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DEVELOPMENT OF ANALYTICAL PROCEDURES FOR COPROCESSING

QUARTERLY TECHNICAL PROGRESS REPORT FOR THE PERIOD APRIL 1 THROUGH JUNE 30, 1991

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DEVELOPMENT OF ANALYTICAL PROCEDURES FOR COPROCESSING

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DEVELOPMENT OF ANALYTICAL PROCEDURES FOR COPROCESSING

ABSTRACT

This is the eleventh and final quarterly report under DOE Contract No. DE-AC22-88PC88810, Development of Analytical Procedures for Coprocessing. The overall objective of the contract is to improve our understanding of the fundamental chemistry of coprocessing. A primary objective is to evaluate methods to distinguish between compound classes originating from coal versus those originating from petroleum resid while a corollary objective is to provide detailed knowledge on the composition of coprocessing products.

This and the prior quarterly report summarize work conducted in support of the latter objective in which process development unit samples produced by HRI, Inc. were subjected to detailed analysis. Coprocessing resid samples selected for detailed analysis were made under constant conditions except for variations in coal concentration or in the coal (New Mexico subbituminous or Texas lignite). Separation of the resids into acid, base, saturate, and neutral-aromatic subfractions, separation of the neutral-aromatics by ring number and high temperature gas chromatography were discussed in the previous quarterly. This report includes results of nonaqueous titrations, elemental analyses and infrared spectroscopy. The hydrocarbon skeletons of saturated hydrocarbons in the coprocessing resids appear to be fundamentally different than those of aromatic species. Neutral-aromatic fractions contain minor levels of sulfur compounds, an unknown proportion of ether or other oxygen-containing species, and major concentrations of aromatic hydrocarbons containing from 3 to 7 aromatic rings. Base fractions contain predominantly single nitrogen compounds of azaarene or aminoaromatic type. Aminoaromatics (compounds analogous to aniline) are present in significant amounts in products made from New Mexico subbituminous coal but are nearly absent in the Texas lignite product. Acid fractions contain appreciable quantities of pyrrolic benzologs, but surprisingly low concentrations of compounds with a free OH group.

INTRODUCTION

Earlier phases of this project have been involved with improving our understanding of the fundamental chemistry of coprocessing through the development of the ability to distinguish between compound classes originating from coal versus those originating from petroleum resid. Two approaches were evaluated as potential methods to accomplish this objective. One was based on the use of carbon isotope ratios and the other was based on variations in compound classes in

the coprocessing products in response to changes in the ratio of coal to resid in the coprocessing feed. Results of these studies have been summarized in prior quarterlies (1)(2).

An additional objective is to develop an expanded knowledge of the detailed composition of coprocessing products. This knowledge will aid in the development of improvements in coprocessing, guide development of refining methods for coprocessing liquids and provide information on the quality of coprocessing products. Studies of the detailed composition of coprocessing products made over a range of coal concentration in a batch autoclave have been reported in previous quarterlies (2).

This and the previous quarterly report (3) address the detailed analysis of coprocessing process development unit (PDU) samples obtained from HRI, Inc. Emphasis was placed on resid samples; detailed knowledge of the composition of the resid fractions is of interest as this is material which failed to be converted to distillate range materials. Coprocessing resid samples selected for detailed analysis were made under constant conditions except for variations in coal concentration or in the coal (New Mexico subbituminous or Texas lignite). The soluble material was separated into strong and weak acids, strong and weak bases, and neutrals. The predominant fraction from the ABN separations was the neutral fraction (67-81%). All of the polar fractions increase with increasing coal concentration. The concentration of saturates in the neutrals is high in a run with 33% subbituminous coal but drops substantially with either increasing coal concentration or the substitution of lignite for subbituminous coal. High temperature gas chromatography showed that both the neutral aromatics fractions and saturates fractions from all of the runs are extremely similar regardless of the coal concentration or coal type. The neutral aromatics fractions were further separated by ring number separation. Chromatograms were again very similar regardless of the initial coal concentration or coal type with most material eluting in the 3-ring to 6-ring region. This report includes results of nonaqueous titrations, elemental analyses and infrared spectroscopy.

