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DOE/PC/92521--T157

TECHNICAL REPORT
March 1, 1994 through May 31, 1994

Project Title: **REACTIVITY OF TARGET COMPOUNDS FOR CHEMICAL COAL DESULFURIZATION**

DOE Grant Number: DE-FC22-92PC92521 (Year 2)
ICCI Project Number: 93-1/5.3A-1P
Principal Investigator: Dr. David H. Buchanan, EIU
Other Investigators: Ms. Mandy Amin, EIU
Mr. Richard Cunningham, EIU
Ms. Janette Galyen, EIU
Project Manager: Dr. Ken K. Ho, ICCI

ABSTRACT

This project seeks to identify representative organosulfur compounds which are removed by known coal desulfurization reactions. Demineralized coals are solvent extracted and the extracts fractionated to concentrate organosulfur compounds for analysis by Gas Chromatography/Mass Spectroscopy. After sulfur compounds are characterized, the parent extracts are subjected to reactions previously shown to reduce the organic sulfur content of Illinois coals, fractionated and again analyzed for organosulfur content to determine if the identified compounds reacted during the chemical treatment. The original coal also will be subjected to chemical desulfurization, extraction, fractionation and analysis in order to correlate changes in organic sulfur content of the coal with reactions of specific sulfur compounds. These compounds can thus be reliably considered as target molecules for the next generation of desulfurization processes.

Work during this quarter has shown that fractionation and chromatography of pyridine extracts to isolate suitable samples for GC/MS analysis, although time-consuming, appears to be better than direct toluene extraction in terms of providing a representative set of compounds for analysis. The toluene soluble fractions prepared by this route contain aromatic sulfur compounds and O, N, S-containing hetrocycles. A set of these compounds has been identified and their fate following desulfurization of the parent coal extracts is under investigation.

Previously studied desulfurization reactions using the single electron transfer reagent, K/THF/naphthalene, and the reactive nickel boride reagent have been repeated and analyzed by GC/MS. SET and nickel boride reactions of the THF soluble portions of pyridine extract of IBC-101 coal are currently in progress.

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EXECUTIVE SUMMARY

The goal of this project is improved chemical coal cleaning by determination of the reactivity of several desulfurization reagents with specific organosulfur compounds shown to be present in the feed coal. Reagent testing has been preceded by identification of a representative group of organosulfur compounds which can be extracted from coal with good solvents and then separated and concentrated by chromatography. Analytical methods such as Gas Chromatography / Mass Spectroscopy are used to determine which sulfur compounds are converted to sulfur-free products during the course of the desulfurization reactions.

Work during this quarter has shown that fractionation of pyridine extracts to isolate suitable samples for GC/MS analysis of coal extracts, although more time-consuming, appears to be better than direct toluene extraction in terms of providing a representative set of compounds for analysis. Suitable pyridine extracts are available or in preparation from Argonne 301, IBC-101, -105 and -108 coals.

All coal extracts are fractionated and purified by column chromatography before GC/MS analysis. Best results are obtained by stirring overnight a pyridine extract with 10-20 times its weight of distilled THF, separating the insoluble reside and concentrating the THF soluble portion by rotary evaporation followed by chromatography on a neutral alumina column packed in hexane. Elution with hexane followed by toluene gave aliphatic and aromatic hydrocarbon fractions containing non-polar organosulfur compounds. The toluene fractions also contain hetrocyclic sulfur compounds containing oxygen and/or nitrogen. A set of the sulfur compounds has been identified by GC/MS and their fate following desulfurization of the parent coal extracts is under investigation.

Previously studied desulfurization reactions using the single electron transfer reagent, K/THF/naphthalene, and the reactive nickel boride reagent have been repeated on dibenzothiophene to give 52 and 49% desulfurization respectively, as analyzed by GC/MS. SET and nickel boride reactions of the THF soluble portions of the pyridine extract of IBC-101 coal are being carried out and these results will be used to determine the extracts to study next.

The failure to detect aliphatic sulfur compounds in any coal extract is disappointing and points out the limits of using only a low-resolution GC/MS for these analyses. Future work along these lines should involve several labs with complimentary analytical abilities.

OBJECTIVES

The primary objective of this project is to identify as many as possible of those organosulfur compounds which may be extracted from coal by simple solvent treatment below 150°C and to determine their fate when the coal fractions in which they are found are treated with known coal desulfurization chemical reactions.

Specific goals of this project are to:

1. Maximize the organic material extracted from IBC-101 and -108 using pyridine/nBu_4NOH/ultrasound.

2. Demineralize Argonne Premium Coal Sample 301 (parent of IBC-105) and extract as in 1.

3. Fractionate pyridine extracts into sub-fractions using solubility in hexane and toluene for further analysis.

4. Separate and concentrate sub-fractions containing sulfur compounds using preparative chromatography and/or extrography.

5. Analyze sulfur-containing sub-fractions by GC/MS correlated with SEC, FT-IR and FT-NMR analyses to identify as many organosulfur compounds as possible.

6. Correlate analytical data with extraction and fractionation yields to develop mass balances for identified sulfur species.

7. Desulfurize characterized sub-fractions and total pyridine extracts by several methods such as: Dissolving metals, Nickel boride, Ni/Al alloy, and Lochmann's BASE.

8. Repeat goals number 3-6 on the desulfurized sub-fractions and extracts.

9. Use the best methods from goal 7 on demineralized coals and repeat goals 1-6 on the coal product.

10. Correlate and summarize the data to determine which organosulfur compounds react in the desulfurization methods and determine how much of the observed desulfurization can be accounted for in the reactions of identified compounds.

