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LABORATORY LEACHING OF COAL CONVERSION SOLID WASTES

Progress Report for November 1, 1979—January 31, 1980

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March 1, 1980

Work Performed Under Contract No. AC22-79PC20023

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U. S. DEPARTMENT OF ENERGY

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CONVERSION SOLID WASTES

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for the period November 1, 1979 to January 31, 1980

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March 1, 1980

Prepared for

THE U.S. DEPARTMENT OF ENERGY, PITTSBURGH ENERGY TECHNOLOGY CENTER

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Project # DE-AC22-79PC-20023

submitted by:

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Laboratory evaluations have been conducted on four solid waste samples from coal conversion processes. These samples include two liquefaction wastes and two gasification wastes. The two liquefaction wastes were obtained from two different runs at a facility using the H-Coal process and were solid bottoms from this process. These samples, for purposes of this report, are designated H-Coal #3 and H-Coal #4.

One of the gasification wastes was obtained from the cyclone fly ash collector of an existing Chapman gasifier. The other gasification waste is bottom ash resulting from the gasification of lignite coal at the Grand Forks Energy Technology Center.

All samples were sieved using number 20, 40, 60, 100, and 200 mesh U.S. Standard Sieves. Sieving was conducted by placing the samples on a "ro-tap" for about 30 minutes each "sieving session". Many sieving sessions were found necessary to obtain sufficient quantities of the smaller sized fractions.

Using this approach, five different particle size sub-samples were obtained for each waste sample. Both the Chapman fly ash and the GFETC bottom ash were subjected to the above procedure without altering the nature of the sample (ie.. without grinding). The natural particle size distribution of the Grand Forks bottom ash material is shown in Figures 1 and 2, and table 1. Particle size distribution for the Chapman fly ash was presented in our earlier reports.

The H-Coal samples were crushed prior to the sieving procedure because of the large sizes of the "chunks" of waste. The majority of these irregularly shaped chunks were larger than three inches by three inches. The H-Coal samples were first crushed in a jaw crusher, and then in a sample pulverizer located in the Pitt Mining Engineering Laboratory. The result was a sample which varied in particle size from greater than 20 mesh, to smaller than 200 mesh. The procedure resulted in five sample sizes of 20-40 mesh, 40-60mesh, 60-100 mesh, 100-200 mesh, and finer than 200 mesh.

The two H-Coal samples and the Chapman fly ash have been subjected to the ASTM-A, EPA-"EP", and the University of Pittsburgh leaching tests. The Grand Forks bottom ash has been subjected to the ASTM-A and EPA-"EP" tests, and will be subjected to the University of Pittsburgh procedure during the next quarter. All leaching tests have been performed in duplicate on all five particle size fractions of each sample. Thus, one solid waste sample has generated 5x2x3 or 30 samples for subsequent heavy metal analysis.

Samples of solid wastes have been submitted to the PETC-TPO for spark source mass spec analysis of the heavy metal fraction. Preliminary indications are that delays exist in getting the samples analyzed.

The ASTM-A leaching test is a water shake extraction procedure. A sample of 250 grams of each particle size fraction is placed into one-half gallon glass bottles. A volume of distilled deionized water equal in milliliters to four times the weight in grams of the sample used was then added to each container. In these experiments, this volume was equal to 1000 ml. The containers were then closed and placed on an Eberbach reciprocating platform shaker which was operated at 70 one-inch strokes per minute. After 48 hours, the containers were removed from the shaker, and filtered through 0.45 micron filter paper using a special Millipore pressure filter apparatus. Conductivity and pH values were measured immediately. A 250 ml sample was placed in a glass bottle for AMES testing. The remaining sample was acidified to a pH less than 2.0 with concentrated nitric acid and stored in glass at 4°C for subsequent metal analysis. Storage in this manner for metal analysis is an EPA acceptable technique.

The Chapman fly ash samples were found clear after filtering.

The H-Coal #3 samples were pale yellow in color, but with no observable precipitate. The H-Coal #4 leachates were a darker yellow in color, and a rust colored precipitate (most likely iron hydroxide) formed upon acidification and standing of the samples. The Grand Forks bottom ash was clear after initial filtering, but turned slightly turbid with a yellow precipitate after standing and acidification.