EXPERIMENTAL

Total C, H, N and S were determined on all liquid chromatographic (LC) fractions as well as the insolubles left over from the initial separation of each sample into benzene solubles and insolubles. A Perkin Elmer Model 240 Elemental Analyzer was used for determination of C and H. Nitrogen and sulfur were determined after ASTM methods D 4629 and D 1552/IR, respectively.

Infrared spectra were recorded on dichloromethane solutions of LC fractions using a Perkin Elmer Model 283 double beam dispersive instrument. Concentrations ranged from 10-20 mg/mL; 0.5mm path length KBr liquid cells were employed. Concentrations of acidic nitrogen in acid fractions were calculated using the response of the N-H stretching band of dibenzo[a,i]carbazole for calibration.

Nonaqueous titration of base fractions was carried out in acetonitrile and acetic anhydride solvents. Perchloric acid in dioxane was used as the titrant. Further details of the method and calculation of the various types of bases from results obtained in the two solvents is described elsewhere (4,5)

RESULTS

A set of eight coprocessing samples was received from HRI, Inc. Based on run conditions and preliminary analyses, a set of vacuum still bottoms samples was selected for detailed analysis. The samples were selected so that processing conditions were constant except for variations in feed composition. The samples selected allowed a determination of the effect of changing coal concentration and a comparison of results with subbituminous coal versus lignite.

Samples were selected from run 238; in all cases reaction temperature was 810° F, relative space velocity was 1 and the petroleum resid was Hondo. Variable conditions are as follows:

<u>Sample, wt %</u>	<u>Coal</u>	<u>Coal Concentration, %</u>	<u>Resid Conversion</u>
238-8-5	New Mexico*	33	93
238-8-9	New Mexico	50	90
238-10-8	Texas Lignite	33	93

*Subbituminous coal from the McKinley Mine

Thus, 238-8-5 and 238-8-9 vary only in coal concentration and 238-8-5 and 238-10-8 vary only in the coal feedstock. In all cases the samples further analyzed were vacuum still bottoms (VSB). Separation of the resid into acid, base and neutral fractions and further separation of the neutral fraction into saturates and neutral-aromatics was reported in the previous quarterly (3). For convenience of the reader, results of these separations are summarized in Table 1 and 2.

TABLE 1. - Liquid chromatographic fractionation of HRI resids

Fraction	238-8-5	238-8-9	238-10-8
---Vacuum Still Bottoms Basis, wt%---			
A. Separation of Benzene Solubles			
Benzene Solubles	47.2	44.7	29.8
Benzene Insolubles	53.8	55.3	70.2
----Benzene Solubles Basis, wt%----			
B. ABN Separation			
Strong Acids	12.2	17.4	21.0
Weak Acids	7.0	13.2	14.2
Total Acids	19.2	30.6	35.2
Strong Bases	5.5	9.3	6.8
Weak Bases	0.8	1.2	1.0
Total Bases	6.3	10.5	7.8
Neutrals	80.8	67.3	67.4
Total	106.3	108.4	110.4

TABLE 2. - Liquid chromatographic separation of neutrals into saturates and neutral aromatics

Fraction	238-8-5	238-8-9	238-10-8
-----VSB Benzene Solubles Basis ^a , wt%---			
Saturates	55.5(69.8)	17.4(25.7)	20.8(31.7)
Neutral Aromatics	24.0(30.2)	50.2(74.3)	44.9(68.3)
Total	79.5	67.6	65.7
Recovery, %	98.4	100.4	97.5

^a Values within parentheses are on a normalized neutrals basis

Table 3 shows results from elemental analyses. In general, the elemental content of a given fraction, e.g. saturates, over each of the resids is relatively constant. Thus, differences in elemental analyses for each of the whole resids, noted in the previously quarterly report, indicate differences in their fraction distributions to a much greater extent than variations in elemental content for the fractions themselves.

