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EFFECT OF COAL BENEFICIATION PROCESS ON RHEOLOGY/ATOMIZATION OF COAL WATER SLURRIES.

Quarterly Progress Report
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Work Performed for the Department of Energy Under
Contract #DE-FG22-92MT92019

DISCLAIMER

OVERALL OBJECTIVE:

The overall objective of this project is to perform experiments to understand the effect of coal beneficiation processes and high shear rheological properties on the atomization of coal-water slurries (CWS). In the atomization studies, the mean drop size of the CWS sprays will be determined at various air-to CWS. A correlation between the high shear rheological properties, particle size distributions and the atomization will be made in order to determine the influence of these parameters on the atomization of CWS.

PROJECT STATUS:

Three different coal water slurry samples were received from Amax R&D. The samples were prepared from Upper Elkhorn No. 3 (Taggart). The properties of the slurries are as shown in the appendix. The samples supplied were -20 mesh-size samples, wet ground uncleaned slurry (99% passing through 48 mesh), wet ground heavy-media cleaned slurry (99% passing through 48 mesh), and wet ground froth flotation cleaned slurry (99% passing through 48 mesh). Also, a portion of the -20 mesh samples were dry ground to produce -48 mesh samples. FTIR analysis were performed on these samples in order to elucidate any differences in the functional groups due to the preparation of the CWS samples.

FTIR Analysis

The samples were dried in a nitrogen atmosphere and the FTIR transmittance spectrum of each sample was measured using a Mattson FTIR spectrophotometer model 2025, using KBr as a diluent.

Results

The absorption spectra of as received coal is as shown in Figure 1. Based on previous infrared spectral studies of coals, the following assignments were made.

1. A broad composite band, arising from the O-H groups within the organic matrix of the coals (alcoholic, phenolic, acidic, etc.) spans the region from 3600 to 3000 wavenumbers.
2. A band at 3025 wavenumber is due to the presence of aromatic hydrocarbons (Ar-H). This is a band present in all aromatic hydrocarbons. The peaks at 2917 and 2854 wavenumbers are to the presence of terminal vibrational modes of methyl (CH_3) groups and the methylene $-CH_2-$ and methene CH= gropus appended to the polynuclear matrix
3. The bands at 1608, 1437, and 1370 wavenumbers are due to -C=C-, CH_2, CH_3 and organic oxygenates, C-O-C, C-O-H, C-C-O. Clay minerals also exhibit lattice mode of vibration in this region.
4. The band at 1030 is due lattice mode of vibration of clay and silicates.
5. The bands at 855, 810 and 748 wavenumbers are due to out of plane bending modes of Ar-H and presence of silicates.

Dry Grinding

After grinding the original sample to -100 mesh, the spectrum in Figure 2 was obtained. The bands associated with the

O-H are much larger. There is a peak at 2337 cm^{-1} as a result of the grinding. Also the peak at 1030 cm^{-1} is more prominent. This is due to the exposure of the clay minerals to the surface. Figure 3 is the difference spectrum of Figure 1 from Figure 2. The CH_3, Ar-H and CH_2- absorption bands have disappered in the difference spectrum. The positive absorption bands (1094-773) observed in the difference spectrum is due to exposure of the clay and silicate minerals to the surface due to grinding.

Wet Grinding

Figure 4 is the spectrum of the uncleaned CWS sample and Figure 5 is the difference spectra of the uncleaned slurry and as received coal. The strong feature spanning the 3600 to 2500 wavenumbers in the difference spectrum results from the incorporation of considerable amounts of O-H into the organic coal matrix as a result of the wet grinding. This is due to hydrolysis to form phenolic (Ar-O-H) or acidic (-COO-H) groups. The positive absorption bands (1094-773) observed in the difference spectrum is due to exposure of the clay and silicate minerals to the surface due to grinding.

Flotation Cleaned Coal

Figure 6 is the spectrum of slurry prepared from a floation cleaned process and Figure 7 is the difference spectrum of the Floation and uncleaned CWS. Also, there is a lesser amount of O-H groups on the coal surface. The strong negative feature spanning the 3600 to 2500 wavenumbers results from lesser amounts of OH-groups on the coal surface than in the uncleaned slurry.

The addition of methyl isobutyl carbinol to the floatation media and subsequent reaction with the acid groups on the coal surface resulted in a lesser amount of OH-functional groups on the coal surface. The negative absorption bands (1094-773) observed in the difference spectrum is due to loss of clay and silicate minerals as a result of this cleaning process.

Heavy Media Cleaned Coal

Figure 8 is the spectrum CWS after heavy media cleaning and Figure 9 is the difference spectra of the wet ground (uncleaned) and wet ground (Heavy Media) cleaned coal. The negative absorption around 3600 wavenumbers is due to loss of O-H groups due to incoporation of some of the cleaning media in the coal matrix. The negative absorption bands (1094-773) observed in the difference spectrum is due to loss of clay and silicate minerals as a result of the cleaning process.

Comparison of Figures 7 and 9 indicate that the floatation cleaning process gives more negative absorption values in the region 1094-773 cm^{-1} than that obtained from the heavy media cleaning. This diference shows that the floatation process gives a much better cleaning process.

Reference

1. E. L. Fuller, Jr., and N. R. Smyrl, "Diffuse Refectance Infrared Spectroscopy Studies of the Air Oxidation of Coal Surfaces, "Proc. Div. Fuel Chem. ACS, 691 (1988).

