

Chemical Characteristics of Tars Produced in a Novel
Low Severity, Entrained Flow Reactor

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Introduction and Approach

The devolatilization of a high volatile bituminous coal follows the sequence indicated in Figure 1. The formation and evolution of heavy molecular weight hydrocarbons, tars, account for more than half the total mass loss of such coals (1,2,3,4). As observed in a wide range of heating conditions, the tars dominate the initial mass loss of bituminous particles. In addition, gas phase, "secondary" reactions of tars can account for major fractions of the light gas yields, depending on the heating conditions. The distribution of the light gases and chemical characteristics of the collected tars are dependent on the both the transient particle temperature and ambient gas temperature (5,6).

A number of investigators have reported that a range of coal types follows the same phenomenological sequence, although the tar yields vary significantly with coal rank characteristics (4,7,8). Moreover, chemical structural characteristics of "primary" tars are reported "similar" to those present in the parent coal, that is, the tar "monomers" reflect the coal "polymer" (3,9,10). "Primary" tars are those collected in conditions in which "secondary" reactions have been minimized. Some of these investigators have noted that, within the limits of experimental resolution, the chemical kinetic parameters that describe tar evolution from a wide range of coals do not vary with coal type (3,9). From this perspective, the phenomenological sequence, monomer-polymer relationship between tar and parent coal, and chemical kinetic parameters describing tar formation and evolution are all invariants in coal devolatilization.

On the other hand, some investigators note the phenomenological sequence of disperse phase devolatilization is the only commonality among coals of varying rank characteristics. Tar yields and chemical characteristics, relative to other coal tars as well as to the parent coal, vary significantly with coal type. In addition, secondary reaction behavior varies according to chemical characteristics of the primary tars (7,11). Implicit in these structural observations with respect to primary tars is a questioning of the invariance of devolatilization/pyrolysis kinetics with coal type. Since all primary tars are not equally "similar" to the parent coal and the distribution of functional groups varies so extensively with coal type, it becomes difficult to understand the extent to which the same tar evolution and light gas formation chemical kinetic parameters could be used for a wide range of coal types.

Other investigators have noted the appreciable role transport parameters can exert in determining devolatilization phenomena. The formation of a glass-like "melt" as an intermediate phase during devolatilization of a

bituminous coal (12,13,14) and the observed changes in molecular weight characteristics of heavy hydrocarbons with ambient pressure (15,16), have led some investigators into a detailed examination of intraphase and interphase mass transport phenomena contributions in mass loss kinetics. Ignoring the details of product distributions and chemical characteristics of tars with changes in pyrolysis conditions, still other investigators have indicated that weight loss kinetics is determined primarily by heat transfer considerations (17,18).

As apparent from the diverse perspectives, a conceptual understanding of disperse phase coal devolatilization remains elusive and, consequently, comprehensive rate models remain difficult to extrapolate to a wide range of conditions. Among the considerable experimental difficulties are: rapidly quenching a heterogeneous process whose overall mass loss rate appears to adjust itself to heating conditions; isolating char particles, tar aerosols and light gas species that are coupled in product streams; minimizing gas phase reactions of thermally labile heavy molecular weight organic compounds; subsequent analysis of large, fragile organic species which have considerably different functional forms, depending on heating conditions and parent coal characteristics.

Batch reactors and single particle systems are often criticized for generating too small a tar sample in conditions unrepresentative of rapid heating. Continuous reactor systems, such as entrained flow reactors, provide acceptable heating conditions but due to residence time, the hot entrainment gases, and the large entrainment flow/sample mass, make it difficult to either time resolve weight loss kinetics or isolate primary tars for subsequent analyses.

This communication reports the utilization of a novel entrained flow reactor and product isolation system. In contrast to the operation of most entrained flow reactors, no attempt is made to match the entrainment gas and wall temperatures. Entrainment gas temperatures are purposely kept below wall temperatures to minimize extra-particle gas phase reactions of tars. To obtain char-free tar samples, an aerosol phase separation system is employed to isolate a significant fraction of entrained tar species from the char mass. Both the char particle and aerosol arms of the product separation system contain serially staged particulate separation systems followed by porous metal final filters that collect "pure" tar species.

Experimental

Reactor and Product Separation System

Figures 2 - 4 display the essential components of the entrained flow reactor, product separation and gas analysis systems. Figures 5 - 7 display the total power density, radiative power density and gas temperature profiles as a function of reactor position. The flux rate profiles are measured by specially designed, calibrated probes. Incident, center-line radiative flux rates range from several watts/sq. cm. at wall temperatures of 750 C to approximately 25 watts/sq. cm. at wall temperatures of 1270 C. Gas temperature

profiles vary as shown. Gas temperature profiles were obtained by making a series of measurements with a set of decreasing thermocouple bead sizes with the asymptotic temperatures approached defined as the "true" local gas temperature. In general, the gas temperatures measured in this manner are consistent with those estimated from the magnitude of the convective heat transfer determined from the power density measurements.

