Dotriacontane | 2.125 ± 0.124 | 0.20 | 1.80 | 6.9 |

[a] These analyses were done in triplicate by using the Varian-Vista GC/FID and a fused-silica SE54 capillary column. The triplicate analyses for each solution were carried out within a single day. The individual dilute standard solutions were analyzed on different days, however.

[b] As determined vs. naphthalene held constant at 2%:

$$RF = \frac{\text{integration area (naphthalene)}}{\text{weight percent (naphthalene)}} \times \frac{\text{weight percent (compound)}}{\text{integration area (compound)}}$$

[c] $$\text{Weight percent} = \frac{\text{amount weighed x purity}}{\text{total weight of solution}} \times 100$$

The solvent was toluene.

[d] $$\mu g/\mu L = \frac{\text{(amount weighed in grams) x purity}}{10\ \mu L}$$

[e] $$ng = \frac{(\mu g/\mu L)(0.2\ \mu L\ \text{injected})(1{,}000\ ng/\mu g)}{53\ \text{(split ratio factor)}}$$

This is the amount actually delivered to the column by assuming strict splitting linearity.

## Table 10

## Precision of 10-mg Micropellet Analysis

| Sample | % calculated $Al_2O_3$ | $SiO_2$ | $K_2O$ | CaO | $TiO_2$ | $Fe_2O_3$ |
|---|---|---|---|---|---|---|
| 81-2015 | | | | | | |
| Pellet A | 23.6 | 55.2 | 1.80 | 0.70 | 0.67 | 18.0 |
| Pellet B | 24.1 | 55.8 | 1.82 | 0.69 | 0.65 | 17.0 |
| Pellet C | 23.2 | 56.1 | 1.76 | 0.58 | 0.66 | 17.7 |
| Mean concn | 23.6 | 55.7 | 1.79 | 0.66 | 0.66 | 17.6 |
| $1\sigma = [\Sigma\Delta^2/(n - 1)]^{\frac{1}{2}} =$ | ±0.45 | ±0.46 | ±0.03 | ±0.07 | ±0.01 | ±0.51 |
| % relative error | 1.91 | 0.83 | 1.67 | 10.6 | 1.51 | 2.90 |

## Table 11

### EDXRF Coal and KMAC Method: Summary of 12 Coal Standards Analyzed as Samples by EDXRF

| | $Al_2O_3$ | $SiO_2$ | $SO_3$ | $K_2O$ | CaO | $TiO_2$ | $Fe_2O_3$ |
|---|---|---|---|---|---|---|---|
| Range (%) | 0.87-5.96 | 1.53-14.71 | 1.30-11.44 | 0.09-0.67 | 0.08-2.04 | 0.06-0.27 | 0.32-5.2 |
| Mean concentraction (%) | 2.54 | 5.77 | 6.55 | 0.27 | 0.44 | 0.13 | 2.37 |
| Mean % relative error | 3.18 | 2.30 | 2.67 | 3.33 | 3.43 | 5.42 | 3.25 |

## Table 12

### Metals Analysis cf SRC and KMAC (Weight Percent of As-Received, Whole Sample)[a]

| | SRC 1 | | | | | SRC 2 | | | | | SRC 3 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Element | Gal | IMMR | APCI, EDXRF | APCI, AAS | Alcoa | Gal | IMMR | APCI, EDXRF | ACPI, AAS | Alcoa | Gal | IMMR | APCI, EDXRF | APCI, AAS | Alcoa |
| Ash | 0.041/<br>0.096 | 0.0999 | 0.11 | 0.09 | 0.08 | 0.024/<br>0.075 | 0.0789 | 0.08 | 0.21 | 0.07 | 0.037/<br>0.067 | 0.0759 | 0.09 | 0.10 | 0.05 |
| S | 0.99 | 0.98 | - | 0.71 | 1.07 | 0.96 | 0.99 | - | 0.82 | 1.15 | 0.94 | 0.99 | - | 0.75 | 1.10 |
| P | 0.0004 | - | <0.0001 | <0.0036 | <0.0001 | 0.0023 | - | <0.00008 | <0.0036 | <0.0001 | 0.0030 | - | <0.00009 | 0.0060 | <0.0001 |
| Si | 0.019 | 0.00271 | 0.00213 | <0.045 | 0.001 | 0.015 | 0.00147 | 0.00248 | <0.045 | 0.003 | 0.02 | 0.00111 | 0.00092 | <0.045 | 0.001 |
| Fe | 0.0028 | 0.005 | 0.0225 | <0.0065 | 0.004 | 0.0027 | 0.00374 | 0.01472 | <0.0065 | 0.006 | 0.0022 | 0.0031 | 0.0148 | <0.0065 | 0.003 |
| Ti | <0.0050 | 0.0115 | 0.02117 | 0.018 | 0.008 | <0.0050 | 0.0122 | 0.01842 | 0.018 | 0.010 | <0.0050 | 0.0073 | 0.02142 | 0.0150 | 0.008 |
| Ca | 0.0035 | 0.00269 | 0.00395 | 0.0055 | 0.002 | 0.0031 | 0.00225 | 0.00257 | 0.0050 | 0.002 | 0.0030 | 0.00183 | 0.00332 | 0.0050 | 0.004 |
| Mg | <0.0003 | 0.000397 | <0.0003 | <0.0030 | 0.001 | 0.00017 | 0.00032 | <0.00024 | <0.0030 | 0.001 | 0.0002 | 0.00034 | <0.0003 | <0.0030 | 0.001 |
| Na | 0.0022 | 0.0131 | -0.0044 | 0.24 | 0.026 | 0.0015 | 0.0069 | ~0.0016 | 0.0120 | 0.012 | 0.0021 | 0.00711 | -0.0036 | 0.0140 | 0.014 |
| K | 0.0013 | 0.00045 | 0.000612 | 0.020 | 0.002 | 0.0011 | 0.00029 | 0.00096 | <0.0040 | 0.003 | 0.0014 | 0.000201 | 0.0003 | <0.0040 | 0.002 |
| Ni | 0.0006 | 0.000103 | <0.0011 | <0.0035 | 0.000 | 0.0006 | 0.000097 | <0.0008 | <0.0035 | 0.000 | <0.0005 | 0.000091 | <0.0009 | <0.0035 | 0.000 |
| Mn | <0.0002 | 0.000088 | <0.0011 | <0.0006 | 0.000 | <0.0002 | 0.000079 | <0.00008 | <0.0006 | 0.000 | <0.0002 | 0.000073 | <0.00009 | <0.0006 | 0.000 |
| Zn | 0.0002 | 0.00044 | <0.00011 | <0.0006 | 0.000 | 0.0009 | 0.000035 | <0.00008 | <0.0006 | 0.000 | 0.0001 | 0.000036 | <0.00009 | <0.0006 | 0.000 |
| V | <0.0025 | 0.00143 | -0.001 | <0.008 | 0.001 | <0.0025 | 0.0014 | -0.0009 | <0.008 | 0.001 | <0.0025 | 0.00125 | -0.00108 | <0.0080 | 0.001 |
| B | 0.0098 | 0.00518 | - | <0.0075 | 0.010 | 0.0072 | 0.00808 | - | 0.0120 | 0.011 | 0.0067 | 0.00436 | - | 0.0120 | 0.012 |
| Al | 0.0072 | 0.00714 | 0.01112 | 0.0085 | 0.005 | 0.0062 | 0.00598 | 0.00694 | 0.0085 | 0.007 | 0.0059 | 0.00534 | 0.00943 | 0.0085 | 0.005 |