FIGURE 1 FTIR SPECTRUM OF AS RECEIVED COAL

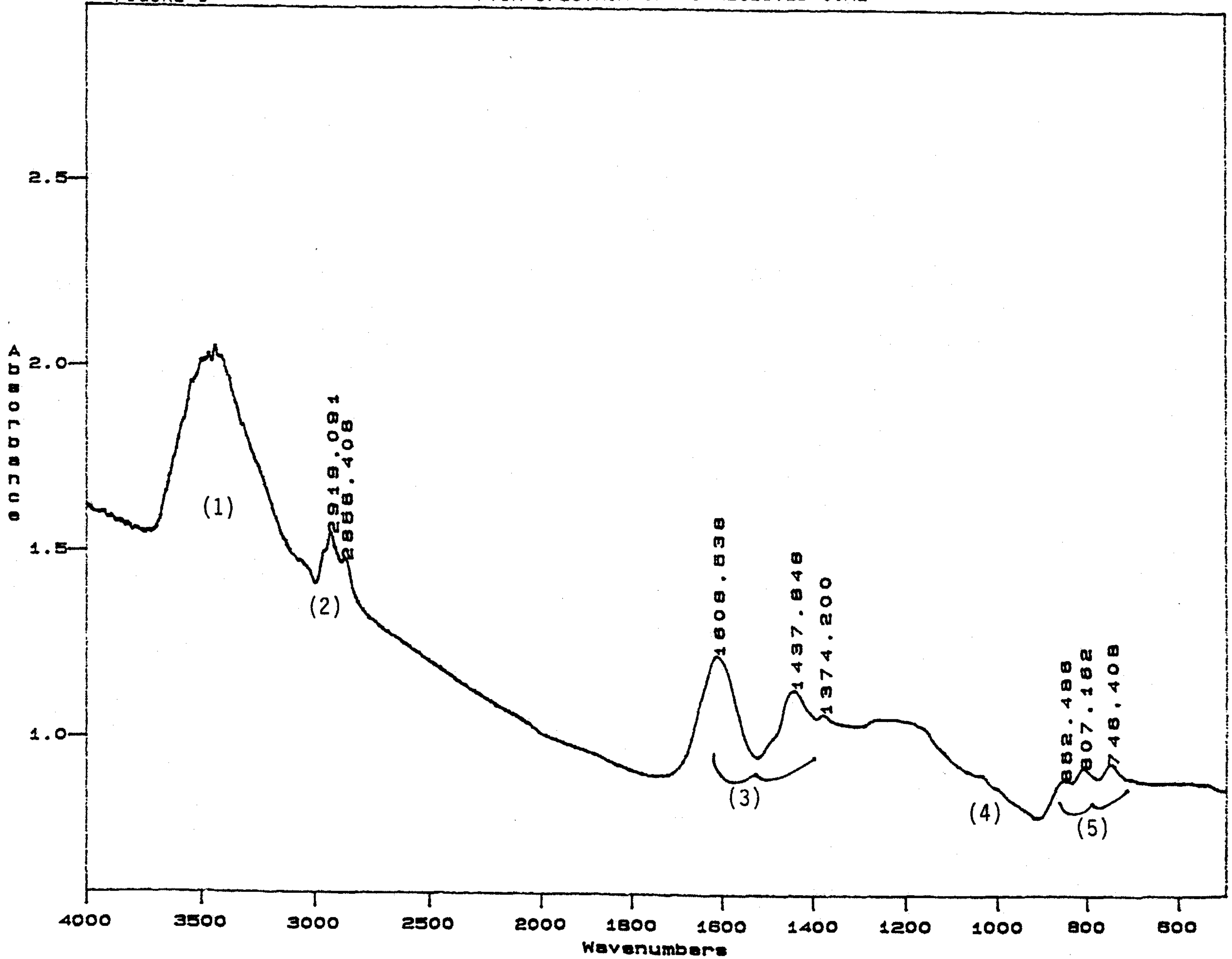


FIGURE 2

FTIR SPECTRUM OF DRY GROUND COAL

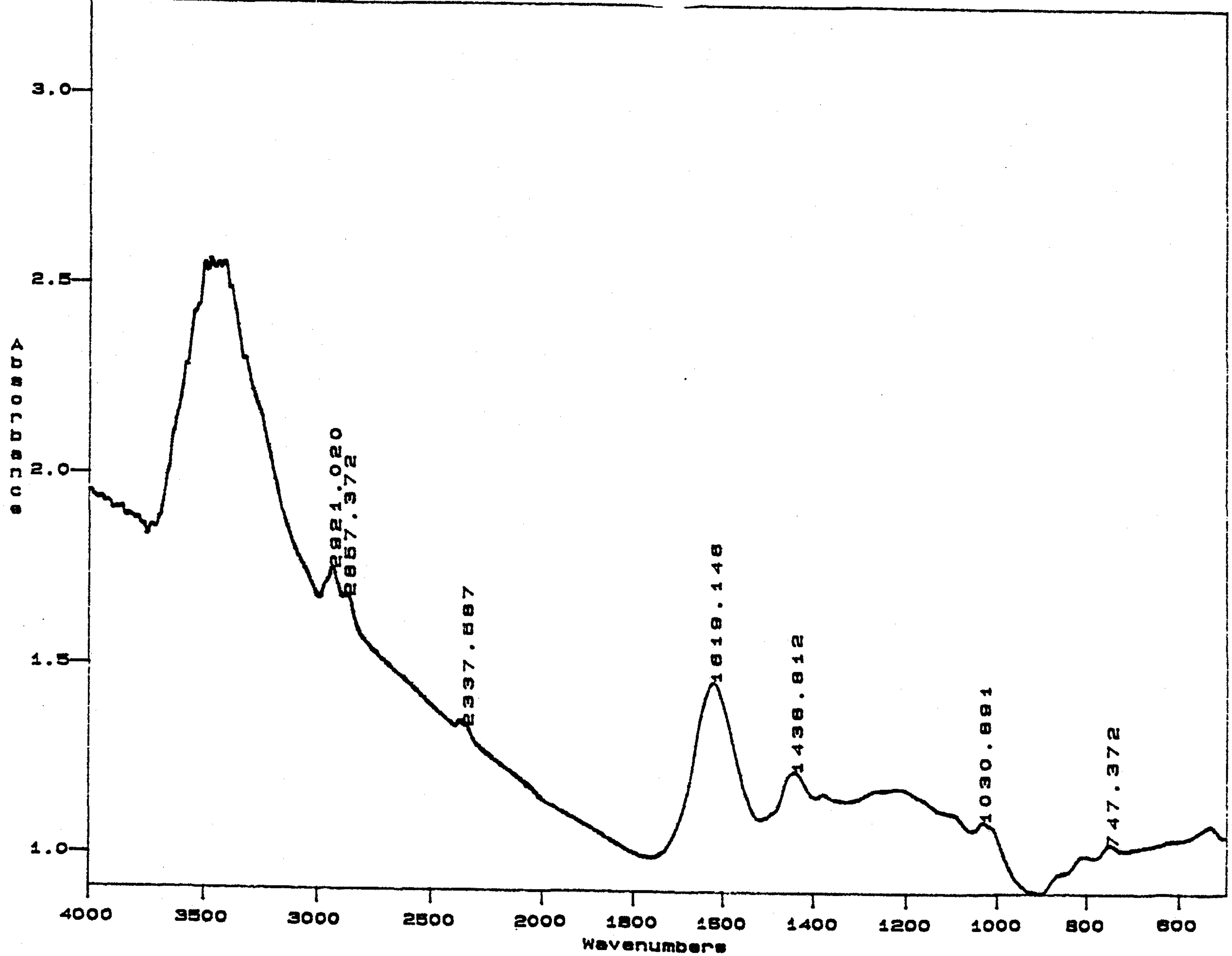


FIGURE 3 DIFFERENCE SPECTRUM OF DRY GROUND AND AS RECEIVED COAL

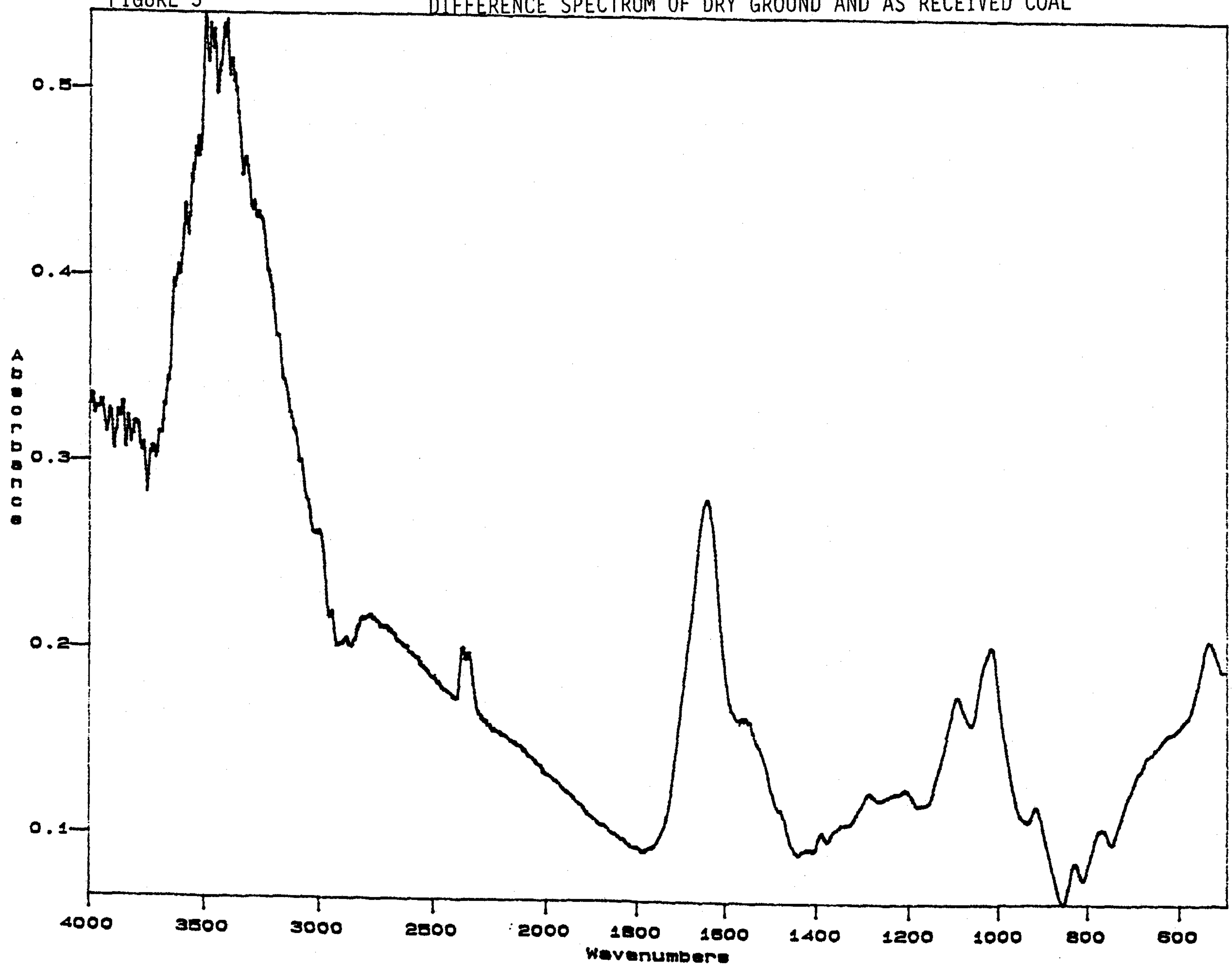


FIGURE 4 DIFFERENCE SPECTRUM OF WET GROUND AND AS RECEIVED COAL

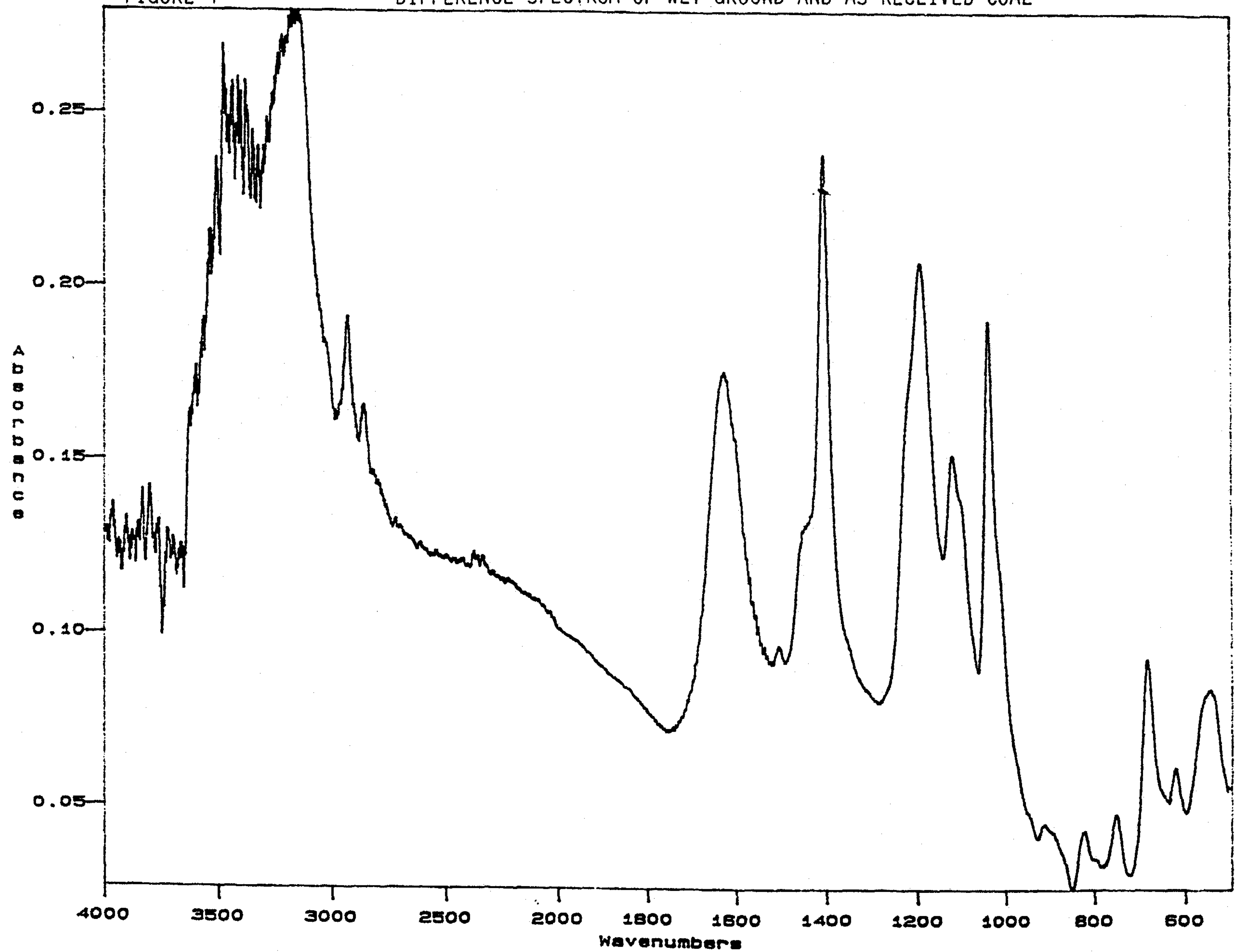


FIGURE 5 FTIR SPECTRUM OF FLOTATION CLEANED COAL