The reactor creates a heat transfer field in which entrained particles are heated by radiation to the local, axial, entrained gas temperature within the reactor (Figures 8 , 9). The radiation flux induces an inverse diameter dependent heating rate on particles in an attempt to drive the particles to equilibrium with the radiating walls. The carrier gas imposes an inverse diameter squared component on the particle heating rate, which in effect insures small particle temperatures are not very different from the local gas temperature, assuming the devolatilization process is weakly endothermic or thermally neutral. Estimated particle heating rates are of the order of 5000 7000 C/sec in these conditions. Such heating rates are greater than that reproducibly obtainable in a heated grid apparatus operated in the same laboratory, but less than that expected in conventional entrained flow reactors wherein heating rates approaching 100,000 C/sec are indicated with estimated transient flux rates at the particle surface of nearly 100 watts/sq. cm. Operating an entrained flow reactor in this manner minimizes gas phase pyrolysis reactions of the initial tar species and allows collection of large quantities of tar species.

Normally, 60 - 75% of the total reactor flow is drawn through the aerosol separation (impactor train) system, that is F of Figures 3 and 4 is of the order 0.25. The temperature and path length of the cooled aerosol transfer line are varied according to the nature of the tars being collected, which, in turn, varies with the parent coal characteristics and reactor heating conditions. The aerosol phase separator is designed to "pull off" all particles or aerosols that are less than 2 microns. A qualitative check of the performance by scanning electron microscope examination of the deposits indicated that designed behavior is followed, provided proper flow rates are maintained throughout an experiment. Inertia carries larger particles into the char separation (cyclone train) system. Both separation trains contain porous metal filters as final stages. Performance of the system is monitored by measuring the flow through the separation arms, pressure drop across the filter housings and determining the tar mass deposited on each of the filter systems. Tar mass ratios are expected to be of the ratio of the average mass flow ratio through the two separation systems during an experiment. This is observed to be the case.

Sample Selection

The feed system utilized is capable of sustained delivery of optically thin streams of particles at constant mass delivery rate, provided the feeder is loaded with a narrow particle size range initially. Particle sizes as low as 10 microns and as large as 300 micron have been utilized. Since the feeder operates on aerodynamic principles, aerodynamically separated samples were employed (See acknowledgements). The location of the parent coal sample set on

a pseudo-coalification band plot is shown in Figure 10.

Sample Measurements

Mass loss is determined by ash tracer techniques. It is observed that experiment specific determinations of the parent coal ash need to be determined. This is accomplished by operating the system in cold flow conditions before and after a particular hot wall devolatilization test and determining the ash content of the particles collected in the first stage cyclone. This value becomes the initial ash value. It is observed that lower density particles are fed into the reactor initially despite the rather extensive efforts to match the aerodynamic characteristics of the feeder with those of the size separated samples provided. Consequently, sustained operation over a sequence of devolatilization conditions leads to significant variations in the average density, mineral content, of the feed, which, in turn, leads to inconsistent determinations in particle mass loss via ash tracer.

Elemental characteristics of evolved tars and chars are determined by use of a Perkin-Elmer 240 instrument. IR absorbance characteristics are determined by an alkali-halide technique using a Fourier Transform - Infrared Spectrometer (FT-IR). The FT-IR is also used to determine the composition of the pyrolysis gases. The streams from the two separation systems are recombined and continuously passed through a multi-pass cell. The 43.5 meter path length is needed to measure the low levels of IR active light gases generated. Concentrations are low because the initial stages of devolatilization, the focus of this investigation, generate small amounts of such gases which are entrained in a relatively large volume of carrier gas.

Results

HVA Bituminous Coal

For the PSOC 1451D coal, an Appalachian high volatile bituminous coal, the ash tracer volatile yields for two particle sizes is shown in Figure 11. As indicated, despite the factor of three difference in particle size, the reactor temperature sensitivity of the mass loss is very similar between the size cuts. Temperature calculations using the measured reactor characteristics as heat transfer conditions indicate this should indeed be the case (See above). Figure 12 shows the relative gas yields, normalized with respect to maximum yields of each gas and plotted with respect to the peak reactor gas temperature, since gas phase reactions of tars are thought to account for substantial fractions of "coal" pyrolysis gases. It is noted that significant increases in acetylene and hydrogen cyanide gases are not observed until peak gas temperatures of 700 C are achieved. At lower gas temperatures, light gas yields are dominated by CH₄, C₂H₄, CO and H₂O, but these gases account for only 10 - 15% of the total 0.2 - 0.25 particle mass fraction loss observed for these reactor conditions. For this coal, tar yields dominate the mass loss at these and lower gas-particle temperatures. Appreciable tar yields, 0.10 - 0.15 mass fraction, are observed at gas temperatures of 350C (wall temperatures of 790C).