| | SRC 4 (sp. proc) | | | | SRC 5 (without $Na_2CO_3$) | | KMAC 1 | | | | | KMAC 2 | KMAC 3 | | KMAC 4 | | KMAC 5 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Element | Gal | IMMR | Alcoa | APCI, EDXRF | Gal | IMMR | Gal | IMMR | APCI, EDXRF | APCI, AAS | Alcoa | IMMR | Gal | IMMR | Gal | IMMR | IMMR |
| Ash | 0.013/<br>0.18 | 0.1524 | 0.10 | 0.14 | 0.13 | 0.0658 | 29.39 | 26.89 | 28.73 | 29.4 | 29.1 | 25.35 | 28.93 | 29.57 | 27.53 | 27.80 | 31.13 |
| S | 1.05 | 1.05 | 1.11 | | 0.92 | 0.87 | - | 2.66 | - | 3.30 | 2.98 | 2.47 | | 3.08 | - | 2.76 | 3.20 |
| P | 0.0045 | - | <0.0001 | | 0.0010 | | 0.01796 | 0.0107 | <0.0287 | 0.030 | <0.03 | 0.0088 | 0.0076 | 0.0098 | 0.0084 | 0.0097 | 0.0136 |
| Si | 0.053 | 0.0128 | 0.002 | 0.0028 | 0.0402 | 0.00657 | 6.879 | 6.17 | 6.808 | 1.50 | 6.14 | 5.84 | 6.714 | 6.78 | 6.49 | 6.45 | 7.06 |
| Fe | 0.0019 | 0.00667 | 0.008 | 0.0179 | 0.0077 | 0.00834 | 4.082 | 3.71 | 4.159 | 1.40 | 4.26 | 3.60 | 4.053 | 4.37 | 3.782 | 3.94 | 4.57 |
| Ti | 0.0063 | 0.0131 | 0.012 | 0.0311 | 0.0075 | 0.00737 | <0.0529 | 0.17 | 0.1412 | 0.25 | 0.15 | 0.15 | 0.196 | 0.17 | 0.1815 | 0.17 | 0.18 |
| Ca | 0.0016 | 0.00765 | 0.005 | 0.008 | 0.0032 | 0.00337 | 0.464 | 0.44 | 0.3716 | 0.41 | 0.35 | 0.41 | 0.4197 | 0.49 | 0.419 | 0.46 | 0.54 |
| Mg | 0.0002 | 0.00219 | 0.002 | | 0.0002 | 0.00036 | 0.1312 | 0.12 | <0.0862 | 0.11 | 0.12 | 0.11 | 0.122 | 0.13 | 0.1195 | 0.13 | 0.14 |
| Na | 0.0105 | 0.0152 | 0.020 | | 0.0019 | 0.00111 | 0.543 | 0.62 | -0.5746 | 0.30 | 0.65 | 0.36 | 0.47 | 0.49 | 0.2369 | 0.25 | 0.65 |
| K | 0.0002 | 0.00145 | 0.002 | | 0.0006 | 0.00052 | 0.4026 | 0.48 | 0.5009 | 0.46 | 0.51 | 0.46 | 0.387 | 0.53 | 0.3771 | 0.51 | 0.57 |
| Ni | <0.0001 | 0.00012 | 0.000 | | <0.0001 | 0.00009 | 0.00693 | 0.00226 | <0.00144 | 0.0050 | 0.006 | 0.00205 | 0.00682 | 0.00211 | 0.00649 | 0.00239 | 0.00255 |
| Mn | <0.0001 | 0.00008 | 0.000 | | 0.0001 | 0.00011 | 0.00728 | 0.0064 | <0.0287 | 0.0045 | 0.006 | 0.00662 | 0.00716 | 0.0076 | 0.00682 | 0.00714 | 0.00722 |
| Zr | 0.0001 | 0.00006 | 0.000 | | 0.0002 | 0.00018 | 0.00874 | 0.0078 | <0.0287 | 0.0055 | 0.008 | 0.00783 | 0.0139 | 0.016 | 0.00885 | 0.00784 | 0.0106 |
| V | 0.0013 | 0.00079 | 0.001 | | <0.0009 | 0.00073 | <0.0329 | 0.0407 | <0.0287 | 0.012 | 0.016 | 0.0389 | 0.0146 | 0.0439 | 0.01388 | 0.0398 | 0.0556 |
| B | 0.0038 | 0.0072 | 0.014 | | 0.0043 | 0.00412 | 0.00274 | - | - | 0.038 | 0.05 | - | 0.0036 | - | 0.00171 | - | - |
| Al | 0.0075 | 0.00921 | 0.008 | 0.0091 | 0.0050 | 0.00598 | 2.669 | 2.77 | 3.254 | 2.50 | 2.72 | 2.62 | 2.754 | 3.02 | 2.706 | 2.90 | 3.14 |

---

[a] Column headings refer to sample numbers and laboratories.

Table 13

**Definitions of the Solvent-Extracted Fractions**
**(Listed by Participants and Procedure)**

| Lab. | Fraction (procedure) | | | |
|---|---|---|---|---|
| | Oils | Asphaltenes | Preasphaltenes | Residue |
| Wilsonville | Benzene soluble<br>Pentane soluble<br>(Soxhlet/beaker extraction) | Benzene soluble<br>Pentane insoluble<br>(Soxhlet/beaker extraction) | Cresol soluble<br>Benzene insoluble<br>(Calculated) | Cresol insoluble<br>(Beaker extraction) |
| ICRC | Pentane soluble<br>(Sequential solvent extraction) | Benzene soluble<br>Pentane insoluble<br>(Sequential solvent extraction) | Pyridine soluble<br>Benzene insoluble<br>(Sequential solvent extraction) | Pyridine insoluble<br>(Sequential solvent extraction) |
| Kerr-McGee | Heptane soluble<br>(Beaker extraction) | Toluene soluble<br>Heptane insoluble<br>(Beaker extraction) | Pyridine soluble<br>Toluene insoluble<br>(Calculated) | Pyridine insoluble<br>(Beaker extraction) |
| Conoco | Hexanes soluble<br>(LCF) | Benzene soluble<br>Hexanes insoluble<br>(LCF) | THF soluble<br>Benzene insoluble<br>(LCF) | THF insoluble<br>(Beaker extraction) |