Approximately one third of the benzene insolubles obtained is comprised of carbon. The H/C ratio of these materials is extremely low, near that of petroleum coke, which accounts for the insolubility of the organic matter present. Some or all of the sulfur and nitrogen present in the insolubles may exist in inorganic forms.

Table 4 provides a summary of the nonaqueous titration data for strong and weak base fractions. The distribution of basic types measured by titration depends significantly on the type and amount of coal used during coprocessing. For example, aminoaromatics (compounds analogous to aniline) are present in significant amounts in the two products made from New Mexico subbituminous coal, but are nearly absent in the Texas lignite product. Also, the product from 50% subbituminous coal contains more aminoaromatics than that from 33% subbituminous coal. The trend for very weak bases closely follows that of aminoaromatics, but the pattern for azaarenes (compounds analogous to pyridine) opposes that of the other two types.

Each sample contains 6-8% of bases too weak to be detected by the titration system employed. Those types partition into the weak base fraction exclusively.

Equivalent weights for strong base fractions are also listed in the table. Equivalent weights can normally be used as estimates of average molecular weights for base fractions free of nontitratable species. As shown in table 5, the equivalent weights thus determined compare favorably with average molecular weights for saturate and neutral-aromatic fractions estimated from boiling point distributions shown in the previous quarterly report. Fractions from the two resids produced from subbituminous coal have slightly lower average molecular weights than those from the lignite coal product. The variation in molecular weight between compound types in a given resid is relatively small.

Infrared (IR) spectra of base fractions exhibited bands consistent with nitrogen present only in aromatic systems, e.g. azaarenes, and, in the case of the two subbituminous coal products, primary aromatic amine groups. Other heteroatoms present are probably

TABLE 3. - Elemental analyses of LC fractions and benzene insolubles from HRI coprocessing resids

Sample No.	Fraction	wt%					H/C atomic Ratio
		C	H	N	S	Total	
238-8-5	saturates	86.46	14.48	<0.01	<0.01	100.94	1.996
(67% Hondo:	neutral-aromatics	89.72	9.07	0.116	0.143	99.05	1.205
33% New	strong acids	74.22	6.49	1.20	0.121	82.03	1.042
Mexico	weak acids	82.22	7.81	1.46	0.157	91.65	1.132
subbituminous) ¹	strong bases	84.18	8.50	2.25	0.414	95.34	1.203
	weak bases	86.64	8.86	1.45	0.56	97.51	1.219
	insolubles	32.36	1.96	0.39	1.11	35.82	0.722
238-8-9	saturates	85.77	14.27	<0.01	<0.01	100.04	1.983
(50% Hondo:	neutral-aromatics	90.15	8.76	0.122	0.206	99.24	1.158
50% New	strong acids	79.91	7.01	1.25	0.180	88.35	1.045
Mexico	weak acids	83.24	8.32	1.00	0.150	92.71	1.190
subbituminous) ¹	strong bases	79.85	7.74	3.33	0.419	91.34	1.155
	weak bases	85.76	8.56	1.75	0.74	96.81	1.189
	insolubles	35.92	2.20	0.39	0.70	39.21	0.305
238-10-8	saturates	86.32	14.38	<0.01	<0.01	100.70	1.985
(67% Hondo:	neutral-aromatics	89.86	9.43	0.126	0.323	99.74	1.250
33% Texas	strong acids	80.99	6.84	1.16	0.217	89.21	1.006
Lignite) ¹	weak acids	82.49	8.22	1.31	0.262	92.28	1.187
	strong bases	86.24	8.02	2.06	0.379	96.70	1.108
	weak bases	83.30	8.62	1.53	0.90	94.85	1.226
	insolubles	27.33	1.46	0.35	1.87	31.01	0.637