Examination of the elemental composition of the tars indicates that the tar evolution process resembles a distillation process from the point of view of hydrogen concentration versus characteristic temperature, gas or reactor wall (Table I). As implied by the insensitivity of the total mass loss with particle size and as indicated by the temperature calculations, the elemental composition of the tars evolved from the two different particle sizes and for a particular stage of the reaction process should be similar. As indicated in Table I the elemental composition of the the 20 -30 micron tars and the 63 - 75 micron tars are quite similar in elemental composition. A distillation-like tar evolution process would also imply changing functionality and relative molecular weight characteristics of the samples with characteristic temperature. Figure 13 displays the relative -CH₂- concentration of the tars for increasing reactor temperature and Table II indicates relative molecular weight moments as a function of reactor temperature.

In summary, the results indicate the hydrogen level, in particular the polymethylene concentration hydrogen level, of the collected tars systematically decreases with peak reactor temperature as the total tar yield increases. As the hydrogen level gradually decreases the relative molecular weight moments of the evolved tars increase to the point where the tar yield is believed to be maximized. Lower temperature tars contain less total oxygen and sulfur mass fractions than the total tar mass evolved to the point of maximum tar yield. Absolute tar yields are difficult to determine using an entrained flow reactor but investigation of the tar evolution process on a heated grid apparatus indicate total tar yields are 0.30 - 0.35 of the parent coal mass on an ash-free basis for this coal.

Coal Rank Effects

It is desirable to determine the change in the nature of the tars produced with changes in the chemical characteristics of the parent coal, while keeping the heating conditions constant. The heating conditions that maximized the tar yields for the high volatile bituminous coal (930C wall, 500 - 600 C gas temperature) while minimizing gas phase secondary reactions were selected, although it is recognized that each coal type may require a unique set of heating conditions to maximize its tar yield.]

Figures 14 and 15 compare the hydrogen and sulfur + oxygen levels of the tar species to those observed in the parent coals. As the rank characteristics of the parent coal increase the elemental composition of the tars become more like the parent coal. The similarity ratios asymptote toward unity. Conversely, the lower the rank of coal the more unlike in elemental composition are the primary tars. Figure 16 indicates the %H of the low rank coal tars can be as great as 9% in these conditions.

The most striking difference in the IR absorbance spectra of the low rank coal tars relative to the higher ranks resides in the polymethylene absorbance region (2920 cm⁻¹). The low rank coal tars generally display much greater absorbance levels in this region per unit mass of sample than the high rank coal tars. Assuming the absorption coefficients of this band do not vary significantly with coal rank, such behavior implies increasing concentrations of -CH₂- levels with decreasing coal rank. The -CH₂- absorbance band

correlates with the % H level in the tars (Figures 16,17,18).

The difference in structural characteristics of the low temperature tars evolved from the lignite and subbituminous coals, relative to the bituminous coal tars, is again emphasized by comparing the molecular weight moments obtained by gel permeation chromatography. The data in Figure 19 indicates the tars evolved from the low rank coals at a particular temperature have greater molecular weight moments than the corresponding high rank coal tars. In the most elementary sense, the polystyrene calibrated gpc technique indicates only that aliphatic, hydrogen-rich low rank coal tars are structurally "larger" than the more aromatic bituminous coal tars with respect to a length to weight ratio parameter. These results likely reflect variation in conformational aspects of the tars as a function of rank rather than molecular weight or polarity differences. The long chain aliphatic tars characteristic of the low rank coals are not efficiently retained by the gel permeation column and, consequently, elute in the short retention times characteristic of heavy polystyrene standards. The molecular "weight" moments appear to be more characteristic of relative molecular geometry than actual mass size when comparing tars from a range of coal ranks. The general observations with respect to the variations in low temperature coal tars with rank characteristics of the primary coal are indicated in Figure 20, the lower the rank of parent coal the more unlike the parent coal are the low temperature, primary tars. The dissimilarity appears primarily related to the aliphatic to aromatic hydrogen distribution and the oxygen-sulfur heteroatom content of the tars relative to the parent coal. Significant polymethylene concentrations variations are observed with variation in coal type.

Although the nature of tars varies significantly with coal type, the particle devolatilization and gas phase reaction sequence did not. Table III displays the composition of tars from a subbituminous coal (PSOC 1520D) at various reactor temperatures. As in the high volatile bituminous coal, the hydrogen rich, more aliphatic, lighter molecular weight species vaporize before the heavier, more aromatic species during the evolution to the point of maximum tar yield. At gas temperatures of 700 C or above, gas phase reactions of pyrolysis of the polymethylene rich tars quickly leads to ring formation in the tar species. C₂H₄ and CO are major light gas products of these reactions and, at still higher temperatures, C₂H₂, CO and HCN. In short, the same light gas formation temperature pattern as indicated in Figure 12 for a HVA bituminous coal is followed by the lower rank coals. However, the absolute quantities vary significantly with the nature of the low temperature tars.