The EPA-"EP" leaching test is an acid shake extraction procedure with controlled pH. A sample size of 60 grams of each particle size fraction was placed into a series of 2000 ml glass beakers. A weight of distilled deionized water equal to sixteen times the weight of solid material was added to each beaker. In our case, this represented a volume of 960 ml of water. A Phipps & Bird "jar test" apparatus was used for gang stirring for this procedure.

This extractor was operated at a speed which prevented stratification of the sample, and allowed all sample surfaces to be continuously brought into contact with the extraction fluid. After agitation begins, the pH of the solution is manually maintained at 5.0 ± 0.2 with 0.5N acetic acid for 24 hours. If four milli-liters of acid per gram of solid is added to a beaker, the 24 hour extraction is completed without adding any additional acid. In our tests, the maximum amount of acid added to each beaker was 240 ml. After 24 hours,

each sample was filtered through 0.45 micron millipore filter paper via a pressure filter. The volume of the resulting liquid was then adjusted with a weight of distilled deionized water so that the volume is twenty times the weight of solid material added to the extractor, in our case, 1200ml.

Conductivity and pH values were again measured immediately and a 250 ml sample was placed in a glass bottle for AMES testing. The remaining sample was acidified to a pH of less than 2.0 with concentrated nitric acid and stored at 4°C. The GFETC samples finer than 200 mesh, 100 to 200 mesh, and 60 to 100 mesh were the only samples that used the maximum amount of acetic acid within the 24 hour extraction period.

The "University of Pittsburgh leaching test" is also an acid shake extraction procedure with a controlled pH. The basic procedure and apparatus are similar to the EPA-"EP" procedure with the exception that the pH is manually maintained at 1.6 to 2.0 with 1:1 (ie..50% concentration) nitric acid solution, and there is no limit to the amount of acid that can be added. The Chapman fly ash and H-Coal #3 material were clear while the H-Coal #4 sample was pale yellow with no precipitate after filtering and acidification after use of this procedure.

Conductivity and pH data obtained from the ASTM-A, EPA-"EP", and University of Pittsburgh leaching tests are shown for the different particle sizes of Chapman, H-Coal, and GFETC materials in tables 2,3,4, and 5, and figures 3,4, 5, and 6.