## Table 14

### Weight Percent Yields of Distillates and Solvent-Extracted Fractions from Wilsonville Process-Stream Samples as Analyzed by the Participants

| | Lab. | Dist. | Oils | Asph. | Preasph. | Residue | TI/BI[a] |
|---|---|---|---|---|---|---|---|
| T102 btm | WV | 11.4 | 19.4 | 39.3 | 25.5 | 15.8 | 41.3 |
| | ICRC/APCI | 15.5 | 6.9 | 48.4 | 28.5 | 16.0 | 44.5 |
| | KMG | 25.3 | 7.4 | 47.8 | 29.2 | 15.6 | 44.8 |
| | Conoco | - | 31.4 | 22.7 | 24.3 | 21.6 | 45.9 |
| LSRC[d] | WV | 38.5 | 39.4 | 60.3 | 0.3 | 0 | 0.3 |
| | ICRC/APCI | 35.3 | 47.4 | 47.2 | 4.1 | 0 | 4.1 |
| | KMG | 62.2 | 31.5 | 65.4 | 3.1 | 0 | 3.1 |
| | Conoco | - | 71.9 | 22.1 | 6.0 | 0 | 6.0 |
| SRC | WV | 11.4 | 20.2 | 58.8 | 20.5 | 0.5 | 21.0 |
| | ICRC/APCI | 17.9 | 8.9 | 62.2 | 28.9 | 0 | 28.9 |
| | KMG | 24.6 | 8.6 | 60.7 | 30.7 | 0 | 30.7 |
| | Conoco | - | 41.0 | 33.9 | 25.1 | 0 | 25.1 |
| KMAC | WV | 2.8 | 1.5 | 1.2 | 30.3 | 67.0 | 97.3 |
| | ICRC/APCI | 1.9 | 1.8 | 2.3 | 34.3 | 61.6 | 95.9 |
| | KMG | 3.4 | 1.5 | 4.8 | 39.3 | 54.4 | 93.7 |
| | Conoco | - | 4.2 | 4.9 | 10.6 | 80.6 | 91.2 |
| V110 | WV | 56.7 | 63.5 | 17.2 | 11.9 | 7.4 | 19.3 |
| | ICRC/APCI | 54.4 | 61.5 | 16.5 | 13.6 | 8.3 | 21.9 |
| | KMG | 64.7 | 59.4 | 21.5 | 13.8 | 5.3 | 19.1 |
| | Conoco | 53.0 | 67.8[b] | 10.5 | 11.0 | 10.7 | 21.7 |
| 30/70 | WV | 75.1 | 85.1[b] | 13.8 | 0.9 | 0.2 | 1.1 |
| | ICRC/APCI | 81.4 | 88.1 | 10.6 | 1.3 | 0 | 1.3 |
| | KMG | 89.0 | 87.2 | 12.8 | 0 | 0 | 0 |
| | Conoco | 75.2 | 91.2[b] | 7.0 | 1.8 | 0 | 1.8 |
| V131A | WV | 98.7 | 99.7[b] | 0.3 | 0 | 0 | 0 |
| | ICRC/APCI | 98.6 | 98.1 | 1.6 | 0.3 | 0 | 0.3 |
| | KMG | 99.6 | 99.6 | 0.4 | 0 | 0 | 0 |
| | Conoco | - | 100.0[c] | 0 | 0 | 0 | 0 |

[a] Toluene or benzene insolubles; sum of preasphaltenes and residue.

[b] Sum of distillate and oils after distillation of sample.

[c] Assumed.

[d] Data on all LSRC fractions except distillate is proprietary to Kerr-McGee.

## Table 15

### Material Balance of Benzene/Toluene Solubles
### (Sum of Oils and Asphaltenes for the T102 Bottoms Samples)

| Participant | Wt % oils + asph. in feed | Σ, wt % oils + asph. in summed product fractions | % recovery |
|---|---|---|---|
| WV | 58.7 | 57.8 | 98 |
| ICRC | 55.3 | 53.2 | 96 |
| Kerr-McGee | 55.2 | 53.3 | 97 |
| Conoco | 54.1 | 56.7 | 105 |

Table 16

Comparison of Distillate Yield (by Distillation I, Column Heated) with Oil Yield (by Solvent Separation)

| Sample | Oil yield (wt %) | Distillate yield (wt %) |
|---|---|---|
| 23-169 | 72.00[a] | 72.2 |
| 32-15,37 | 68.1[b] | 69.1 |
| 32-87,101 | 63.9[b] | 65.4[b] |
| 32-125,139 | 70.0[b] | 69.4 |
| 33-21,28 | 66.5[b] | 66.9 |
| 33-94,101,108 | 72.3[b] | 71.6[b] |
| 33-137,151 | 68.9[b] | 66.2[b] |
| 33-173,180,187 | 69.2[a] | 68.5[a] |
| 36-15,30 | 72.6[b] | 69.5 |
| 36-56,63 | 71.9[b] | 71.2 |
| 36-85,92 | 67.1[b] | 63.6 |
| 36-110,117 | 68.4 | 65.0 |
| 36-136 | 67.8 | 64.1 |

[a] Average of three determinations.

[b] Average of two determinations.

## Table 17

### Weight Percent Distillate from Each Distillation Procedure

| Laboratory | Distillation method[a] I | II | III |
|---|---|---|---|
| (A) Conoco | 14.8 | 6.4 | 5.0 |
| (B) Wilsonville | 11.9 | 9.4 | 9.3 |
| (C) Kerr-McGee | 32.3 | 11.4 | 20.5 |
| (D) APCI/ICRC | 9.1 | 4.9 | 11.1 |
| $\bar{x}$ | 17.1 | 8.0 | 11.5 |
| s | 10.5 | 3.0 | 6.5 |

### Conditions for Three Distillation Procedures Analyzed in Round-Robin Study

| Condition | Distillation method I | II | III |
|---|---|---|---|
| Final pot temperature (°F)[b] | 600 | 554 | 500 |
| Pressure (mm Hg) | 0.1 | 1.0 | 0.1 |
| End point (°F) (measured at pot temperature) | 600 | 554 | 500, hold 1 hr |

---

[a] The reproducibility of any one distillation procedure at any one laboratory was less than ±1 wt % on average.

[b] Pot temperatures adjusted to atmospheric pressure are 1,370, 1,250, and 1,245°F for methods I, II, and III, respectively (according to Maxwell and Bonnell, 1955).