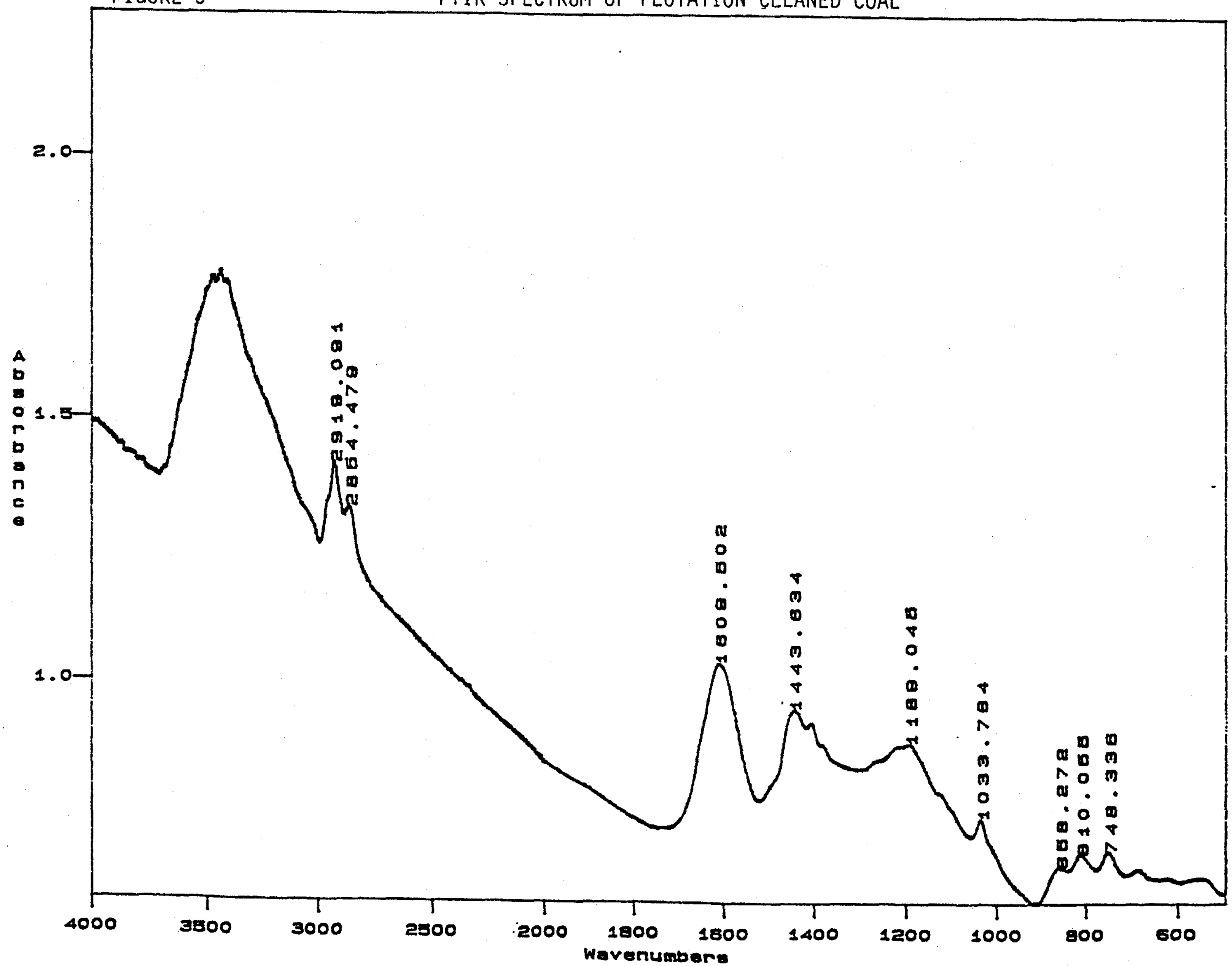


FIGURE 6 FTIR SPECTRUM OF UNCLEANED COAL

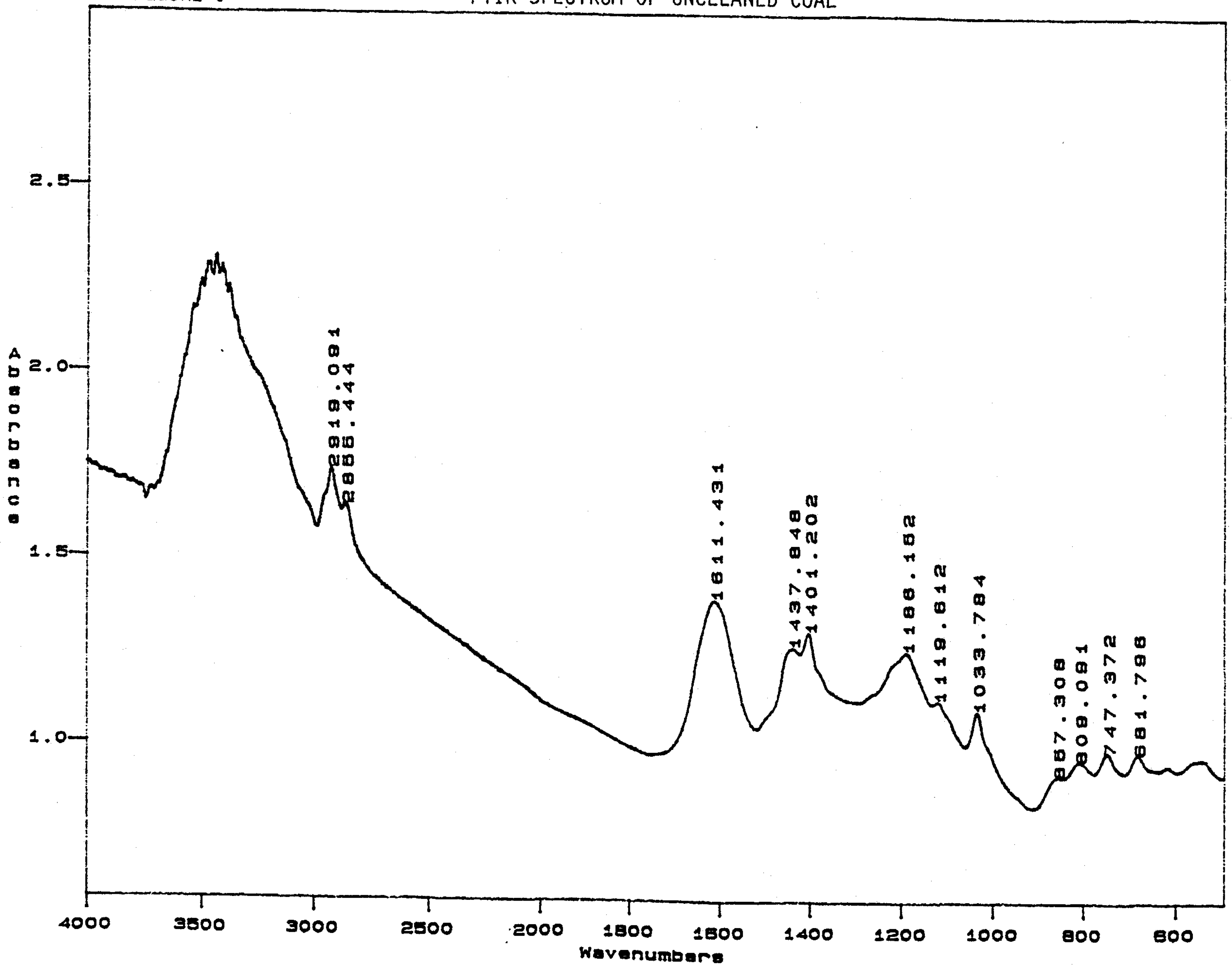


FIGURE 7 DIFFERENCE SPECTRUM OF UNCLEANED SLURRY AND FLOTATION CLEANED SLURRY

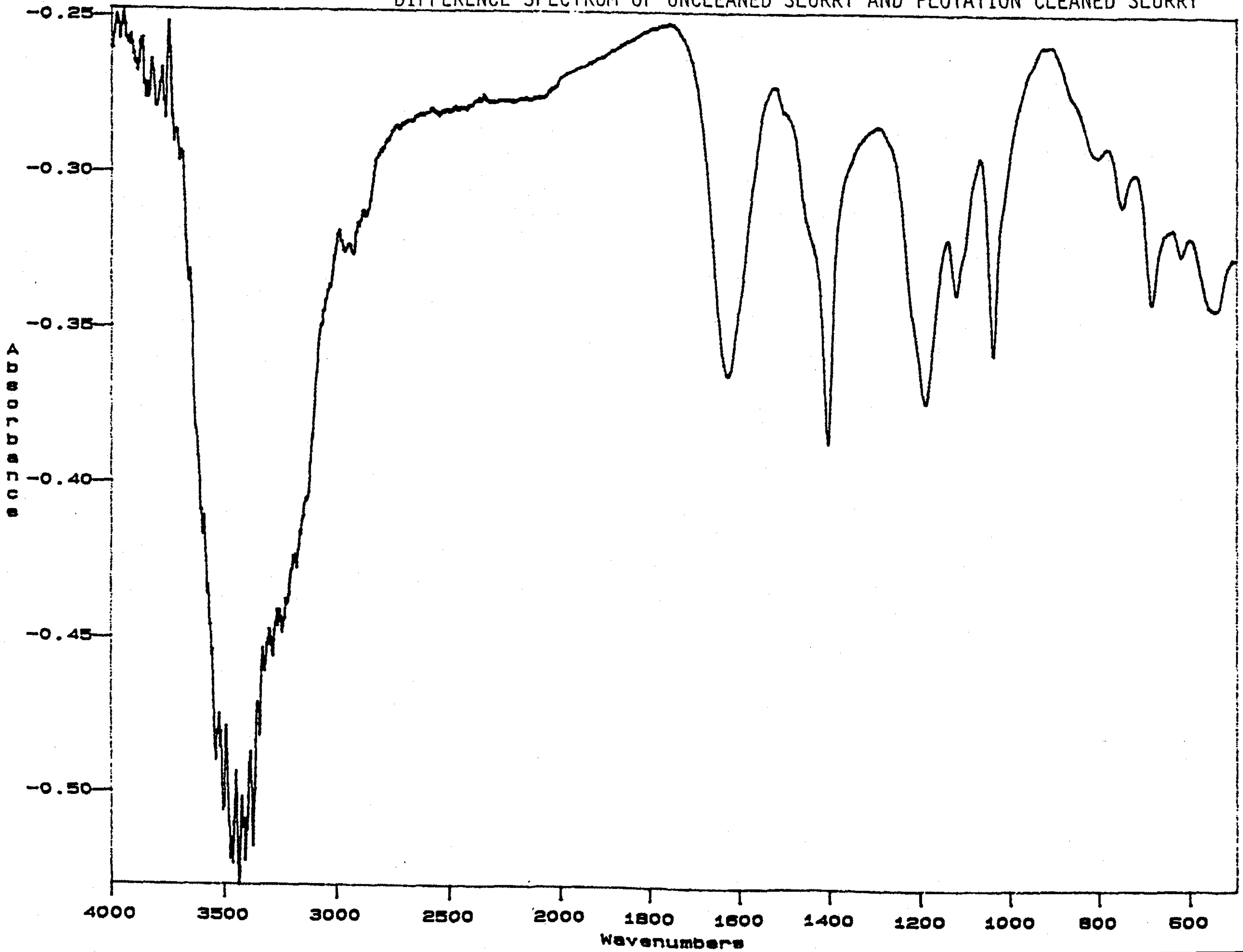


FIGURE 8 FTIR SPECTRUM OF HEAVY MEDIA CLEANED COAL

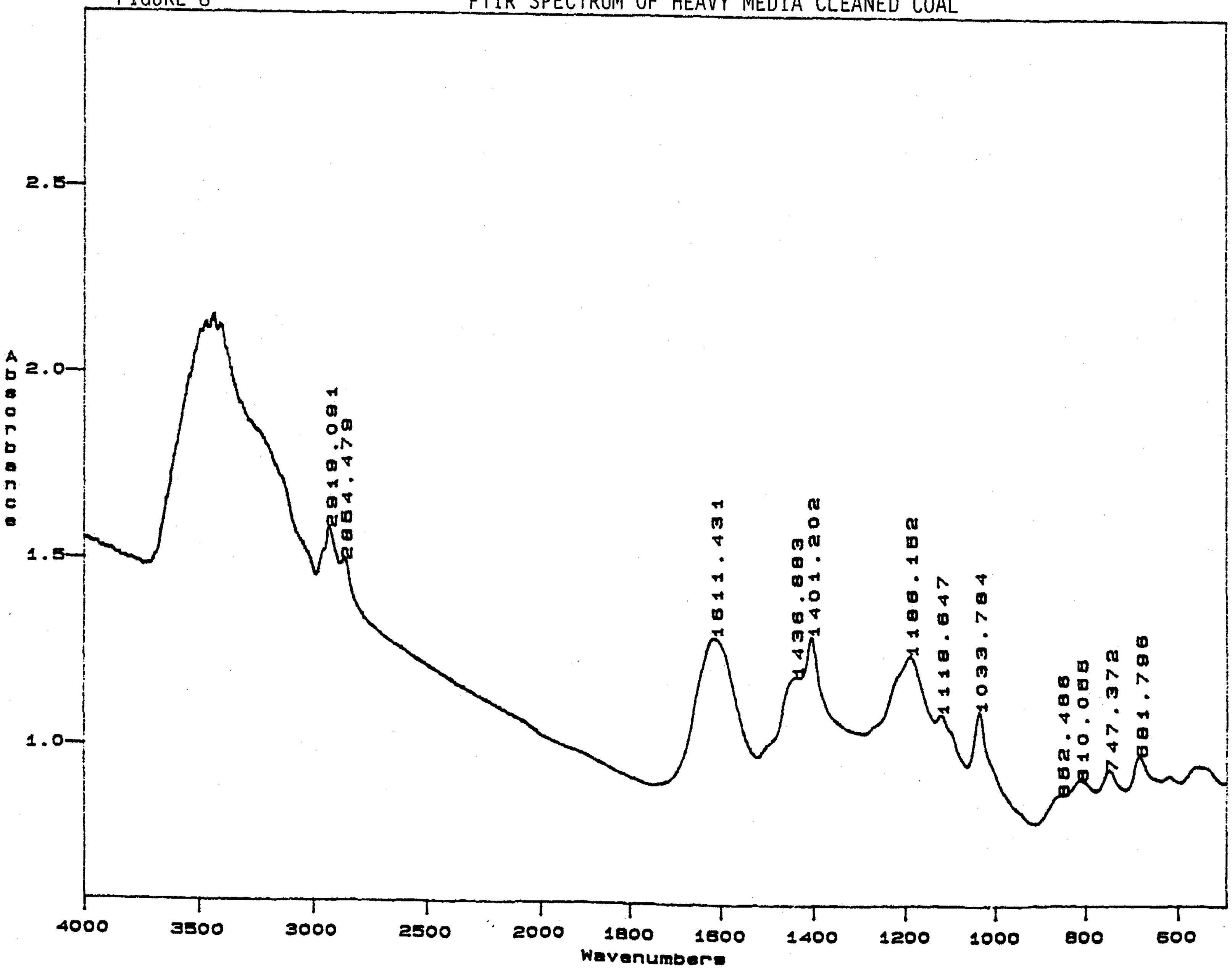
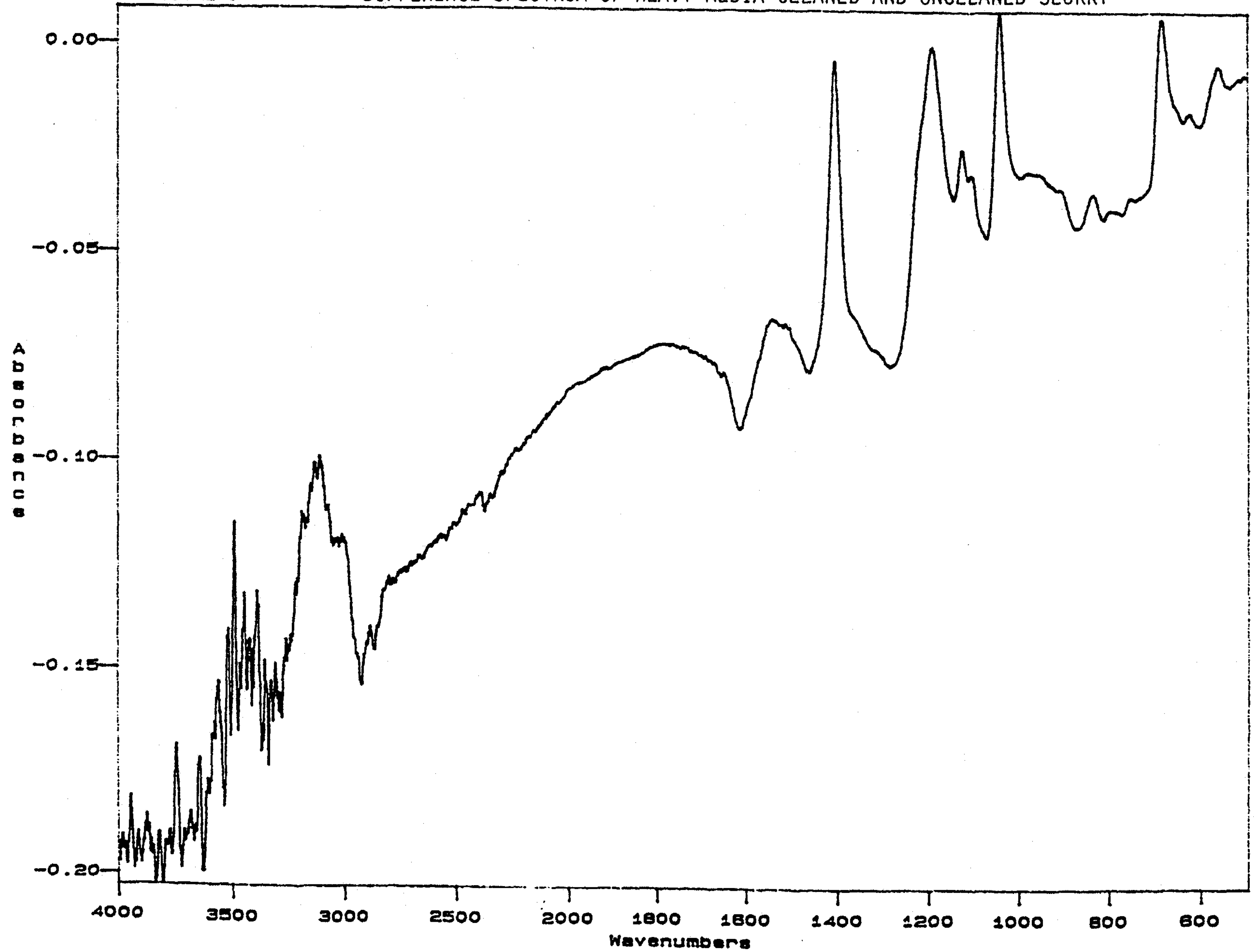