INTRODUCTION AND BACKGROUND

The need to understand sulfur chemistry in Illinois coals is central to our need to protect both the environment and the

job base in the Illinois coal fields. Even if all mineral sulfur were removed, most Illinois coals could not meet existing or scheduled sulfur emission standards. Organic sulfur must be removed and this will involve selective chemical reactions. Previous work at EIU has shown that simple processes such as solvent extraction do not selectively remove sulfur from coal - that is, during solvent extractions sulfur compounds were removed by both polar and non-polar solvents in approximately the same concentration as they were found in the original solid coal.[1] One impediment to the development of effective pre-combustion desulfurization technology is that the actual target compounds are not known. Although there have been many recent studies of organic sulfur in coal, including those sponsored by ICCI, few sulfur compounds have been identified as existing in whole coal. Non-destructive spectroscopic methods such as XANES and very high field ESR show great promise for determining the major classes of organosulfur functional groups present in coal or coal products. However, at the resolution available now or in the next few years, even these very expensive and sophisticated methods are not able to identify individual compounds.

It is unreasonable to expect advanced coal cleaning or utilization processes to develop if the actual target compounds in sulfur removal schemes are unknown. Knowing the <u>nature</u> of sulfur species present (*ie* relative amounts of aliphatic sulfides, aromatic sulfides, thiophenes, *etc.*) is important, but even more important is knowing the identity and amount present of the actual compounds. A simple example will illustrate the problem. Oxidation of sulfide to sulfoxide is the necessary first step in any oxidative desulfurization process. Dibutyl sulfide and dioctyl sulfide are very similar aliphatic sulfides, yet oxidation of sulfide sulfur to the sulfoxide by air in the presence of hydroaromatic promoter compounds is much faster for the butyl than for the octyl case and both react slower than dibenzylsulfide.[2] Because all three of these compounds involve $-S-CH_2-$ bonds, they would be measured as aliphatic S in the XANES analysis and the first two would not be observed by any current ESR technique. Design of desulfurization processes would be greatly improved by better information on the identity and reactivity of the target molecules in coal.

EXPERIMENTAL PROCEDURES

Coals studied this quarter include IBC-101, -105 and Argonne Premium Coal Samples 301 (Illinois #6, the parent of IBC-105) and 401 (Pittsburgh #8). The Argonne coals were included in the study to maximize pyridine extraction yield since they offer a range of volatile and mineral matter contents and have not suffered air oxidation in storage and handling. Solvent extraction and product purification and

isolation follow our published procedures.[3] Coals, extracts and residues are dried at 100°C, 0.1 Torr for several hours before weights are recorded for mass balance calculations. Direct toluene extractions of coal on a 1-2 g scale were conducted as described in the previous Quarterly Report. Solvents are HPLC grade, often distilled from a drying agent before use. Elemental analyses of coal fractions were done by Galbraith Laboratories, Knoxville, TN. Acid demineralization uses a modification of standard methods reported last quarter.

All coal extracts are fractionated and purified by column chromatography before GC/MS analysis. Best results are obtained by stirring overnight a pyridine extract with 10-20 times its weight of distilled THF, separating the insoluble reside and concentrating the THF soluble portion by rotary evaporation followed by chromatography on a neutral alumina column packed in hexane. Elution with hexane and toluene gave aliphatic and aromatic hydrocarbon fractions containing non-polar organosulfur compounds. The toluene fractions also contain hetrocyclic sulfur compounds containing oxygen and/or nitrogen. A set of these compounds have been identified by GC/MS and their fate following desulfurization of the parent coal extractions is under investigation.

Model compound desulfurization reactions were carried out following published procedures. Nickel boride desulfurization of dibenzothiophene duplicated our previously reported method[4] with the addition of GC/MS in place of GC analysis. Single electron transfer (SET) reactions using potassium in THF used our modification[4] of Stock's method[5], also with GC/MS analysis.

RESULTS AND DISCUSSION

Because mineral matter in coal is often found within the pores in the coal, it is possible that potentially soluble material is not being extracted due to blockage by the minerals. Our study of the effect of demineralization on solvent extraction using both toluene and pyridine, discussed in the previous report is now complete. Table 1 and Figure 1 summarize the study. Toluene yields after demineralization represent a significantly larger fraction of the initial extract yields than is the case for pyridine yields following demineralization. Previous work by the PI has shown that the toluene soluble fractions of pyridine extracts of Illinois No. 6 coal account for less than 50% of the material extracted by pyridine.[6] Taken together this suggests that the presence of mineral matter prevents toluene from removing material which is toluene-soluble. A working hypothesis is that minerals block pores such that toluene cannot reach potentially soluble material, which can be removed by pyridine which swells Illinois coals to more than two and one half times the original volume. Diffusion

through the matrix or through pathways opened around mineral grains during swelling are possible routes for removal of this material.

Since the relative amount of toluene soluble material extracted only after demineralization is large, it was of interest to see if it differed in elemental composition from the material extracted before demineralization. Toluene extracts from Argonne 301 and 401 coals were sent to Galbraith Labs for analysis as shown in Table 2. Unfortunately, our only sample of one fraction was lost by the lab during processing and due to the large amount of work needed to prepare another one, we have not repeated the work. Nevertheless, from the limited data in Table 2, it does appear that the second extract may differ from the first. As a result of this study, we believe that fractionation of pyridine extracts (which remove all toluene soluble material in one operation) rather than direct toluene extraction must be used for our future work.

Compounds extractable from Illinois coal which can be analyzed using the EIU system are those which will elute from the GC column below 290°C with mass less than 660 daltons (the upper limit of the HP 5971 quadrupole detector). These are generally those compounds which are found in hexane or toluene coal extracts. During the last two quarters, we attempted to develop a short method for obtaining representative solvent extract fractions without using pyridine. Although direct toluene extraction can work for some samples of some coals, in our hands it did not prove to be reliable. We now believe that the best method is to fractionate a pyridine soluble extract into a THF soluble fraction (usually about 40-60%) of the pyridine extract and to chromatograph the THF fraction on neutral alumina. The hexane and toluene fractions eluting from the column are then concentrated by rotary evaporation before GC/MS analysis. This method is longer and more tedious than other methods but was the only one which gave reliable results on both pristine and oxidized coals from several seams.