¹ Feedstocks for coprocessing run

TABLE 4. - Results from nonaqueous titration of base fractions

Sample No.	Fraction	Equivalent Weight (g/EQ)	mole percent			
			Azaarenes	Amino- aromatics	Very Weak Bases	Nontitratable ⁴ Bases
238-8-5	strong bases	554	54.6	21.4	24.0	-
(67% Hondo:	weak bases	-	12.3	7.0	19.7	61.0
33% NM	total bases ²	554	49.2	19.6	23.5	7.7
subbit.) ¹						
238-8-9	strong bases	521	29.1	27.3	43.6	-
(50% Hondo:	weak bases	-	14.4	nd ³	32.7	52.9
50% NM	total bases ²	521	27.4	24.2	42.4	6.0
subbit.) ¹						
238-10-8	strong bases	654	87.2	3.8	9.0	-
(67% Hondo:	weak bases	-	8.9	2.6	42.0	46.5
33% TX	total bases ²	654	77.2	3.6	13.2	6.0
Lignite)						

¹ Feedstocks for coprocessing run² Calculated from the yields of strong and weak base fractions³ None detected⁴ Estimated assuming the equivalent weight for the corresponding strong base fraction

TABLE 5. - Estimated average molecular weights for selected LC fractions

Sample No.	Fraction	MW (g/mole)
238-8-5	saturates ²	550
(67% Hondo:	neutral-aromatics ²	600
33% NM	strong bases ³	554
subbit.) ¹		
238-8-9	saturates	550
(50% Hondo:	neutral-aromatics	620
50% NM	strong bases	521
subbit.) ¹		
238-10-8	saturates	620
(67% Hondo:	neutral-aromatics	650
33% TX	strong bases	654
Lignite) ¹		

¹ Feedstocks for coprocessing run

² Estimated from midboiling point determined via high temperature GC simulated distillation

³ Equivalent weight determined from nonaqueous titration in acetic anhydride

also contained in aromatic systems based on the lack of IR bands indicating other functional groups. Thus, the very weak base class determined by nonaqueous titration is most likely made up of azaarene species with additional heteroatoms nearby which reduce the overall basicity of the nitrogen (5).

The elemental nitrogen content of strong base fractions (table 3) coupled with their average molecular weights in table 5 indicate 0.89, 1.24, and 0.96 N atoms per molecule for 238-8-5, 238-8-9, and 238-10-8 resids, respectively. Thus, strong base fractions are predominantly comprised of nitrogen-containing species. On the other hand, weak base fractions contain only 0.57, 0.65, and 0.71 N atoms per molecule, respectively, for 238-8-5, 238-8-9 and 239-10-8 resids. The lack of infrared evidence for other basic types and appreciable proportions of nontitratable species

indicated by titration indicate that the weak base fractions, which comprise only 0.8-1.2 wt.% of the benzene solubles from each resid, also contain 30-40% of neutral-aromatic species present as contaminants.

Free OH bands, characteristic of phenolic-type compounds, were of very weak to negligible intensity in infrared spectra of strong and weak acid fractions. This apparent lack of free hydroxyl in groups in partially coal-derived products is quite unexpected. A very broad band, typical of hydrogen-bonded OH, was apparent, however. Strong acid and to a somewhat less extent, weak acid fractions exhibited strong carbonyl absorptions at 1780 and 1730 cm^{-1} . Assignment of these bands to a specific functional group is not possible with the available data. The only unequivocal band observed in acid fraction spectra was that of NH groups present in pyrrolic-type structures.

Table 6 compares the proportion of nitrogen present in pyrrolic type compounds in acid fractions versus other forms of nitrogen. Virtually all of the nitrogen in strong acid fractions is present as pyrrolic-type compounds. Up to about one-third of the nitrogen in weak acid fractions is present in some other form.