Figure 1

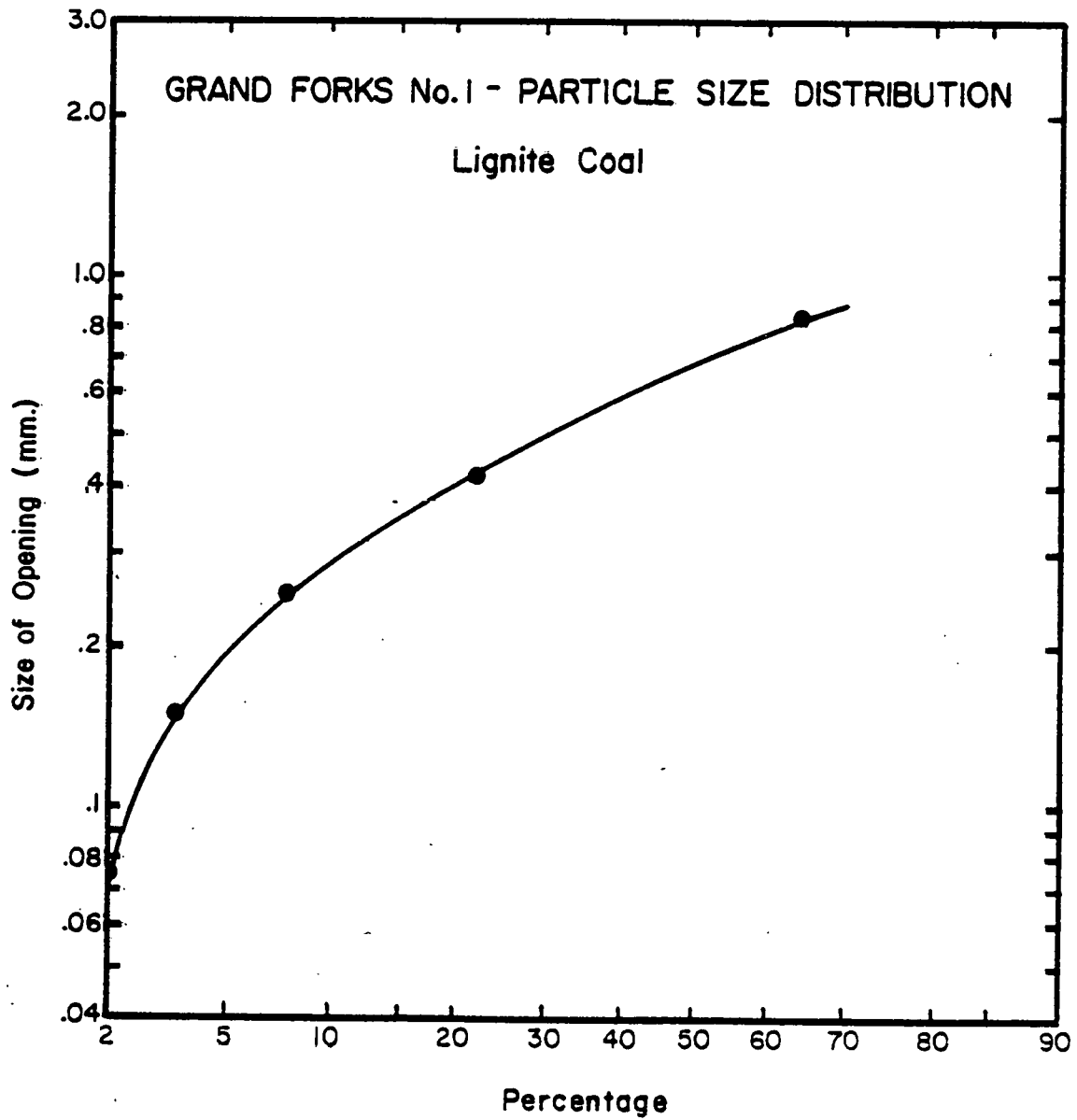