Summary and Conclusions

The entrained flow reactor investigation indicates that the phenomenology of coal devolatilization and pyrolysis is similar for a wide range of coal ranks. The phenomenology is summarized in Figure 1. Heavy hydrocarbons are "formed" within (ATP = Attached Tar Precursors) the coal particle at relatively low temperatures (300 - 450 C). Further heating results in the extra-particle evolution of these heavy hydrocarbons (tars) and the onset of light gas production (450 - 700 C). The low temperature light gases consist mainly of CH₄ and higher alkanes, CO, CO₂, H₂O and some C₂H₄. The absolute quantities of these species vary with coal type as do the absolute yields of

"primary" tars. Gas and particle temperatures greater than 700 C preferentially produce C₂H₂, HCN, CO and C₂H₄.

Although the sequence of tar evolution is phenomenologically similar, the structural characteristics of tars evolved from a range of coal types indicates quite different processes are occurring chemically. The lower the rank of coal, the more unlike the parent coal are the low temperature tars. Low temperature lignite tars appear to consist mainly of long chain aliphatic species with significant levels of associated carboxylic and carbonyl groups. Low temperature tars from bituminous coals have structural characteristics more reflective of, but never identical to, the parent coal.

Gas phase reactions of low temperature, primary tars are rapid at gas temperatures of 700 C and above, common operating temperatures of conventional entrained flow reactors. Such reactions quickly lead to the ring formation reactions from polymethylene-rich low temperature tars coincidental with the evolution of the heavier tar species from the devolatilizing particle itself. Tars collected in these conditions, particularly tars collected in separation systems without extensive phase separation, will appear to be more like the parent coal than the primary tars originally evolved and collected in the above reactor system or other systems that minimize secondary tar reactions. The phenomenological sequence of heavy hydrocarbon formation, tar evolution and gas phase reactions appear similar for a wide range of coals, but the relationship between the chemical structures of primary tars and parent coal varies widely with coal type.

The question of invariance in kinetic parameters among a range of coal types and whether chemical or transport phenomena dominate the evolution processes requires a multi-reactor approach in which the phases of tar formation and evolution can be deconvoluted. This will be the subject of future communications.

Acknowledgements

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TABLE I
 ELEMENTAL COMPOSITION OF TARS - TEMPERATURE EFFECTS
 HVA BITUMINOUS COAL(PSOC 1451D)

REACTOR WALL(C)	MAX. GAS(C)	-----20 - 30 MICRONS-----				
		%C	%H	%N	%(S+O)	H/C
790	350	84.05	6.07	1.64	8.24	0.87
840	400	84.07	5.94	1.67	8.32	0.85
930	510	84.37 84.46	5.86 5.93	1.68 1.76	8.09 7.83	0.83 0.84
1020	665	84.62	5.55	1.69	8.14	0.78
1100	790	85.22	5.40	1.73	7.65	0.76
1190	890	85.55	5.27	1.74	7.44	0.74
1270	1000	86.00	5.08	1.73	7.19	0.71
PARENT COAL.....		82.42 81.07	5.35 5.24	1.57 1.62	10.65 12.04	0.79 0.78
		-----63 - 75 MICRONS-----				
790	350	83.97	6.22	1.64	8.14	0.89
930	510	84.47 84.16	5.83 5.79	1.72 1.74	7.98 8.29	0.83 0.83
1270	1000	85.50	5.29	1.76	7.43	0.74
PARENT COAL.....		82.55 81.86	5.53 5.43	1.59 1.66	10.32 11.02	0.80 0.80

TABLE II
 Relative Molecular Weight of Characteristic of Tars
 Reactor Temperature Effects *
 PSOC 1451D, 20-30 Microns

Reactor Wall Temperature (C)	Position (cm)	Residence Time (msec)	Mn	Mw
750	36	620	438	641
840	36	580	452	665
930	36	520	481	719
1020	36	470	491	743
1100	36	420	518	801
1190	36	385	509	803
1270	36	360	506	807

* Argon Carrier Gas

TABLE III
 ELEMENTAL COMPOSITION OF TARS - TEMPERATURE EFFECTS
 SUBBITUMINOUS COAL (PSOC 1520D)