## Table 18

## Total Weight Percent of Distillate (<850°F) of T102 Btm Sample after Adjustment by Encapsulated GCSD

| | Distillation method | | |
|---|---|---|---|
| Laboratory | I | II | III |
| (A) Conoco | 8.2 | 4.9 | 4.0 |
| (B) Wilsonville | 7.5 | 5.8 | 6.5 |
| (C) Kerr-McGee | 9.6 | 5.1 | 8.1 |
| (D) APCI/ICRC | 5.6 | 3.6 | 6.7 |
| $\bar{x}$ | 7.7 | 4.8 | 6.3 |
| s | 1.7 | 0.9 | 1.7 |

## Table 19

## Summary of Nitrogen Analysis, by Method or Laboratory

| | | | | | | Galbraith | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Sample | WV | Huffman | APCI | PETC | Kerr-McGee | Leco | Kjeldahl | Antek | Perkin-Elmer | Carlo Erba |
| Process wastewater (91155/V-7016) | -- | 1.35 | 1.22 | 1.3 | 1.41 | -- | 1.50 | 1.32 | 1.55 | 0.80 |
| Flashed distillate (91156/V-7017) | 1.44 | 0.56 | 0.11 | 0.2 | 0.19 | -- | 0.26 | 0.24 | 0.28 | 0.31 |
| Heavy resid (91158/V-7018) | 1.49 | 1.53 | 1.44 | 1.5 | 1.38 | 1.75 | 1.56 | 1.59 | 1.62 | 1.54 |
| SRC melt tank (91160/V-7019) | 1.07 | 1.23 | 1.23 | 1.2 | 1.06 | 1.32 | 1.30 | 1.09 | 1.10 | 1.18 |
| Vacuum flash overhead (91157/V-7020) | 0.36 | 0.59 | 0.37 | 0.4 | 0.40 | -- | 0.41 | 0.33 | 0.63 | 0.56 |
| Recycle solvent (91159/V-7021) | 0.33 | 0.69 | 0.31 | 0.4 | 0.35 | -- | 0.42 | 0.47 | 0.33 | 0.54 |
| Standard A[a] (V-7022) | 2.47 | 1.46 | 1.16 | 1.3 | 1.11 | -- | 0.93 | 0.90 | 1.15 | 0.53 |
| Standard B[b] (V-7023) | 1.70 | <0.11 | <0.08 | 0.2 | 0.13 | -- | 0.06 | 0.19 | 0.20 | 0.29 |
| Standard C[c] (V-8151) | 8.25 | 8.43 | 8.50 | 8.2 | 8.03 | -- | 8.43 | 8.11 | 8.43 | -- |

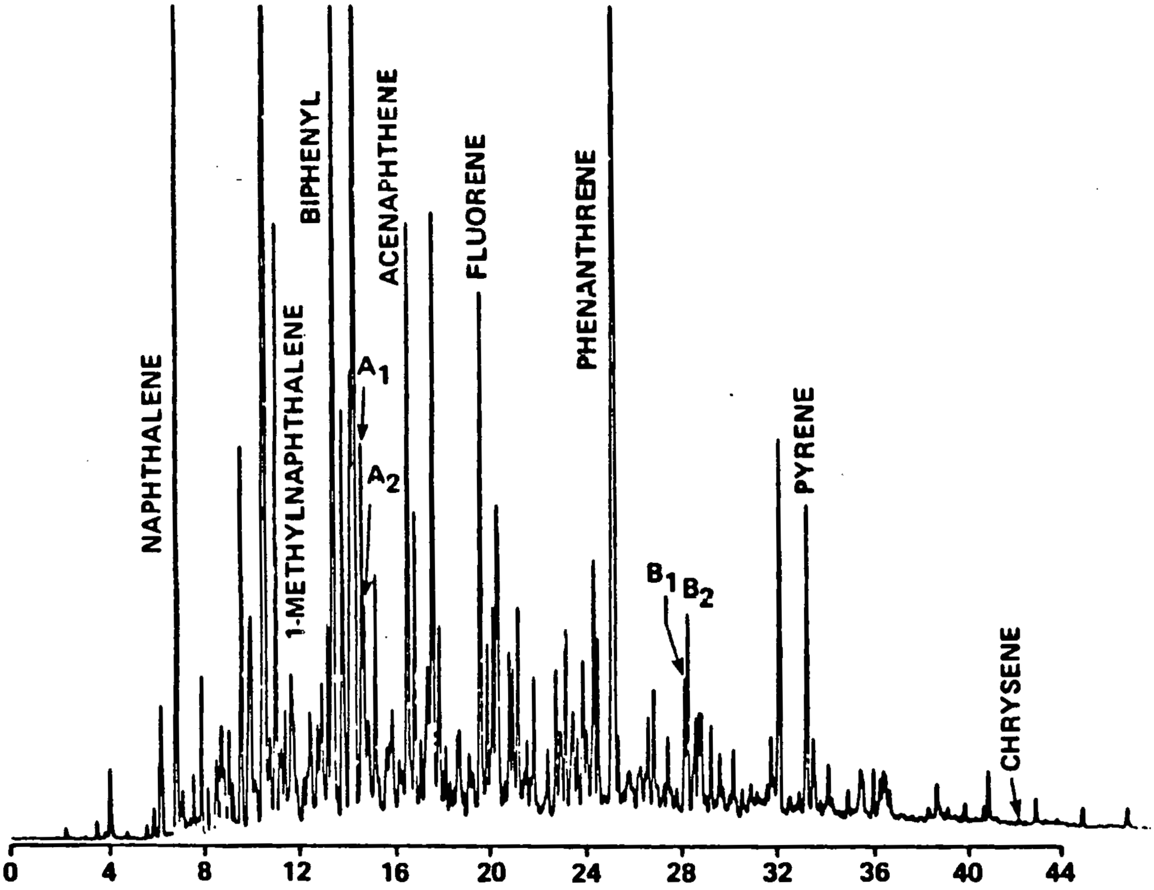
[a] 10% quinoline plus 90% methylnaphthalene.
[b] 100% methylnaphthalene.
[c] 100% quinolinediol.

# Table 20

## Statistical Comparison: Elemental Analysis of Wilsonville Recycle Solvent by Two Laboratories

|  | APCI |  |  |  | $\bar{X}$ | 2 SD | Huffman |  |  |  | $\bar{X}$ | 2 SD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  | CH1 |  | CH3 |  | (% abs) | (% abs) | CH2 |  | CH4 |  | (% abs) | (% abs) |
| C | 87.67 | 87.77 | 87.54 | 87.57 | 87.64 | 0.21 | 87.44 | 87.40 | 87.28 | 87.46 | 87.40 | 0.16 |
| H | 8.27 | 8.24 | 8.26 | 8.28 | 8.26 | 0.03 | 8.20 | 8.25 | 8.17 | 8.22 | 8.21 | 0.07 |
| O | 2.94 | 2.91 | 3.07 | 2.92 | 2.96 | 0.15 | 2.49 | 2.85 | 3.26 | 3.09 | 2.92 | 0.67 |
| N | 0.79 | 0.82 | 0.67 | 0.63 | 0.73 | 0.18 | 0.96 | 0.87 | 0.78 | 0.87 | 0.87 | 0.15 |
| S | 0.53 | 0.54 | 0.45 | 0.51 | 0.51 | 0.08 | 0.53 | 0.50 | 0.50 | 0.49 | 0.51 | 0.03 |
| Total | 100.20 | 100.28 | 99.99 | 99.91 | 100.10 | 0.34 | 99.62 | 99.87 | 99.99 | 100.13 | 99.90 | 0.43 |

Figure 1
GC-FID Chromatogram of SRC-I Process Solvent*



*450-850°F; 15-M X 0.30-MM i.d. GLASS SE-52 CAPILLARY COLUMN; TEMPERATURE PROGRAM 50-275°C AT 4°C/MIN; 2.2ML/MIN. HELIUM CARRIER GAS FLOW RATE.