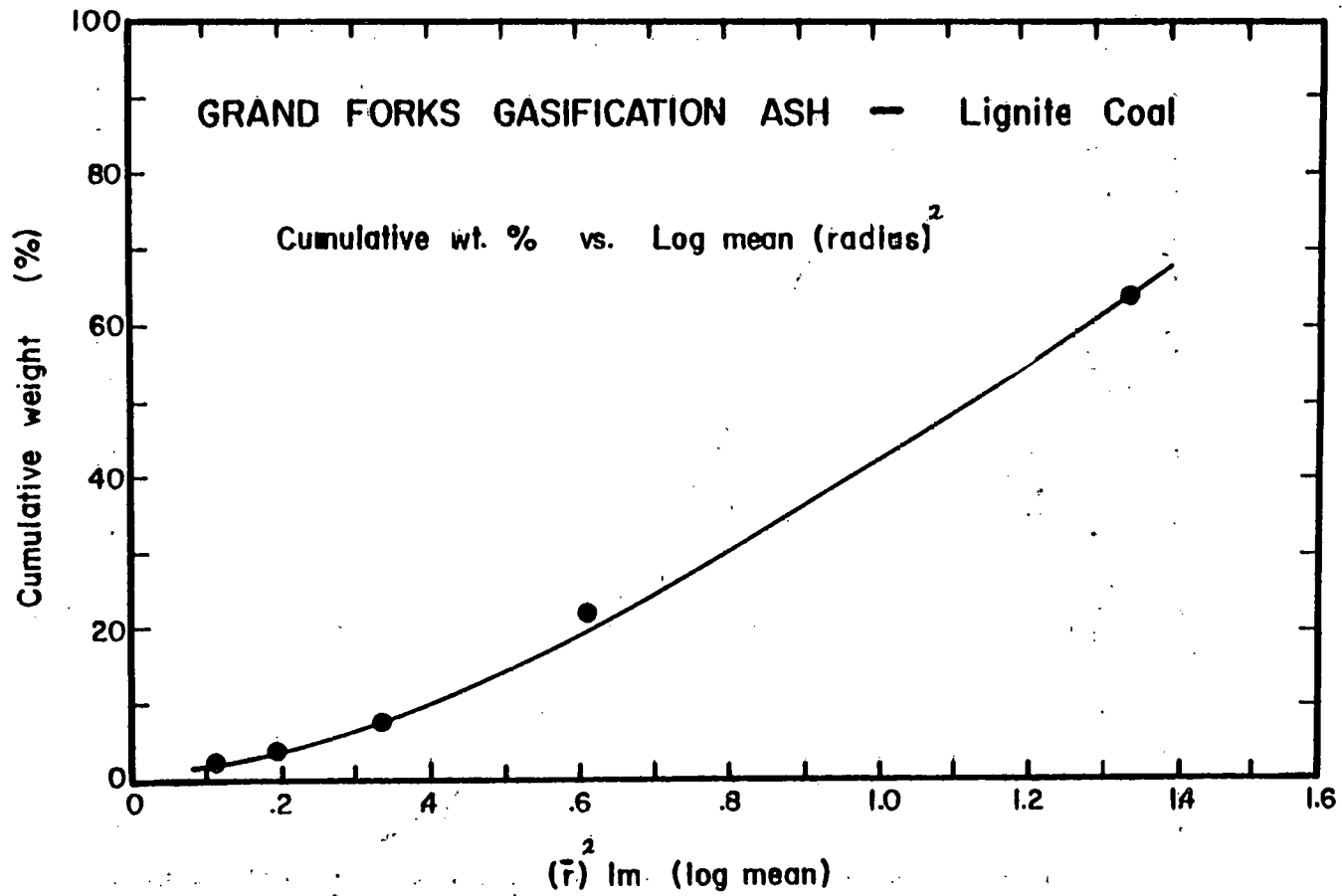
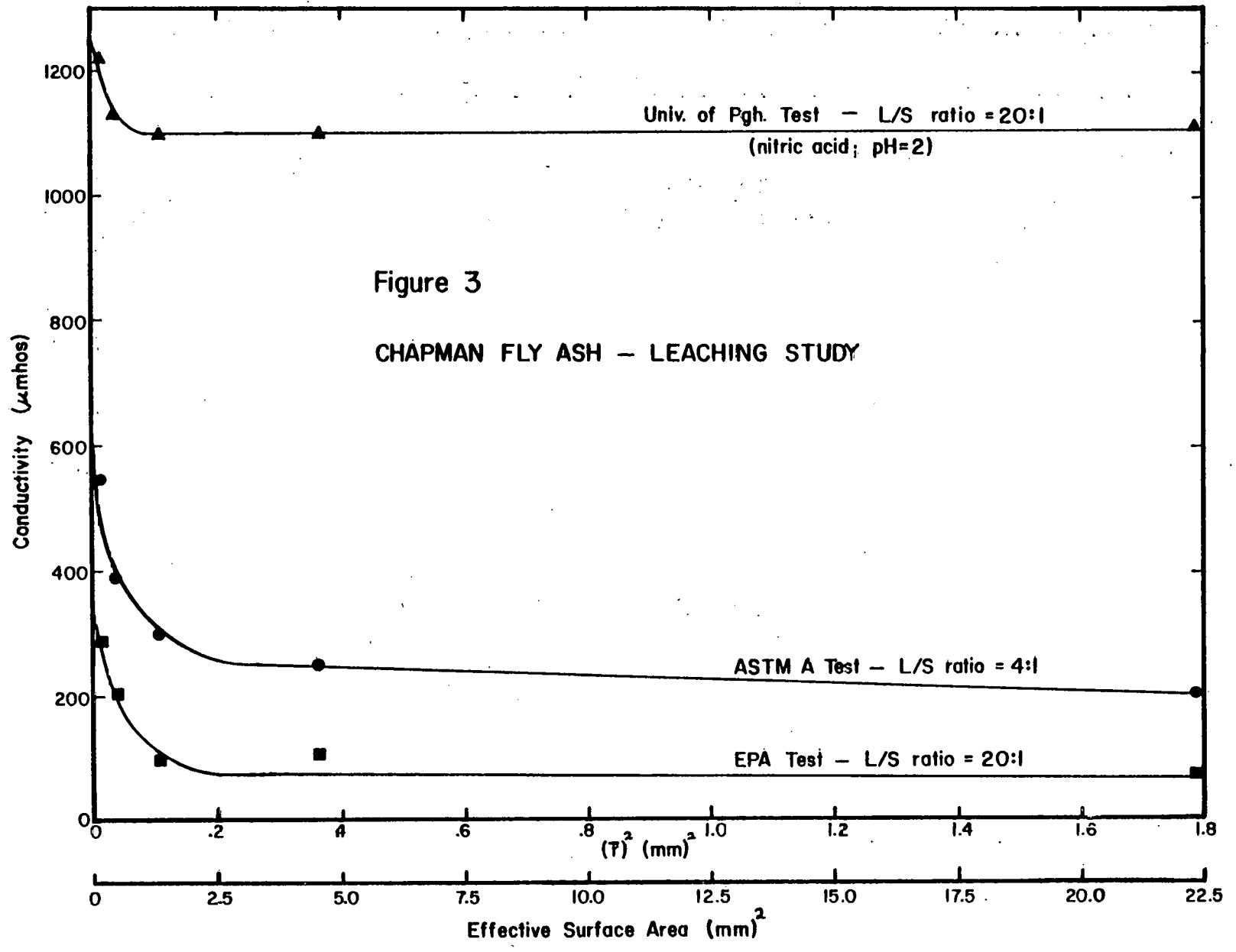