The overall percentage of nitrogen and its speciation in acidic fractions is relatively insensitive to the type and quantity of coal used, unlike the situation for basic fractions. However, since the overall yield of acids is affected by feedstock parameters (see previous quarterly), it follows that the quantity of acidic nitrogen compounds produced is proportionally related to the amount and type of coal used.

DISCUSSION

The combination of H/C ratios, average molecular weights estimated from GC data and, in the case of neutral-aromatic fractions, LC profiles for ring-number distributions may be used to derive average carbon skeletons for saturate and neutral-aromatic fractions from the HRI coprocessing resids.

Table 7 lists H/C ratio as a function of naphthene ring number for saturated hydrocarbon homologs with molecular weights approximately equal to those determined for the coprocessing saturate fractions. Comparison of the H/C ratios of saturate fractions from the Hondo: subbituminous coal products (average molecular weight = 550) with those in table 7 indicates that saturates from either fraction contain, on the average, only 1-2 naphthene rings per molecule. Of course, this is only an average parameter and, in reality, a range of naphthene ring-number species

TABLE 6. - Speciation of nitrogen in acid fractions (wt.% N)

Sample No.	Fraction	Total N ²	N as NH ³	Other N types ⁴
238-8-5	strong acids	1.20	1.26	-
(67% Hondo:	weak acids	1.46	1.04	0.42
33% NM	total acids ⁵	1.29	1.18	0.11
subbit.) ¹				
238-8-9	strong acids	1.25	1.22	0.03
(50% Hondo:	weak acids	1.00	0.80	0.20
50% NM	total acids	1.14	1.04	0.10
subbit.) ¹				
238-10-8	strong acids	1.16	1.17	-
(67% Hondo:	weak acids	1.31	0.83	0.48
33% TX	total acids	1.22	1.03	0.19
Lignite) ¹				

¹ Feedstocks for coprocessing run

² From Table 1

³ From IR. NH functional groups are most likely in pyrrolic structures

⁴ Other N types=Total N - N(NH)

⁵ Calculated from yields of strong and weak acid fractions

TABLE 7. - H/C ratios of saturated hydrocarbon homologs with molecular weights near 550 and 620

Number of Naphthenic Rings	Empirical Formula	MW	H/C Ratio	Empirical Formula	MW	H/C Ratio
0	C ₃₉ H ₈₀	548	2.051	C ₄₄ H ₉₀	618	2.046
1	C ₃₉ H ₇₈	546	2.000	C ₄₄ H ₈₈	616	2.000
2	C ₃₉ H ₇₆	544	1.949	C ₄₄ H ₈₆	614	1.955
3	C ₃₉ H ₇₄	542	1.897	C ₄₄ H ₈₄	612	1.909
4	C ₃₉ H ₇₂	540	1.846	C ₄₅ H ₈₄	624	1.867
5	C ₄₀ H ₇₂	552	1.800	C ₄₅ H ₈₂	622	1.822
6	C ₄₀ H ₇₀	550	1.750	C ₄₅ H ₈₀	620	1.778

are present. The analogous comparison of the Hondo: lignite saturates (average molecular weight = 620) reveals an average ring number of 1-2 for that fraction as well.

The analogous comparison for neutral-aromatic compounds was initially based on the assumption that aliphatic carbons were present solely in acyclic forms. Matching experimental H/C ratios in table 1 with those of *n*-alkyl-substituted aromatics in table 8 resulted in estimates of average aromatic ring numbers near 6 for all three resids. However, reexamination of actual ring number distributions (see previous quarterly) determined by HPLC revealed that this estimate was erroneously high. All three fractions showed very similar HPLC chromatograms indicating distributions from approximately 3 to 6 aromatic rings per molecule with an average near 4 rings per molecule.