Figure 2
Yield of Oils by Soxhlet Extraction of SRC Using Different Nonpolar Extracting Solvents

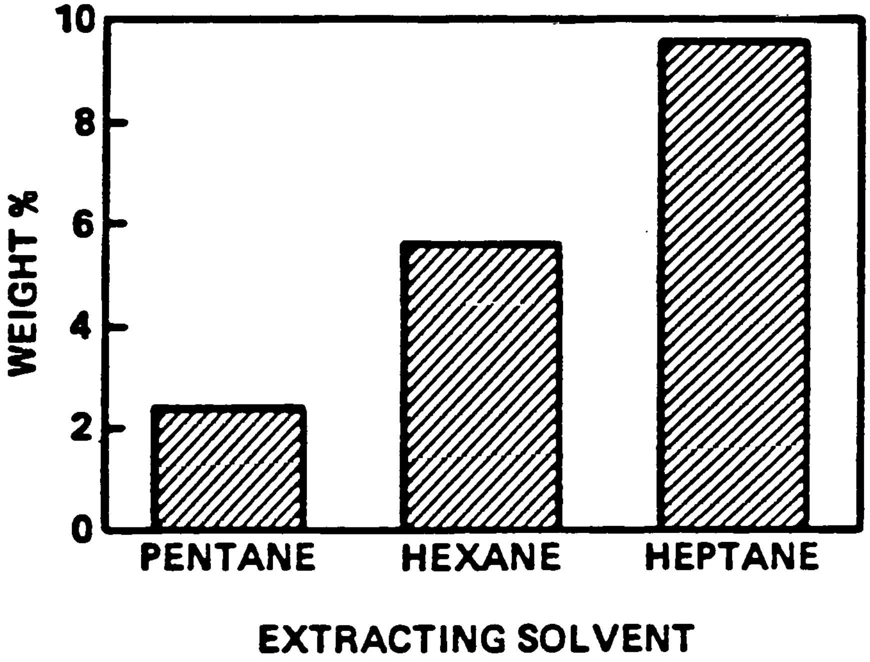


Figure 3
Boiling Point Distribution of Wilsonville Recycle Solvent

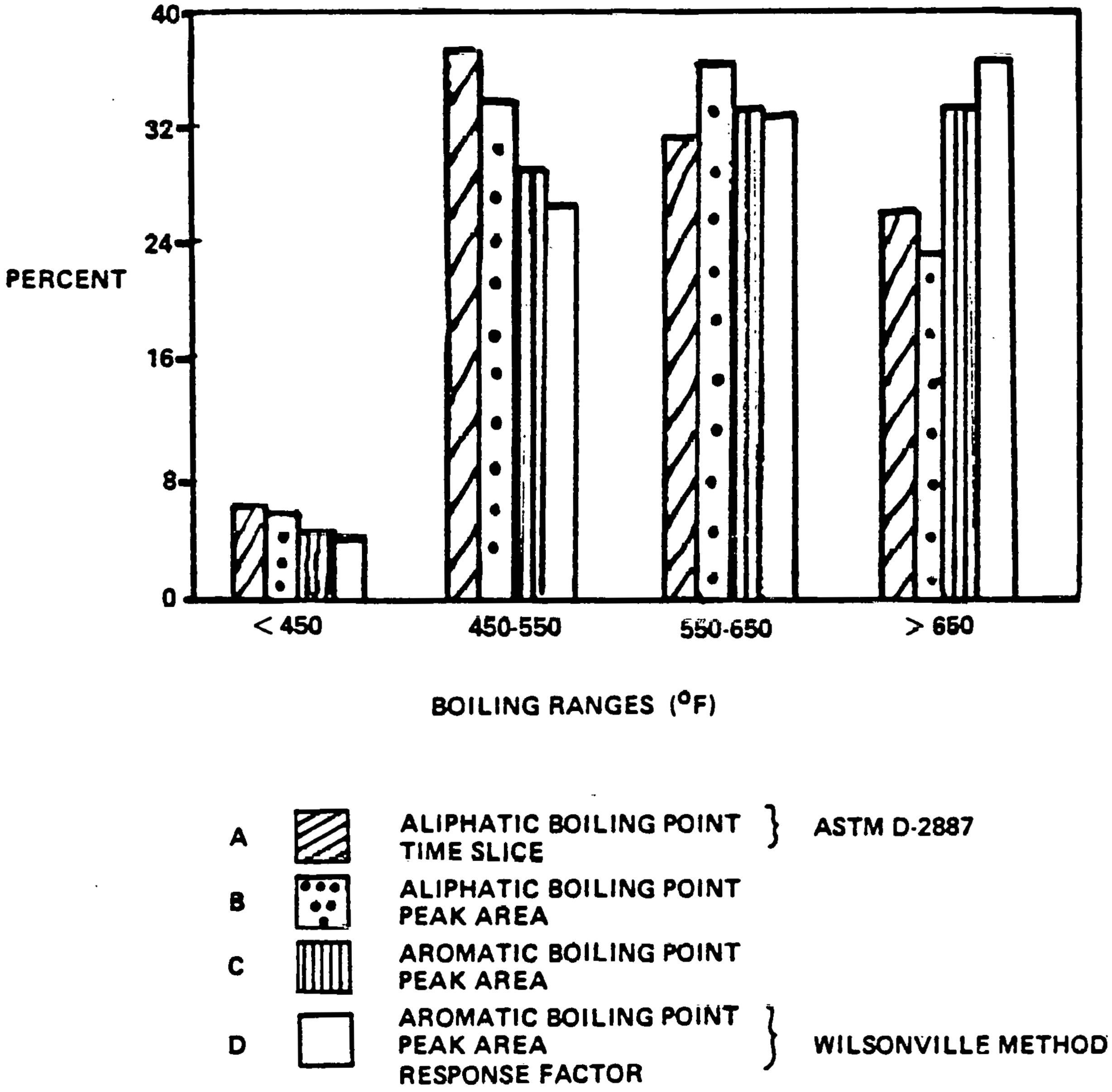


**Figure 4**
**Weight Percents of 100°F Distillate Cuts of Seven Wilsonville Process Stream Samples; Analysis at ICRC Compared to Wilsonville Analysis**