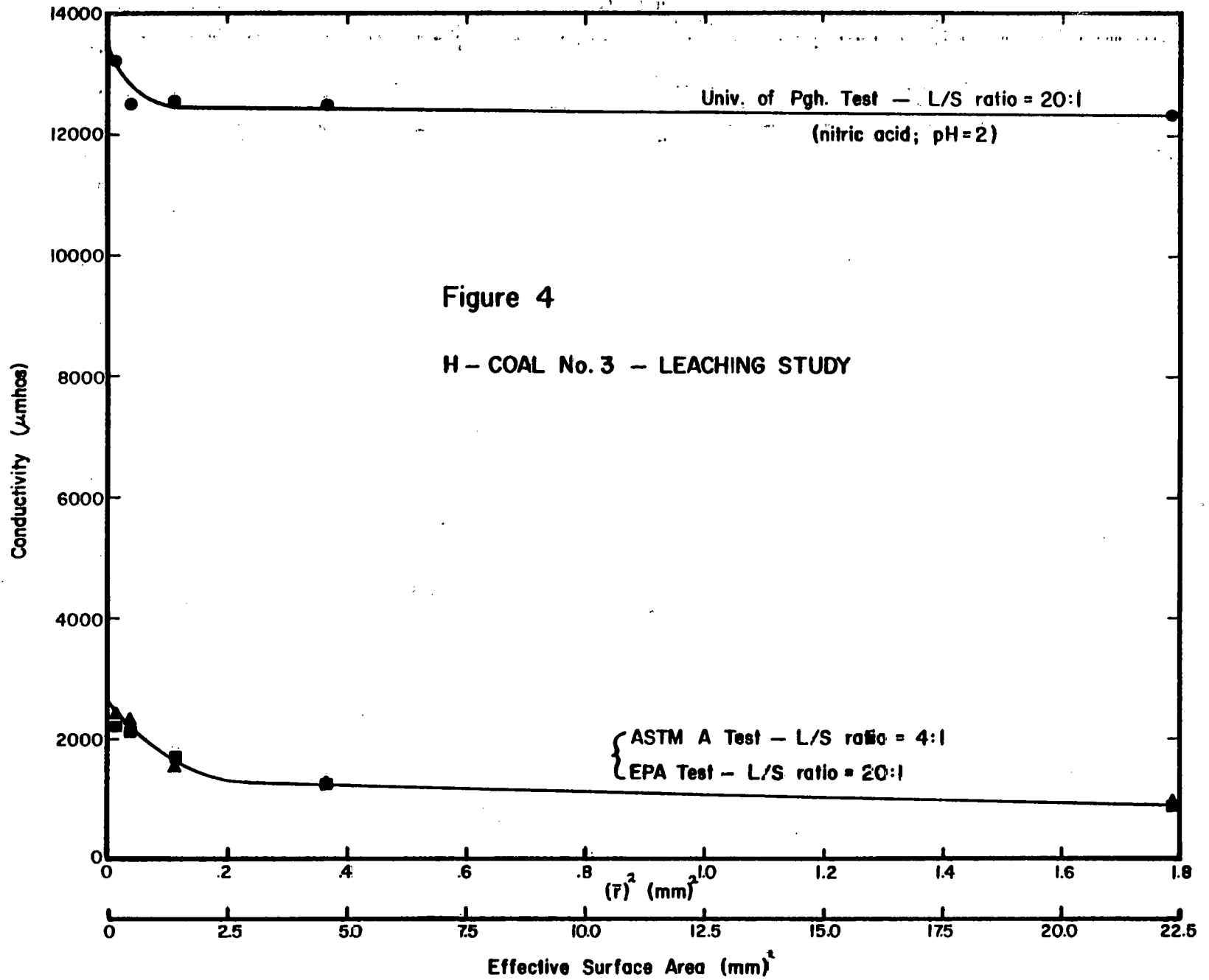