FIGURE 9 DIFFERENCE SPECTRUM OF HEAVY MEDIA CLEANED AND UNCLEANED SLURRY



AMAX Research & Development Center

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March 16, 1993

Dr. Frank Ohene
Department of Chemistry
Grambling State University
Grambling, Louisiana 71245

Dear Dr. Ohene:

Re: **Coal Water Slurries for DOE Contract No. DE-FG22-92MT92019**

The three coal water slurries which we prepared as part of our subcontract with Grambling were shipped to your attention by truck freight on March 5, 1993. The shipment also included a bucket of the parent Upper Elkhorn No. 3 (Taggart) crushed coal and a smaller sample of the pulverized parent coal. The slurries were packed in 55-gallon open-head steel drums lined with an epoxy paint. The three slurries were as follows:

Coal Cleaning - Uncleaned	45 Gallons
Coal Cleaning - Heavy Media	40 Gallons
Coal Cleaning - Froth Flotation	40 Gallons

Properties of each slurry are given in Table 1. We tried to keep the three slurries as much the same as possible with regard to coal content, reagent additions, and particle size distributions. A current Material Safety Data Sheet is enclosed for the slurries.

The Upper Elkhorn No. 3 parent coal we used was from the Wentz plant operated by the Westmoreland Coal Company in Virginia. As I described to you by telephone, the feed coal contained considerably less ash than we had expected. The Upper Elkhorn No. 3 coal we had received in the past from the Wentz plant usually ran about 4 percent ash, but this particular shipment contained only about 2.4 percent. Consequently, the difference in ash content between the slurries prepared from the cleaned coals and the slurry prepared from the parent coal was smaller than anticipated in our proposal.