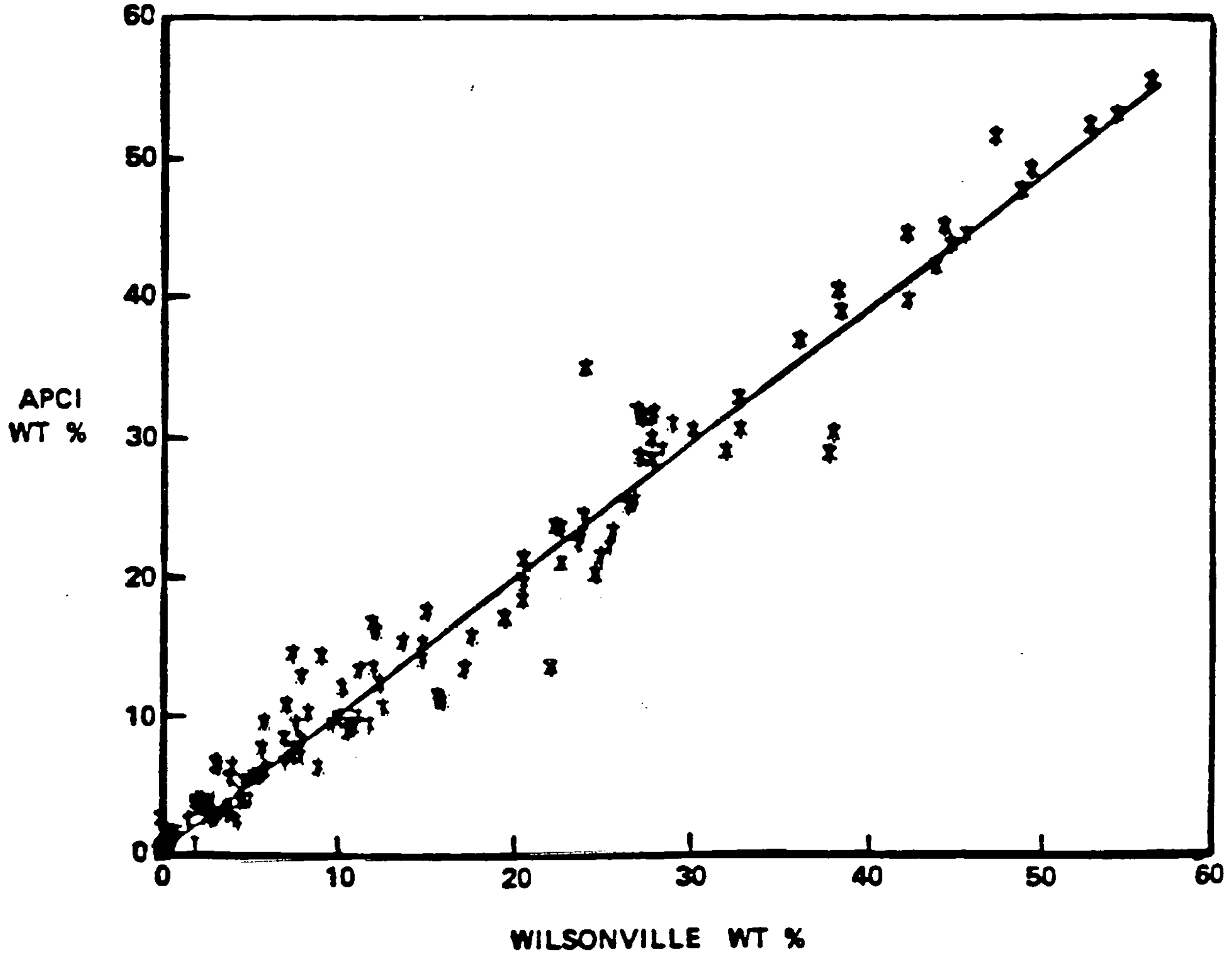


Figure 5
Weight Percent Yields of Distillation Fractions (°F)