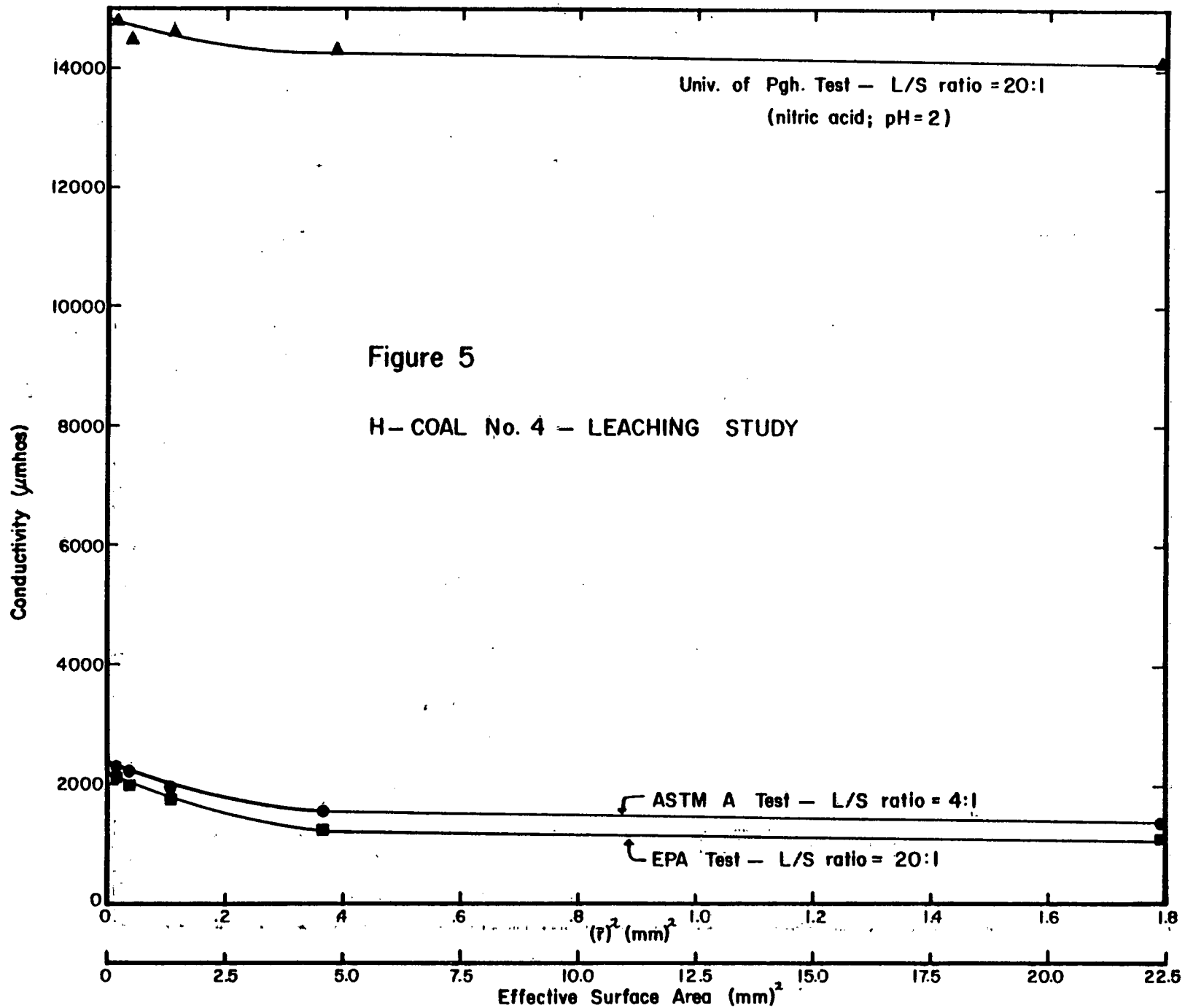
Figure 2