Figures 2 and 3 are GC/MS traces of the hexane and toluene fractions from a pyridine extract of Argonne 301 coal. Neither fraction contained the elemental sulfur which was so prominent in the GC/MS trace of IBC-105 coal reported last quarter. Sulfur containing compounds identified with a high degree of confidence based on the MS fragmentation pattern are marked on the GC traces and are listed in Table 3. At this point no aliphatic sulfur compounds have been identified, most likely due to their low abundance in these extracts and the limitations of the instruments available at EIU. The compounds in Table 3 plus others which may be identified as work continues will be monitored before and after desulfurization reactions on the parent coal extract fractions. By comparing the relative areas of the sulfur

compound peaks with those of nearby alkanes which do not react with the desulfurization reagent, it should be possible to determine whether or not the chemical treatment has removed a particular sulfur compound.

Previous desulfurization work at EIU involved both model compounds and coal fractions. Since both the personnel and analytical instrumentation have changed since that project was completed, we repeated the several reactions on the model compound dibenzothiophene (DBT), which is also a component of the solvent extracts of Illinois coal. The results are shown in Table 4. The SET reaction did not completely reduce DBT even though the green color of the active K^+/naphthalide$^-$ radical ion species was present at the end of the reaction and un-reduced naphthalene was detected in the quenched reaction mixture by GC/MS. In agreement with previous work, not all of the "missing" DBT has been desulfurized to biphenyl. We found 30% of the DBT converted to 2-phenyl thiophenol, the product of cleavage of only one of the two C-S bonds in DBT. Thus SET was only 52% effect in removing the sulfur from DBT during this reaction. This model reaction will be repeated using more naphthalene electron transfer agent.

In agreement with our previous work[4], Nickel boride, produced from $NaBH_4$ reduction of nickel chloride, gave only biphenyl from DBT, in up to 49% yield under reverse addition conditions. Since this reaction involves an insoluble reagent it will be limited to soluble coal fractions.

CONCLUSIONS AND RECOMMENDATIONS

Fractionation of pyridine extracts to isolate suitable samples for GC/MS analysis of coal extracts, although more time-consuming, appears to be better than direct toluene extraction in terms of providing a representative set of compounds for analysis. Suitable pyridine extracts are available or in preparation from Argonne 301, IBC-101, 105 and 108 coals.

Standard conditions for GC//MS sample preparation and analysis have been developed and a set of target sulfur molecules identified. Previously studied desulfurization reactions have been repeated on model compounds and the products analyzed by the new GC/MS system. SET and nickel boride reactions of the THF soluble portions of pyridine extract of IBC-101 coal will be carried out within the next two weeks and the results used to determine the extract to study next.

The failure to detect aliphatic sulfur compou in any extract is disappointing and points out the limits of using only GC/MS for these analyses. Future work should involve several labs with complimentary analytical abilities.

TABLE 1 EFFECT OF DEMINERALIZATION ON SOLVE EXTRACTION OF ARGONNE PREMIUM COAL

COAL SAMPLE BANK	ARGONNE #201	ARGONNE #301	ARGONNE #401
MAF % C	75.0	77.7	83.2
DRY ASH %	8.8	15.5	9.2
PYRIDINE EXTRACTION			
INITIAL			
EXTRACT %	8.4	25.8	30.7
RESIDUE %	90.2	75.9	63.3
AFTER DEMINERAL			
EXTRACT %	9.2	3.1	3.8
RESIDUE %	92.5	95.8	94.0
TOTAL EXTRACT %	17.5	28.9	34.6
% INCREASE DUE TO DEMINERALIZATION	109.3	12.2	12.5
TOLUENE EXTRACTION			
INITIAL			
EXTRACT %	0.92	1.38	0.62
RESIDUE %	98.0	100.0	100.6
AFTER DEMINERAL			
EXTRACT %	1.19	0.62	0.78
RESIDUE %	101.2	101.4	100.8
TOTAL EXTRACT %	2.1	2.0	1.4
% INCREASE DUE TO DEMINERALIZATION	129.3	44.9	125.8

TABLE 2 ELEMENTAL ANALYSES OF TOLUENE EXTRACTS OF ARGONNE PREMIUM COALS

COAL SAMPLE BANK SAMPLE NUMBER		ARGONNE #301	ARGONNE #401
FEED COAL	MAF % C	77.7	83.2
	MAF % H	5.0	5.3
	MAF % ORG S	2.38	0.89
	MAF % O	13.5	8.83
TOLUENE EXTRACTION			
INITIAL EXTRACT			
	MAF % C	84.5	86.2
	MAF % H	8.5	8.3
	MAF % ORG S	2.60	1.05
AFTER DEMINERAL			
	MAF % C	*	78.4
	MAF % H	*	7.5
	MAF % ORG S	*	1.02

* SAMPLE LOST BY ANALYTICAL LAB

TABLE 3

SULFUR CONTAINING COMPOUNDS IDENTIFIED BY GC/MS IN FRACTIONS OF PYRIDINE EXTRACTS OF ARGONNE #301 COAL

COMPOUND	FORMULA	QUALITY OF MS LIBRARY MATCH[a]
Benzothiazole	C_7H_5NS	87
2-Methylthiobenzothiazole	C_8H_7NS	98
Dibenzothiophene	$C_{12}H_8S$	94
2(3H)-Benzothiazolethione	$C_7H_5NS_2$	46
3-Ethyl-1,2,3,4-tetra-hydrodibenzothiophene	$C_{14}H_{16}S$	90
(this co-elutes with: Dibenzothiophene sulfone	$C_{12}H_8O_2S$	53

---

a McLafferty algorithm for judging goodness of match between MS of compound and Wiley Library of MS, Scale 0 - 100, matches below 50 are suspect.