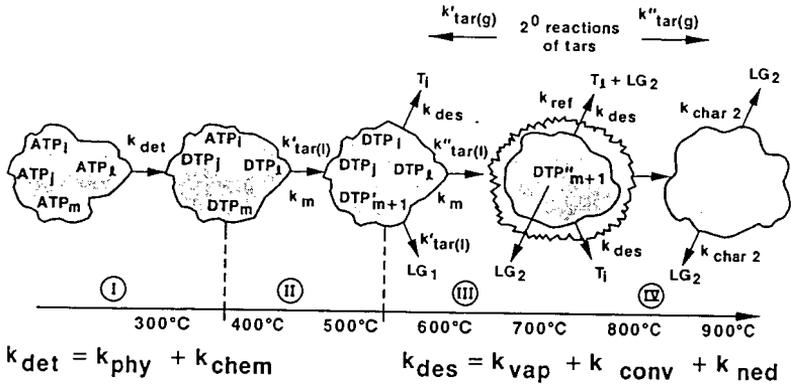
790	350	78.21	8.43	0.63	12.73	1.29
930	510	78.15	7.83	0.79	13.23	1.20
		77.78	7.63	0.81	13.77	1.17
1270	1000	78.97	6.72	0.98	13.32	1.02
PARENT COAL.....		63.77	4.54	0.83	30.84	0.86

* TARS - SPECIES COLLECTED ON FINAL FILTER OF IMPACTOR TRAIN

ARGON CARRIER GAS
 PARTICLE RESIDENCE TIME: 600 msec

Fig. 1

COAL DEVOLATILIZATION / PYROLYSIS



RA24197X.026

Fig. 2

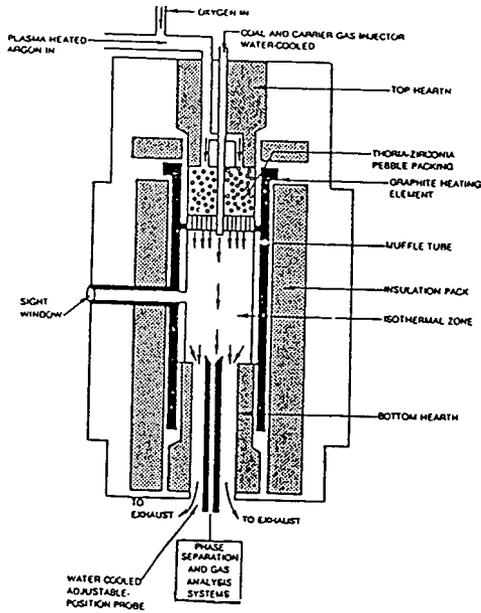
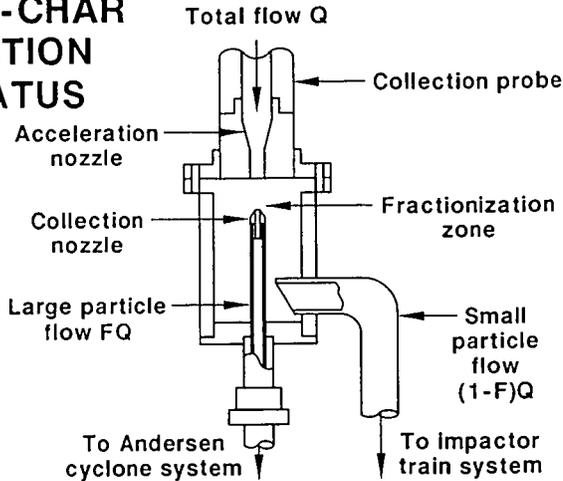