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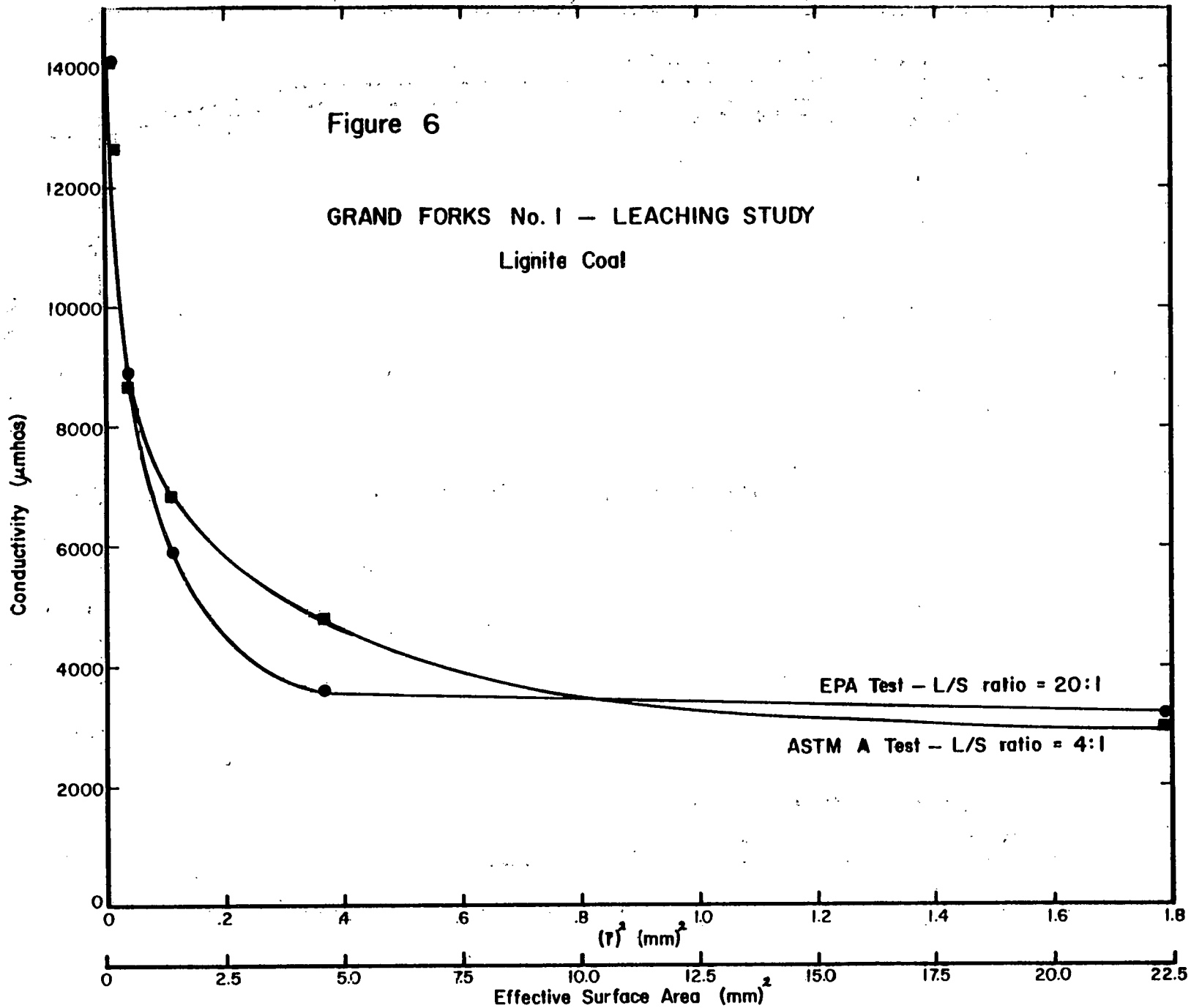