A variety of other S-containing heterocyclic compounds are detected with Quality matches of 30 - 90, but because of their unusual structures, are not considered to be positively identified.

TABLE 4

DIBENZOTHIOPHENE (DBT) DESULFURIZATION

Method	K/Naphthalene[a]	Ni Boride[b] Normal Addn	Ni Boride[b] Reverse Addn
Product Mixture			
mol % DBT	18.3	74.0	50.9
mol % BP[c]	51.6	26.0	49.1
mol % PTP[d]	30.1	0[e]	0
% desulfurization	51.6	26.0	49.1

---

a References 4 and 5 b Reference 4
c Biphenyl d 2-Phenylthiophenol
e None detected, less than 0.1 %

REFERENCES

(1) Buchanan, D.H. in Proceedings: 1987 Workshop on Coal Structure, EPRI Report **1988**, ER-6099-SR, 9-1.

(2) a) Mill, T.; Tse, D.S.; Loo, B.; Yao, C.C.D.; Canavesi, E. *Prepr. Pap.-Am. Chem. Soc., Div. Fuel Chem.* **1992**, *37(3)*, 1367-1375. b) Recent results by L. Warfel at EIU and personnel communication with T. Mill at SRI.

(3) Buchanan, D.H.; Osborne, K.R.; Warfel, L.C.; Mai, W.; Lucas, D. *Energy Fuels* **1988**, *2*, 163.

(4) Buchanan, D.H.; CRSC Final Technical Report "New Reagents for Coal Desulfurization," August 31, 1991.

(5) Chatterjee, K.; Wolny, R.; Stock, L.M. *Energy Fuels*, **1990**, **4**, 402.

(6) Mayo, F.R.; Buchanan, D.H.; Pavelka, L.A.; Hirschon, A.S. *Fuel*, **1988**, *67*, 600-606.

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Figure 1

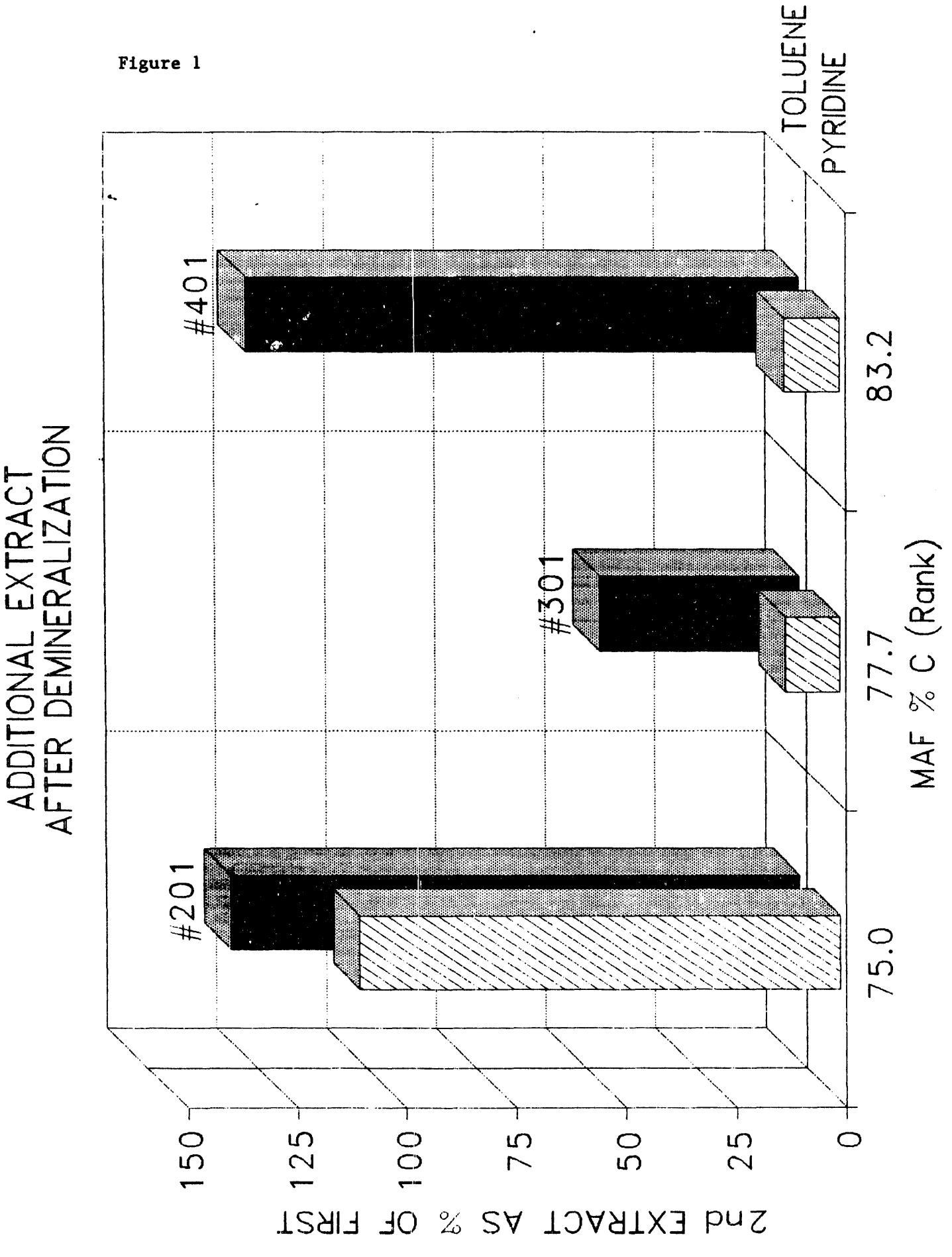
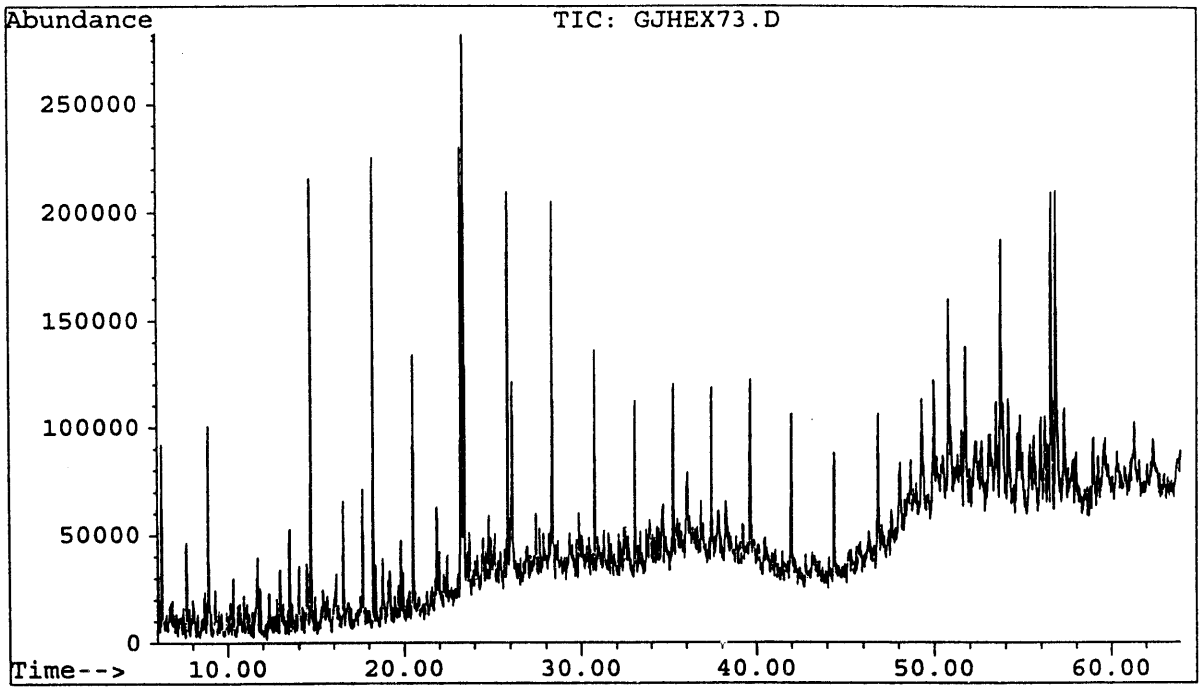