Reconciliation of the HPLC versus H/C data is possible if one considers naphthenic forms of aliphatic carbon. For example, the observed H/C ratios of neutral-aromatic fractions match those listed in table 9 for 4-ring aromatics containing from 5-7 naphthenic rings. The hypothetical aromatic hydrocarbon shown in figure 1 fits the available data for the 238-8-5 sample well; on the average, the 238-8-9 and 238-10-8 neutral-aromatics contain about 1 more and 1 less naphthene ring, respectively.

The appreciably different carbon skeletons for saturated (1-2 naphthene rings) versus neutral-aromatic (ca. 10 rings, 4 of which are aromatic) hydrocarbons points to: 1) a different origin for

TABLE 8. - H/C ratios of *n*-alkyl-substituted aromatic hydrocarbons with molecular weights near 600

Number of Aromatic Rings	Empirical Formula	MW	H/C Ratio
0	C ₄₃ H ₈₈	604	2.047
1	C ₄₃ H ₈₀	596	1.861
2	C ₄₄ H ₇₆	604	1.727
3	C ₄₄ H ₇₀	598	1.591
4	C ₄₄ H ₆₄	592	1.454
5	C ₄₅ H ₆₀	600	1.333
6	C ₄₅ H ₅₄	594	1.200
7	C ₄₆ H ₅₀	602	1.087
8	C ₄₆ H ₄₄	596	0.957

TABLE 9. - H/C ratios of 4-ring aromatic hydrocarbons, ca. 600 molecular weight, with varying numbers of naphthenic rings

Number of Naphthenic Rings	Empirical Formula	MW	H/C Ratio
0	C ₄₅ H ₆₆	606	1.467
1	C ₄₅ H ₆₄	604	1.422
2	C ₄₅ H ₆₂	602	1.378
3	C ₄₅ H ₆₀	600	1.333
4	C ₄₅ H ₅₈	598	1.289
5	C ₄₅ H ₅₆	596	1.244
6	C ₄₅ H ₅₄	594	1.200
7	C ₄₅ H ₅₂	592	1.156
8	C ₄₅ H ₅₀	590	0.111

$C_{45}H_{54}$

H/C = 1.200

MW = 594

FIGURE 1. - Hypothetical neutral-aromatic species in HRI coprocessing resids

each type and 2) little interconversion between saturates and aromatics during coprocessing. Furthermore, if significant hydrogenation of aromatics to saturates occurred, one would expect much more prominent concentrations of one and two aromatic-ring molecules than actually observed in HPLC chromatograms.

Based on their H/C ratios, the carbon skeletons of acid and base fractions are probably similar to those of neutral-aromatics. For example, the H/C ratios of 238-8-5 strong and weak bases are quite similar to that of the corresponding neutral-aromatic fraction, while those of the strong and weak acids are somewhat lower, indicating slightly greater aromaticity (approximately one more aromatic ring) for those fractions.

CONCLUSIONS

The concentration of aminoaromatics in base fractions appears to be a sensitive indicator of the amount and type of coal used in coprocessing. The hydrocarbon skeletons of saturated hydrocarbons in the HRI coprocessing resids appears to be fundamentally different than those of aromatic species. The average molecular weights of various compound types in a given resid are similar to one another; the overall average molecular weights for Hondo: subbituminous coal products are somewhat lower than those for the Hondo: lignite product. The average molecular weights determined for these resids (≤ 650) are well below those of typical straight-run petroleum $>1000^\circ\text{F}$ resids (≥ 900)(5). Base fractions contain predominantly single nitrogen compounds of azaarene or aminoaromatic type. Acid fractions contain appreciable quantities of pyrrolic benzologs, but surprisingly low concentrations of compounds with a free OH group. Neutral-aromatic fractions contain minor levels of sulfur compounds, an unknown proportion of ether or other oxygen-containing species, and major concentrations of aromatic hydrocarbons containing from 3 to 7 aromatic rings.

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