TABLE 1

Grand Forks #1 - Particle Size Distribution

U.S. Standard Sieve Designation Number	Size of Opening	Cumulative Weight (g)	Cumulative Weight (%)	\bar{r}^2 (mm ²)
>200	<0.075	120	2.0	0.0116
100	0.150	213	3.5	0.0384
60	0.250	466	7.6	0.1070
40	0.420	1,349	22.0	0.3670
20	0.841	3,950	64.5	1.7900
< 20	>0.841	6,121	100.0	-

\bar{r} = log mean radius of opening

TABLE 2
Chapman Fly Ash

Particle Size U.S. Standard Sieve No.	ASTM A		EPA		Univ. of Pgh.	
	pH	Conductivity (umhos)	pH	Conductivity (umhos)	pH	Conductivity (umhos)
20-40	7.05	200	5.22	68	1.78	11,100
40-60	7.17	245	5.18	105	1.83	11,000
60-100	7.23	295	5.11	95	1.88	11,000
100-200	7.28	385	5.05	200	1.82	11,300
>200	7.30	540	4.94	285	1.76	12,200

TABLE 3
H - Coal #3

Particle Size (U.S. Standard Sieve No.)	ASTM A		EPA		Univ. of Pgh.	
	pH	Conductivity (umhos)	pH	Conductivity (umhos)	pH	Conductivity (umhos)
Not Crushed	9.31	58				
20-40	11.03	960	4.96	850	1.72	12,300
40-60	11.22	1260	4.90	1260	1.74	12,500
60-100	11.33	1530	4.88	1710	1.76	12,600
100-200	11.48	2300	4.87	2100	1.77	12,500
>200	11.49	2400	4.91	2200	1.71	13,200

TABLE 4
H - Coal #4

Particle Size (U.S. Standard Sieve No.)	ASTM A		EPA		Univ. of Pgh.	
	pH	Conductivity (umhos)	pH	Conductivity (umhos)	pH	Conductivity (umhos)
Not Crushed	9.54	55				
20-40	11.08	1390	4.98	1050	1.74	14,200
40-60	11.19	1560	4.96	1225	1.76	14,400
60-100	11.30	1950	4.93	1740	1.72	14,700
100-200	11.31	2200	4.91	1950	1.72	14,500
>200	11.41	2300	4.93	2100	1.76	14,800

TABLE 5

Grand Forks #1

Particle Size (U.S. Standard Sieve No.)	ASTM A		EPA		Univ. of Pgh.	
	pH	Conductivity (umhos)	pH	Conductivity (umhos)	pH	Conductivity (umhos)
20-40	9.62	3200	4.97	3000		
40-60	9.25	3700	4.98	4800		
60-100	9.01	5900	5.44	6800		
100-200	8.80	8900	5.66	8700		
>200	8.58	14,200	5.76	12,600		

Observations and Conclusions:

The purpose of the sieving was to obtain the natural size distribution of particles from the solid waste sample. This was done for all samples with the exception of the H-Coal material which, as indicated above, was received as large shiny black particles with sizes on the order of several inches in diameter. As shown on Figure 1, the smallest size utilized of the Grand Forks waste material comprised about 2% of the total mass. Thus, in order to collect 1 pound of this finest fraction, it is necessary to shake and process about 50 pounds of as-received GFETC solid wastes.

In all cases, the smaller particle sizes released more material into the leaching media than did the larger particles, the an apparent "breakpoint" at particle diameters of about 0.025 inches; a value comparable to observations reported in the last quarter with results from the Chapman gasifier facility. It should be noted however, that the relative magnitude of the increase of leachate parameters for smaller particles over the larger particles is consistently not as great for the H-Coal samples as it is for the other materials under study. We attribute this to the fact that the smaller H-Coal particles were manufactured by us via

special grinding techniques and thus may be expected to possess more uniform leaching properties than particles from a "natural distribution".

Examination of the plots of particle size vs. cumulative weight percent shows that particles with sizes less than the defined "break-point" (or about 0.025 inches diameter), represent about 40% by weight of the total ash matrix. Accordingly, in our opinion, serious consideration should be given to "fixation" techniques, or agglomeration techniques for the ultimate disposal of coal conversion solid waste residuals.