ADDITIONAL EXTRACT
AFTER DEMINERALIZATION
2nd EXTRACT AS % OF FIRST
150
125
100
75
50
25
0
#201
#301
#401
75.0
77.7
83.2
MAF % C (Rank)
TOLUENE
PYRIDINE

Figure 2

File : C:\HPCHEM\1\DATA\DHBTOL10\GJHEX73.D
Operator : DHB
Acquired : 26 May 94 10:15 am using AcqMethod MODHMLEE.M
Instrument : 5971 - In
Sample Name: HEX CHROMAT FRACT #3 COAL
Misc Info : TONG PY EXTRACT HEX SOL RERUN JGHEX73
Vial Number: 1



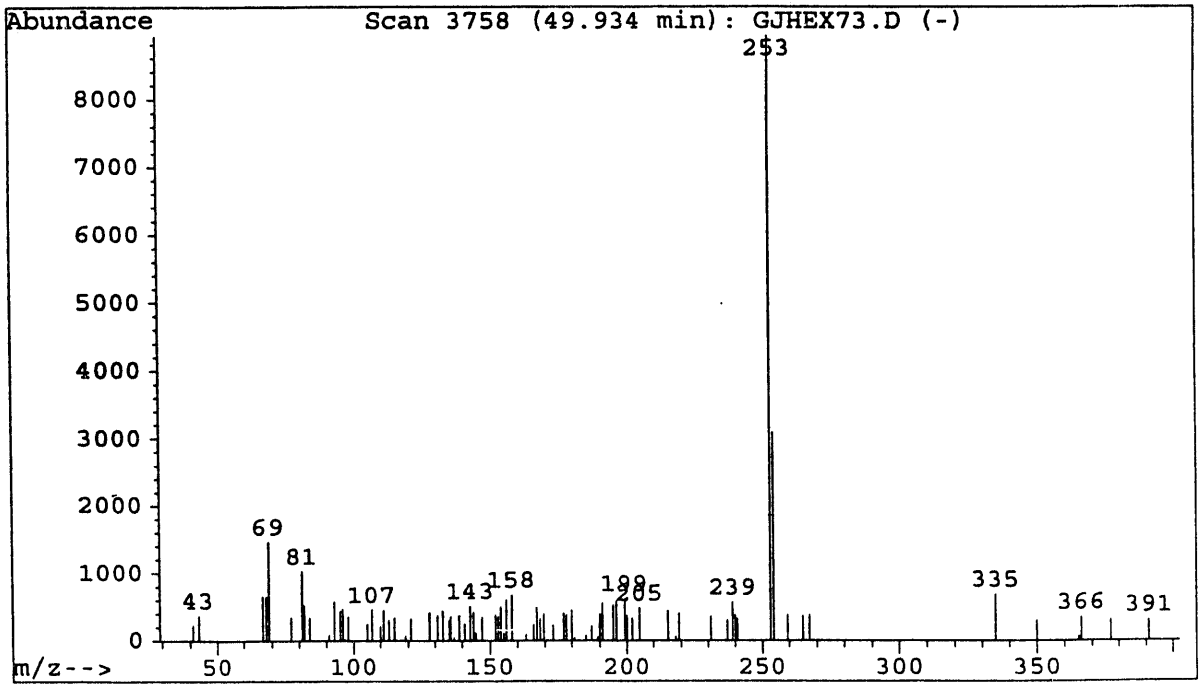
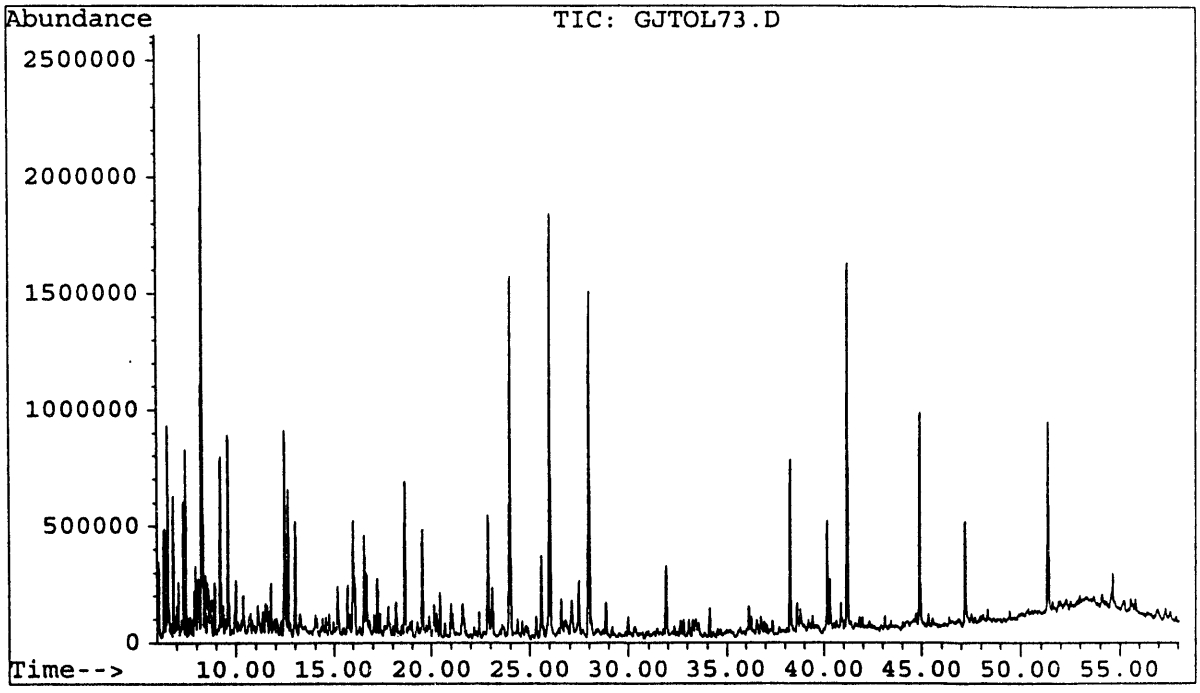