Fig. 3

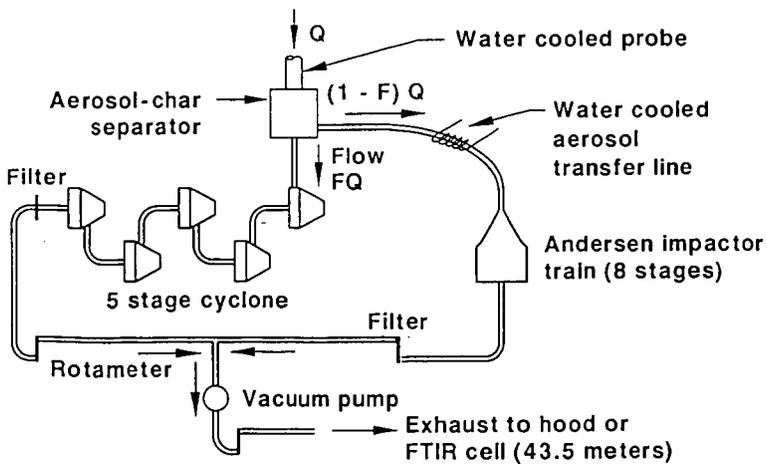
AEROSOL-CHAR SEPARATION APPARATUS



RA21912.M1

Fig. 4

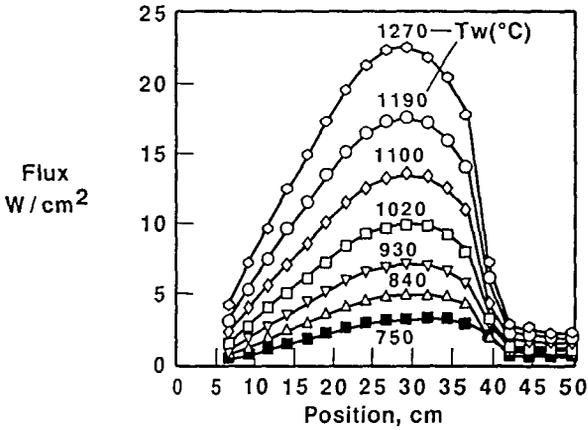
UTRC-EFR SAMPLE COLLECTION SYSTEM



RA21912.002

Fig. 5

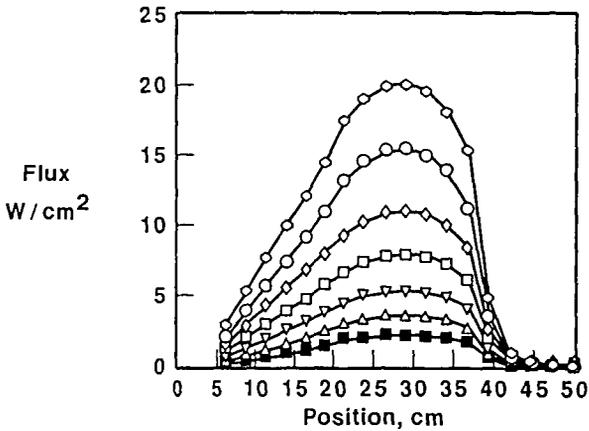
UTRC-EFR REACTOR TOTAL FLUX PROFILES



RA2416TX.001

Fig. 6

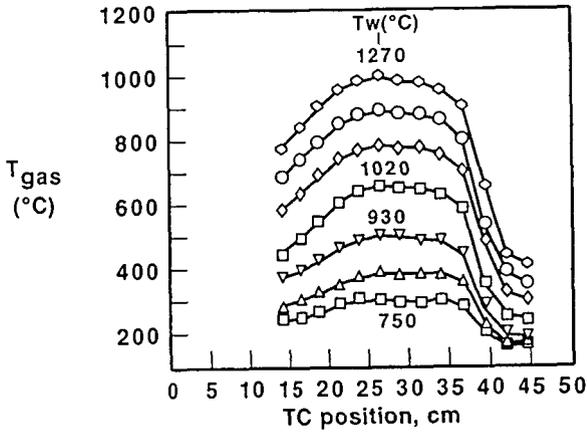
UTRC-EFR REACTOR RADIATIVE FLUX PROFILES