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Table 1. Properties of Coal Water Slurry (CWS) Fuels Prepared for Grambling State University from Upper Elkhorn No. 3 Seam Coal

	Cleaning Procedure		
	None	Heavy Media	Froth Flotation
Slurry Composition, %			
Particulate Coal	62.43	62.39	62.41
Dispersant (A-23-M)	2.51	2.54	2.53
Stabilizer (Flocon 4800C)	0.025	0.025	0.025
Total Solids	65.96	65.95	65.96
Water	34.04	34.05	34.04
Slurry Density, lb/gallon	9.75	9.75	9.75
Slurry pH	7.8	7.9	7.7
Dry Solids Composition, %			
Volatile Matter	35.48	35.92	36.04
Ash	2.33	1.88	1.51
Fixed Carbon	62.19	62.2	62.45
Total Sulfur	1.20	1.19	1.21
Viscosity, Increasing Shear Rates, Centipoise			
100 s^{-1}	1,000	950	1,000
500 s^{-1}	995	900	1,015
1,000 s^{-1}	830	765	840
Viscosity, Decreasing Shear Rates After 3-Minute Hold at 1,000 s^{-1}, Centipoise			
100 s^{-1}	500	400	580
500 s^{-1}	445	440	525
1,000 s^{-1}	435	430	490
Particle Size Distribution, %			
Passing 300 μm	99.61	99.64	99.66
Passing 210 μm	97.93	97.83	98.18
Passing 150 μm	94.21	93.28	95.45
Passing 106 μm	89.05	88.28	91.01
Passing 75 μm	81.71	80.97	82.59
Passing 53 μm	74.67	75.72	75.54
Passing 45 μm	68.89	70.27	68.77
Passing 38 μm	66.51	66.71	68.48
Passing 30 μm	53.77	53.34	55.23
Passing 20 μm	43.49	42.30	42.09
Passing 15 μm	37.61	35.37	34.40
Passing 10 μm	29.78	27.60	27.24
Passing 8 μm	25.69	24.25	23.54
Passing 6 μm	21.26	20.06	19.69
Passing 4 μm	15.02	14.32	14.28
Passing 3 μm	11.63	11.21	11.14
Passing 2 μm	7.64	7.21	7.22
Passing 1 μm	2.35	2.15	2.10
Mass Mean Diameter (MMD), μm	43.9	44.9	42.3

The heavy-media cleaning was done in a stirred vessel with a 1.35 specific-gravity circulating magnetite slurry as the parting medium. The parent feed coal was precrushed to minus 1/2 inch and screened to remove the minus 14-mesh fines before the separation. The fines were discarded. The magnetite media was rinsed from the float coal with spray water on a vibrating screen. The float coal was subsequently ground to minus 48 mesh in a 4 x 4-foot ball mill and filtered.

The parent coal was also ball-mill ground to minus 48 mesh for the froth flotation, and the flotation was accomplished in a series of seven 1-cubic foot Denver mechanical aeration flotation machines using 0.34 pound diesel fuel and 0.51 pound methyl isobutyl carbinol (MIBC) per ton of feed coal as reagents. The flotation clean coal and a portion of the ball-mill ground parent coal were also filtered ahead of the final slurry preparation.

Table 2 presents mass balances for the two coal cleaning operations.

Table 2. Cleaning Wentz Plant Upper Elkhorn No. 3 Coal

	Parent Coal	Heavy Media Cleaning	Froth Flotation Cleaning
Product Coal (Dry Basis)			
Yield (Weight Distribution), %	100.0	68.5	95.4
MAF Coal Recovery, %	100.0	68.8	96.1
Ash, %	2.36	1.9	1.58
Total Sulfur, %	0.62		
Pyrite Sulfur, %	0.05		
HHV, Btu/lb	15,296		
Refuse (Dry Basis)			
Weight Distribution, %	0.0	31.5	4.6
Ash, %		3.4	17.1

The coal water slurries were prepared from the filter cakes. In each case, a portion of the filter cake was ground to minus 45 μm in a stirred ball mill and blended back with the original filter cake in order to achieve the desired particle size distribution for a highly loaded coal slurry. Henkel A-23-M naphthalene sulfonate was used to disperse the coal and reduce the viscosity of slurry, and Pfizer Flocon 4800C xanthan gum was added to prevent formation of a hard packed sediment cake during storage.

The slurry viscosities reported in Table 1 were obtained with a Fann bob and rotating cup viscometer. The shear stress/shear rate curves recorded for each slurry are attached. Our usual procedure is to record shear stress while increasing the shear rate from 0 to 1,000 s^{-1} (reciprocal seconds) over a 1-minute period. Rotation is then held constant for 3 minutes at 1,000 s^{-1} before being slowed back down to 0 over another 1-minute period. There is some noise in the these plots because of grit catching between the bob and the cup. (The alterative is to use an assembly with larger clearances but with less sensitivity.)

The particle size distributions reported in Table 1 were obtained by use of wire mesh sieves down to 38 μm (400 mesh) and by use of a SediGraph 5500 L sedimentation method in the subsieve range.

All three slurries appeared to exhibit good stability when shipped. However, deposition of a soft-pack sediment layer should be expected during storage. Therefore, we always recommend that users stir the slurries thoroughly before withdrawing samples and that they keep the drums covered whenever possible in order to minimize evaporative loss of the water. Please let us know if you encounter any problems handling these slurries.

Amax R&D is pleased to have been able to provide you with these slurries, and we are quite interested in hearing the results of your examination of their properties. Please let us know if you have any questions concerning coal cleaning and slurry preparation or if we can be of any other assistance to you or the project. I can be reached at (303) 273-7201 and Dr. Mahesh Jha can be reached at (303) 273-7284.

Sincerely yours,

Frank J. Smit
Senior Staff Engineer

FJS:wlk
Enclosures

cc: A. K. Bhasin
G. W. Hodge
M. C. Jha