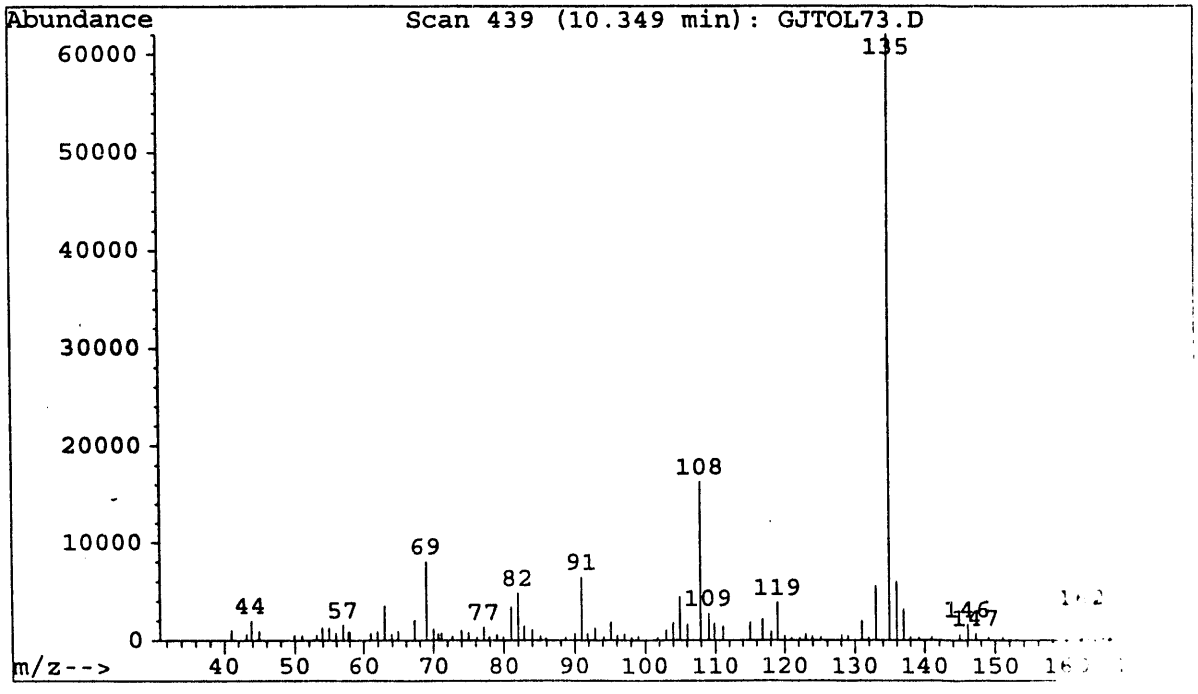


Figure 3

File : C:\HPCHEM\1\DATA\DHBTOL10\GJTOL73.D
Operator : DHB
Acquired : 26 May 94 8:28 am using AcqMethod MODTMLEE.M
Instrument : 5971 - In
Sample Name: TOL CHROMAT FRACT #3 COAL
Misc Info : TONG PY EXTRACT THF SOL RERUN JGTOL73
Vial Number: 1





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(1) Buchanan, D.H. in Proceedings: 1987 Workshop on Coal Structure, EPRI Report **1988**, ER-6099-SR, 9-1.