RA2416TX.002

Fig. 7

UTRC-EFR COOLED PROBE 0.1016 CM BEAD DIA



RA24187X.003

Fig. 8

UTRC ENTRAINED FLOW REACTOR - PARTICLE TEMPERATURE 840 C WALL TEMPERATURE

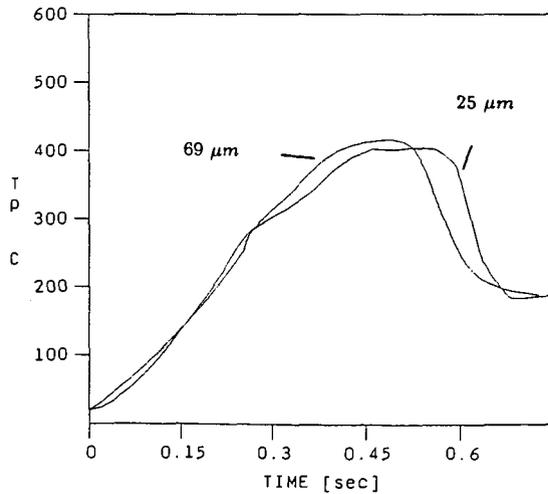


Fig. 9

UTRC ENTRAINED FLOW REACTOR - PARTICLE TEMPERATURE
930 C WALL TEMPERATURE

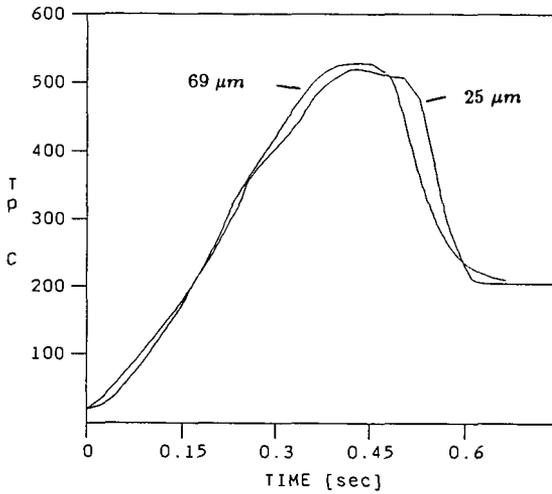


Fig. 10

**H/C vs. (S+O)/C OF
PSOC XXXXD COALS**
20-30 microns, coals fed to UTRC
EFR-cold flow, C₀ capture

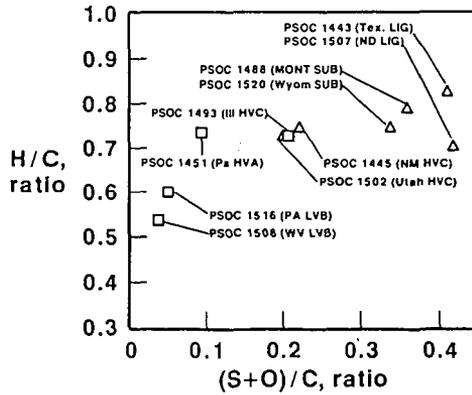
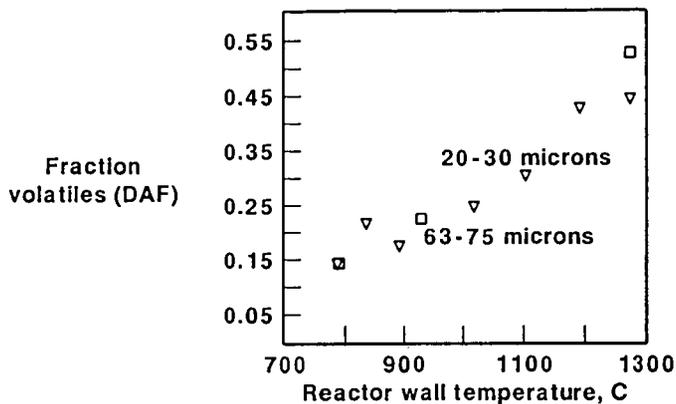