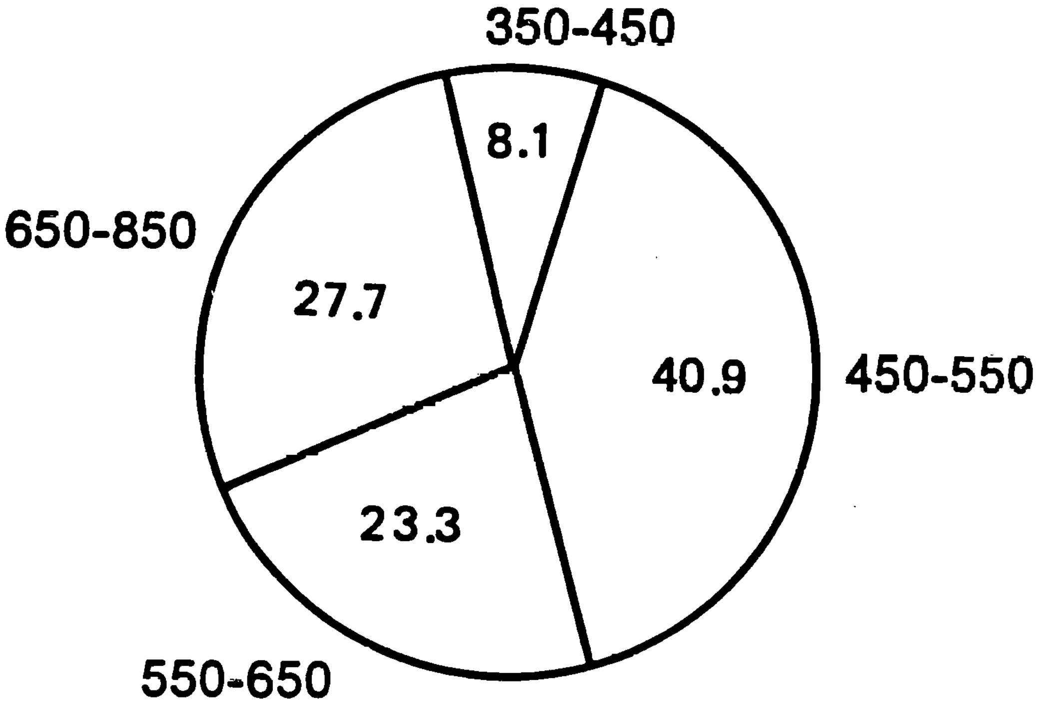


**Figure 6**
**Functional Group Separation Distillation Fractions**

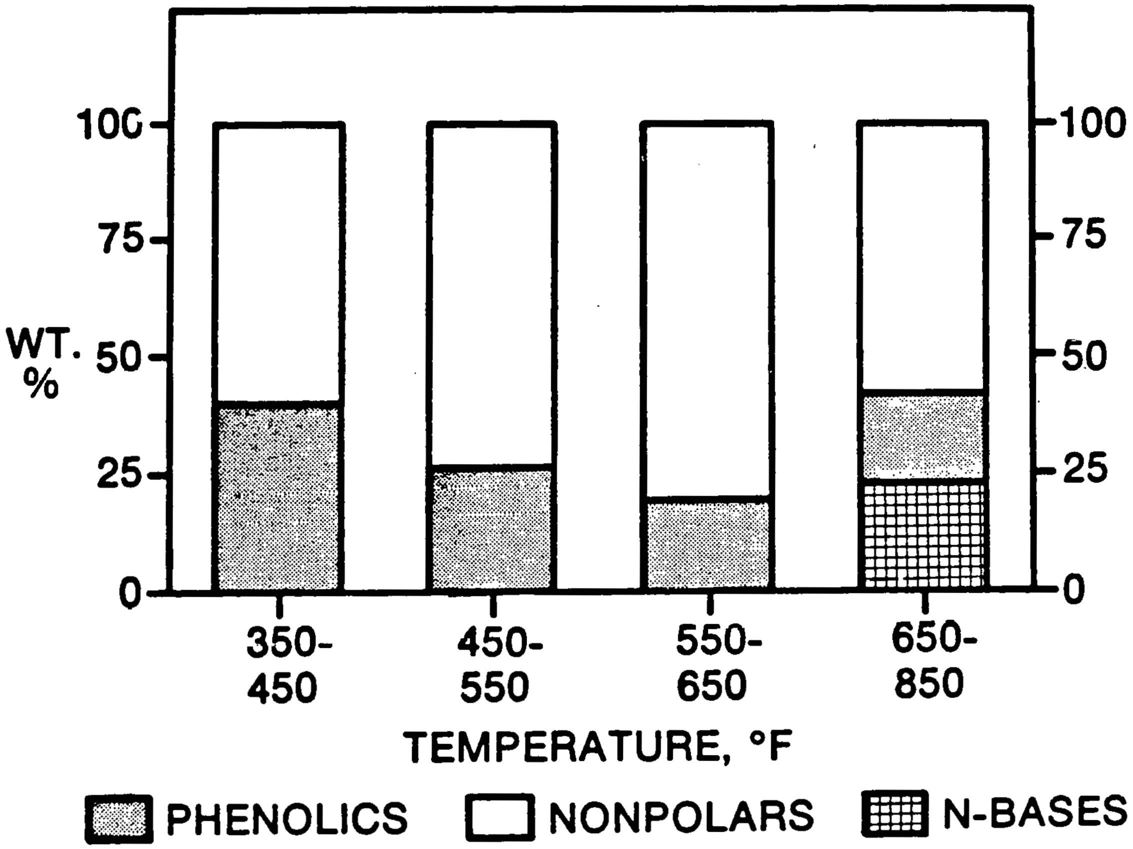


Figure 7

Ring Distribution Distillation Fractions

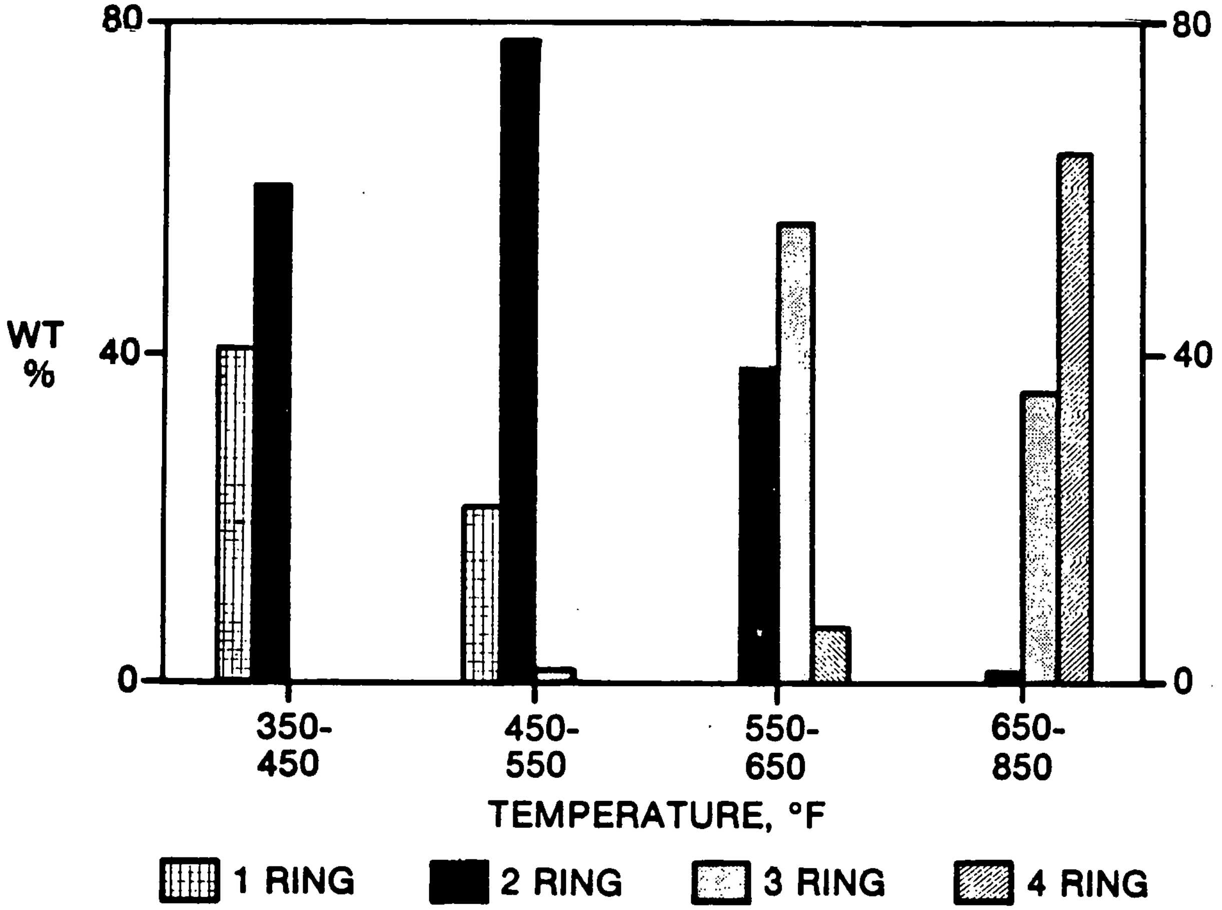


Figure 8
Total Gas (Hydrocarbon plus Heteroatom) as a Function of Time

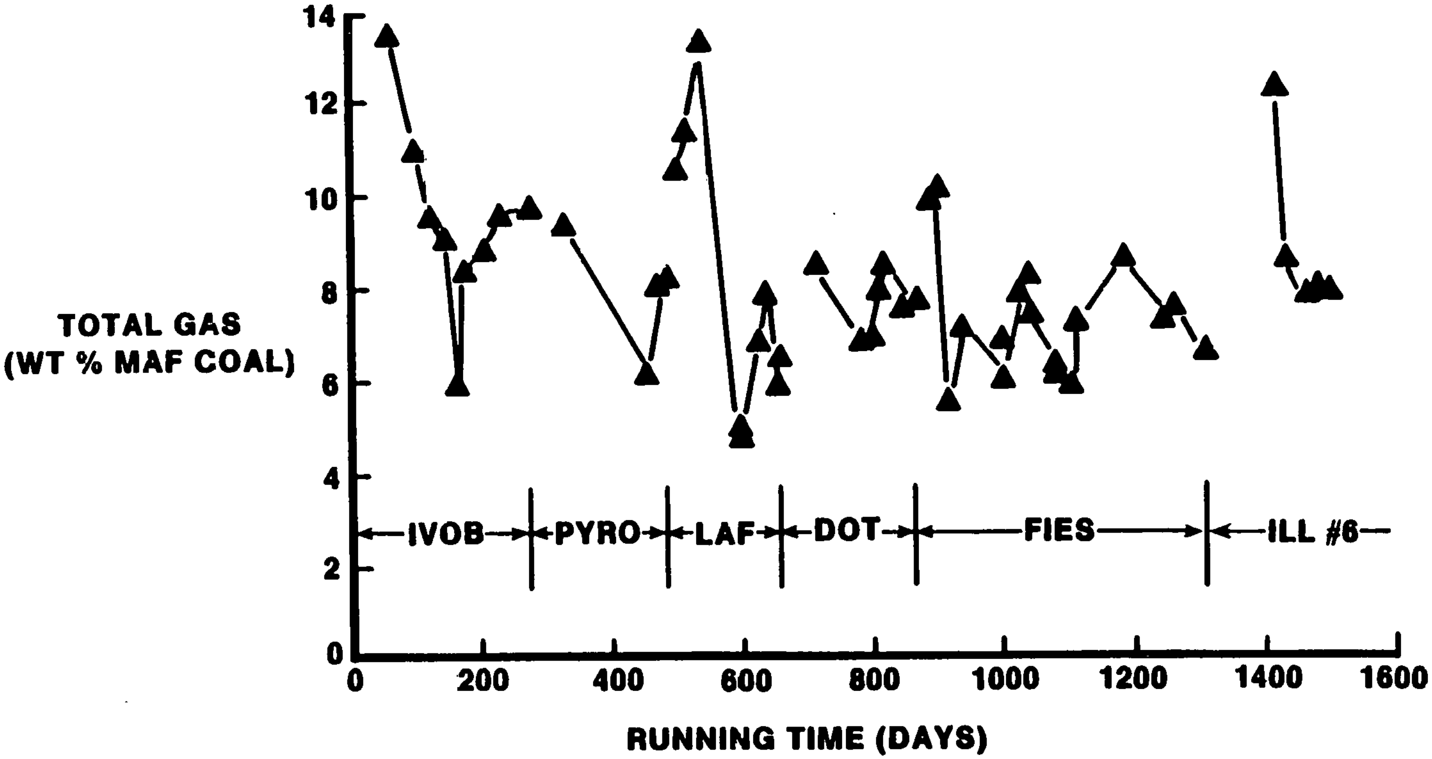


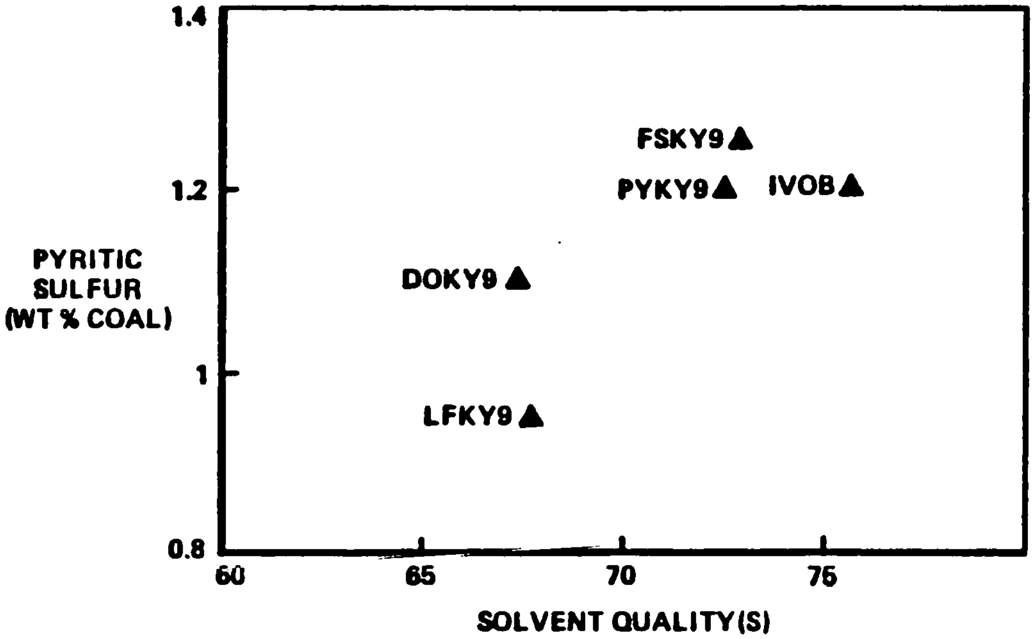
Figure 9
SRC-I Solvent Quality Test

| COAL<br>SOLVENT | STD PROCESS COAL-TYPE (1) | DAILY TOTE-BIN NO $H_2$ (1) | DAILY TOTE-BIN $H_2$ (2) |
|---|---|---|---|
| STD SOLVENT TET/MN | QUALITY ASSURANCE TEST | | |
| DAILY RECYCLE SOLVENT | | PRIMARY COAL DISSOLUTION | SECONDARY PRODUCT DISTRIBUTION |
| DAILY RECYCLE SOLVENT & ADDITIVE | | PRIMARY COAL DISSOLUTION | SECONDARY PRODUCT DISTRIBUTION |

(1) MICROAUTOCLAVE S/C (8/1); 10 MINUTES AT 750°F
DETERMINE: RESIDUE (THR) – PREASPHALTENES – BENZENE SOLUBLES

(2) TEST CONDITIONS TO BE DETERMINED

Figure 10
Coal Pyrite vs. Solvent Quality by Coal Type



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