(2) a) Mill, T.; Tse, D.S.; Loo, B.; Yao, C.C.D.; Canavesi, E. *Prepr. Pap.-Am. Chem. Soc., Div. Fuel Chem.* **1992**, *37(3)*, 1367-1375. b) Recent results by L. Warfel at EIU and personnel communication with T. Mill at SRI.

(3) Buchanan, D.H.; Osborne, K.R.; Warfel, L.C.; Mai, W.; Lucas, D. *Energy Fuels* **1988**, *2*, 163.

(4) Buchanan, D.H.; CRSC Final Technical Report "New Reagents for Coal Desulfurization," August 31, 1991.

(5) Chatterjee, K.; Wolny, R.; Stock, L.M. *Energy Fuels*, **1990**, 4, 402.

(6) Mayo, F.R.; Buchanan, D.H.; Pavelka, L.A.; Hirschon, A.S. *Fuel*, **1988**, *67*, 600-606.

PROJECT MANAGEMENT REPORT
March 1, 1994 through May 31, 1994

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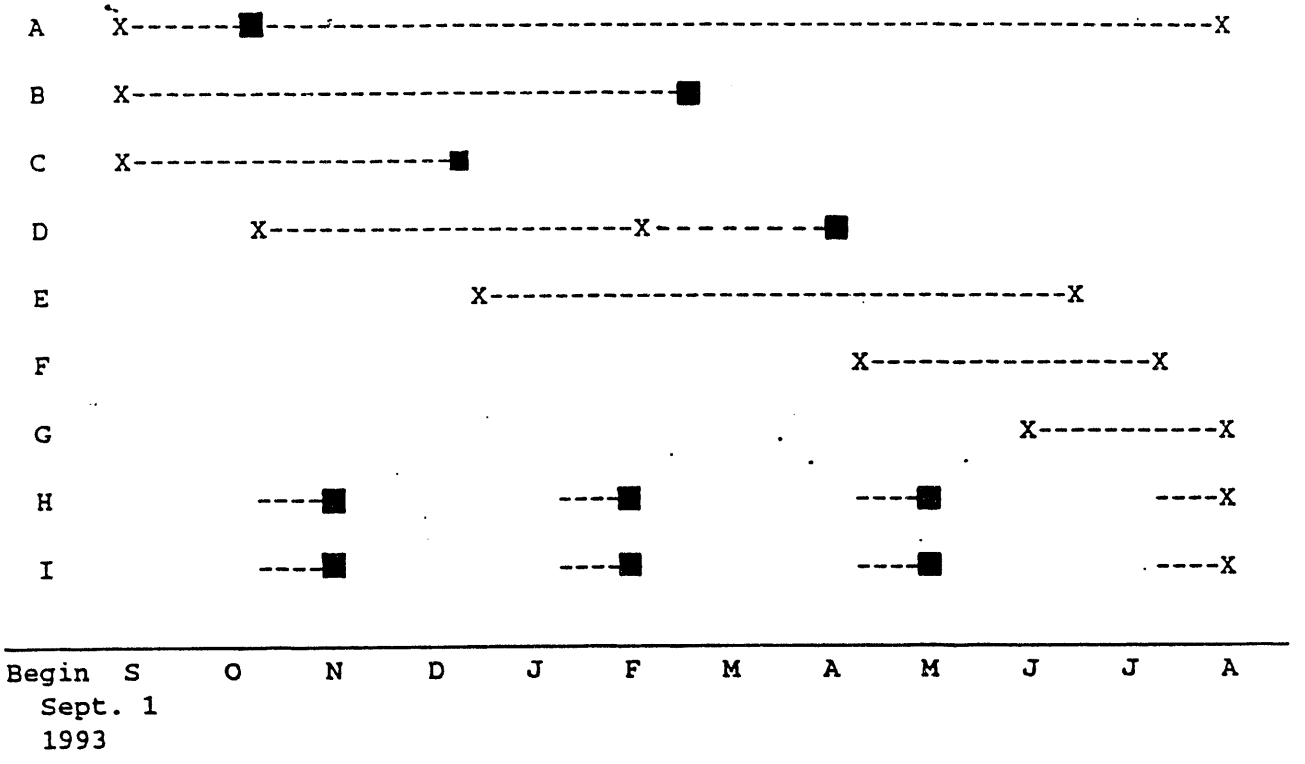
DOE Grant Number:	DE-FC22-92PC92521 (Year 2)
ICCI Project Number:	93-1/5.3A-1P
Principal Investigator:	Dr. David H. Buchanan, EIU
Other Investigators:	Ms. Mandy Amin, EIU
	Mr. Richard Cunningham, EIU
	Ms. Janette Galyen, EIU
Project Manager:	Dr. Ken K. Ho, ICCI

COMMENTS

During this Quarter, an additional $600 was transfered from ICCI to EIU account 5-20574 as partial reimbursement of travel costs for PI, Buchanan to present a paper to the Fuel Science Division of the American Chemical Society at the spring annual meeting in San Diego in March. The budget in Exhibits B and C has been increased by this amount.