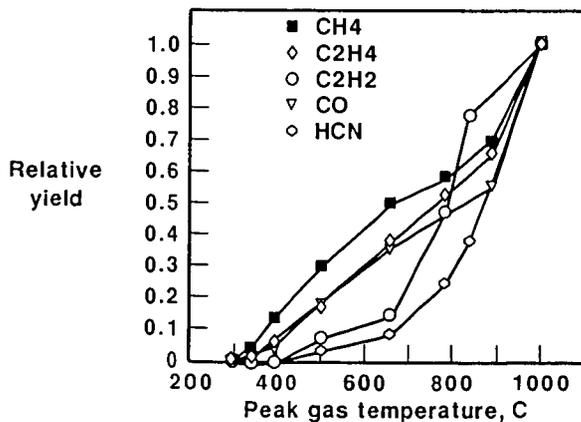
Fig. 11



RA2416TX.010

Fig. 12

LIGHT GAS YIELDS vs. PEAK GAS TEMP. (C) PSOC 145 1D, 20-30 MICRONS, 600 MSEC

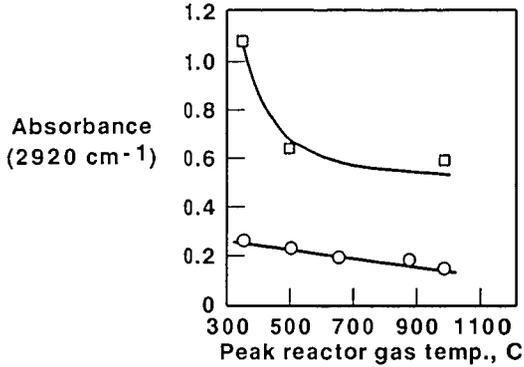


RA2416TX.009

Fig. 13

-CH₂- STRETCH ABSORBANCE (2920 CM⁻¹) vs. GAS TEMPERATURE

Triangle = PSOC 1451D, square = PSOC 1520D, 20-30 microns

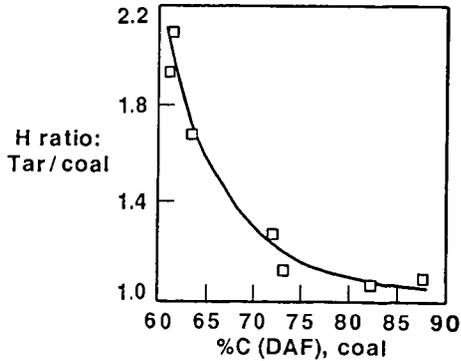


RA2418TX.009

Fig. 14

%H(TAR) / %H(COAL) vs. COAL RANK

(PSOC XXXXD coals) 20-30 micron, particles,
peak reactor gas temperature = 510C

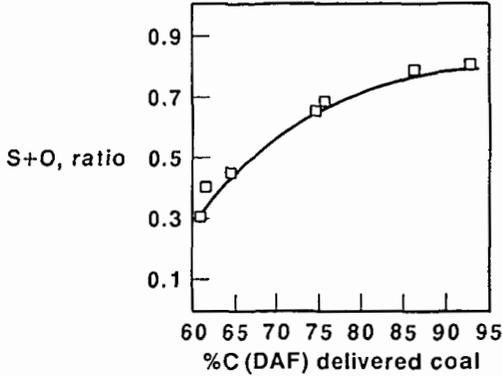


RA2418TX.012

Fig. 15

% (S+O) - TAR / % (S+O) - COAL vs. COAL RANK

(PSOC XXXXD coals) 20-30 micron particles,
reactor peak gas temperature = 510C



RA2415TK.012

Fig. 16

% HYDROGEN IN TAR vs. COAL RANK

(PSOC XXXXD coals) 20-30 micron, particles,
peak gas temperature = 510C

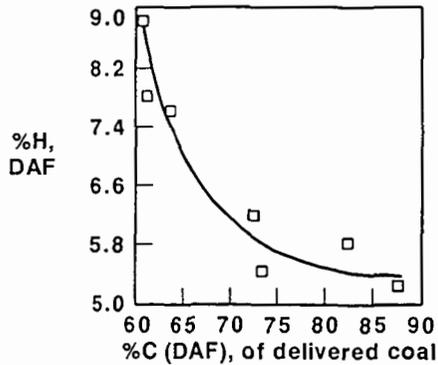
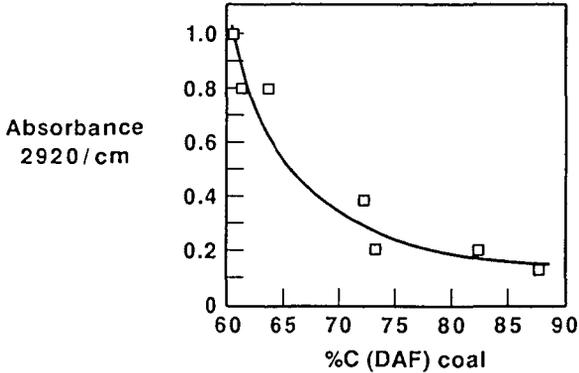


Fig. 17

2920 CM-1 ABSORBANCE OF TARS vs. COAL RANK

(PSOC XXXXD coals) 20-30 micron particles,
peak gas temperature = 510C

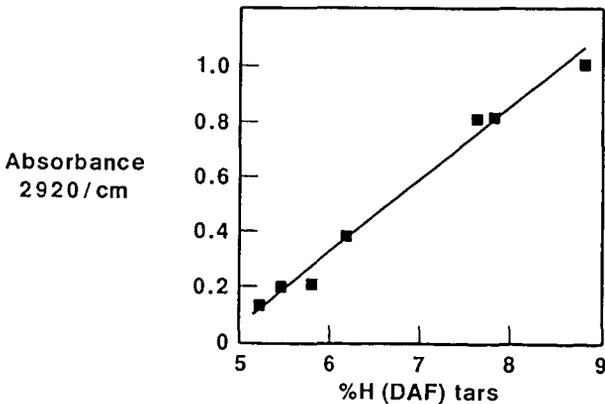


RA2415TX.016

Fig. 18

ABSORBANCE (2920 / CM) vs. % H (DAF) IN TARS

(PSOC XXXXD coals) 20-30 micron particles, peak gas temp. = 510C

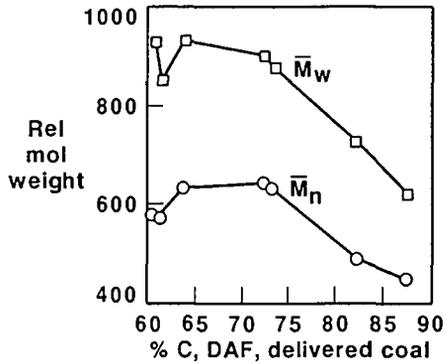


RA2415TX.016

Fig. 19

RELATIVE NUMBER AND MASS AVERAGE MW OF TARS AND COAL TYPE

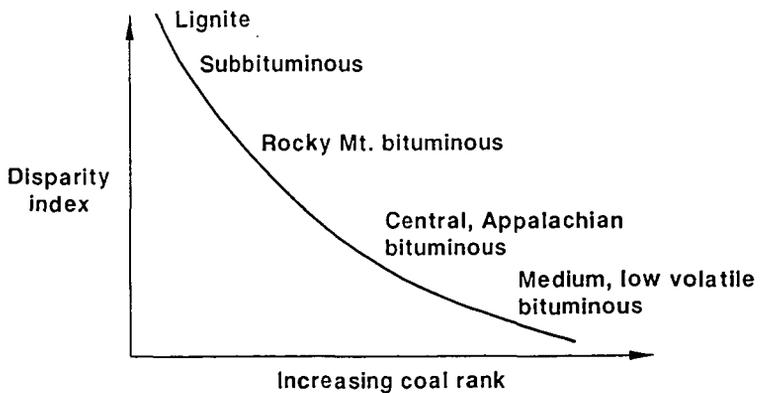
PSOC XXXX D coals, 20-30 microns, 510C peak gas temp.



RA2415TX.008

Fig. 20

PRIMARY TAR ← PARENT COAL: STRUCTURAL COMPARISONS



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