SCHEDULE OF PROJECT MILESTONES



Milestones:

A. Personnel employed
B. Sample acquisition and preparation
C. Extraction of coal to maximize yield
D. Fractionation of extract to concentrate sulfur compounds
E. Analyses of fractions for sulfur compounds
F. Desulfurization reactions of extract and analysis of products
G. Desulfurization of coal, extraction and analysis of fractions
H. Technical Reports Prepared and Submitted
I. Project Management Reports prepared and submitted

Comment: D. Hardware and software probelms with GC/MS has delayed completion of analysis of extracts used to develop the fractionation method.

EXPENDITURES - EXHIBIT B

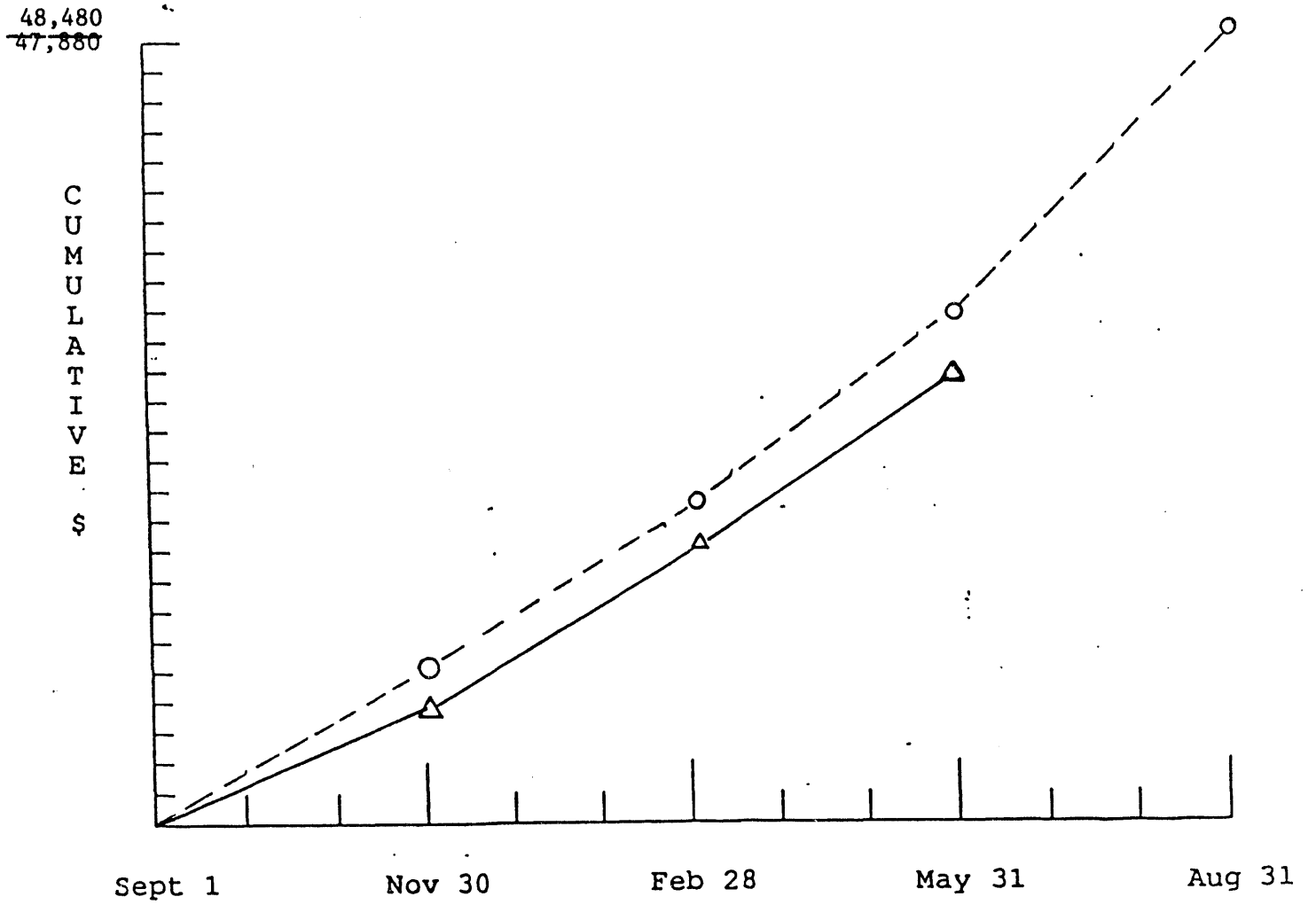
CUMULATIVE PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER

Quarter*	Types of Cost	Direct Labor	Fringe Benefits	Materials & Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept. 1, 1993 to Nov. 30, 1993	Projected	6,860	635	600	25	-0-	700	882	9,702
	Estimated	4,514	268	448	-0-	-0-	20	525	5,775
Sept. 1, 1993 to Feb. 28, 1994	Projected	13,720	1,270	1,200	50	400	1,300	1,794	19,734
	Estimated	10,715	1,639	1,321	-0-	112	366	1,415	15,568
Sept. 1, 1993 to May 31, 1994	Projected	20,580	1,905	1,800	50	850	1,900	2,708	29,793
	Estimated	17,939	2,897	1,643	600	618	535	2,423	26,655
Sept. 1, 1993 to Aug. 31, 1994	Projected	34,591	3,247	2,400	684 ~~139~~	850	2,300	4,408 ~~4,353~~	48,480 ~~41,880~~
	Estimated								

*Cumulative by Quarter

CUMULATIVE COSTS BY QUARTER -- EXHIBIT C

Reactivity of Target Compounds for Chemical Coal Desulfurization



Months and Quarters

O = Projected Expenditures ----------

Δ = Actual Expenditures ————————

Total ICCI Award $ ~~47,880~~ 48,480

DATE
FILMED
10/5/94

END