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U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Standard Reference Materials:

Sampling, Materials Handling, Processing, and Packaging of NBS Sulfur in Coal Standard Reference Materials

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Standard Reference Materials:

Sampling, Materials Handling, Processing, and Packaging of NBS Sulfur in Coal Standard Reference Materials

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PREFACE

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards are "well-characterized materials, produced in quantity, that calibrate a measurement system to assure compatibility of measurement in the Nation." SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. In many industries, traceability of their quality control process to the national measurement system is carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists it is, therefore, of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values and of procedures used at NBS in producing SRM's. An NBS series of papers, of which this publication is a member, called the NBS Special Publication - 260 Series is reserved for this purpose.

This 260 Series is dedicated to the dissemination of information on all phases of the preparation of NBS Sulfur in Coal SRM's. In general, much more detail will be found in this 260 than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity of processes employed, and to learn details of methods utilized for work entailing the greatest care. It is also hoped that this 260 will provide sufficient additional information not found on the certificate so that new applications of these SRM's may be sought and found.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth, will receive prompt attention from:

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Washington, DC 20234

George A. Uriano, Chief
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Catalog of NBS Standard Reference Materials (1981-83 edition), R. W. Seward, ed., NBS Spec. Publ. 260 (November 1981).

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Gills, T. E., Seward, R. W., Collins, R. J., and Webster, W. C., Standard Reference Materials: Sampling, Materials Handling, Processing, and Packaging of NBS Sulfur in Coal Standard Reference Materials, 2682, 2683, 2684, and 2685, NBS Spec. Publ. 260-84 (In Press).

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CONTENTS

LIST OF TABLES

LIST OF FIGURES

SAMPLING, MATERIALS HANDLING, PROCESSING, AND PACKAGING OF NBS SULFUR IN COAL STANDARD REFERENCE MATERIALS

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This publication describes in detail the performance of a grant given to Valley Forge Laboratories, Inc., by the National Bureau of Standards, to obtain and prepare four standard reference coals, with nominal sulfur contents of 0.5, 2.0, 3.0, and 4.5 percent to be issued as SRM's 2682, 2683, 2684, and 2685, respectively. All activities pertaining to the sampling, preparation, packaging, and homogeneity testing of the coal SRM's are documented in this report, including a separate description of each of the four coal sampling activities. Protocols used in the development of these Standard Reference Materials are similar to those used in other NBS SRM preparation procedures to ensure that materials used for SRM's have the highest possible homogeneity and stability.

Key words: bituminous coal; blending; drying; hammermilling; homogeneity; processing; sampling; screening; Standard Reference Materials; sub-bituminous; sulfur.

DISCLAIMER

1. Introduction

Valley Forge Laboratories (VFL) and Webster and Associates furnished technical and logistical services as part of a grant from the National Bureau of Standards (NBS) to provide for the collection and preparation of coal materials for subsequent use by NBS as Standard Reference Materials (SRM's). The purpose of this grant was to obtain, from well-documented sources, at least 1,000 pounds each of four separate coals having nominal sulfur levels of 0.5, 2.0, 3.0, and 4.5 percent. The collected material was crushed and ground to pass a 60 mesh sieve. These four coal materials were intended for certification and distribution by NBS as Coal SRM's for use in standardization of laboratory facilities and testing equipment for the analysis of sulfur content, ash content, and calorific value. The four coals described herein have been certified and issued as SRM's 2682, 2683, 2684, and 2685. Moisture content is reported for each coal, but is not certified.

Of the properties to be certified by NBS, the most important is the sulfur content, particularly because of its impact in the enforcement of environmental regulations. The ash content, moisture, and calorific value are the three additional properties commonly used in assessing coal quality and assigning sale value in the marketplace.

In order to adequately describe materials and experimental procedures discussed in this work, it was occasionally necessary to identify companies or commercial products by manufacturer's name or label. In no instance does such identification imply endorsement by NBS of that particular product or equipment as being the best available for that purpose.

This program for providing and preparing Coal SRM's involved the following activities for each of the coals sampled:

a. Selection of coal sampling locations;

b. Collection and handling of coals;

c. Processing of coals;

d. Packaging of coals; and

e. Sample preparation/homogeneity testing.

A schematic diagram depicting the activities described above is shown in figure 1. All of these activities were completed for each coal before the collection began for the next coal, starting with the coal with lowest sulfur content and progressing in order of increasing sulfur content.

2. Selection of Coal Sampling Locations

From the beginning of the program, the selection of the location and type of coals was recognized as one of the critical factors influencing the attainment of the desired sulfur level and required homogeneity for each of the candidate coal materials. The original plan was to obtain samples from coalburning utility power plants that use coal from a single documented source. The intent was to utilize available published information from government energy agencies pertaining to target sulfur levels of coals purchased for use in electric utility plants. It would then be possible to take advantage of the averaging effect of coal processing equipment at selected utility plants to obtain coal samples with known uniform properties, particularly sulfur level.

After reviewing pertinent publications on sulfur levels of utility coals and identifying candidate locations for each of the four desired sulfur levels, it was decided that sampling at utility locations did not represent the best approach because it was desired to carefully document the coal sources. Coal from a single mine can be obtained from several different locations (possibly involving more than one coal seam) within the mine itself and coal from a preparation plant may be obtained from several mines. Furthermore, the sulfur content of the raw coal from a single mine can vary significantly from one area to another within the same mine and this coal can then be blended at the preparation plant to achieve a desired sulfur level. This is particularly so when considering that some of the larger mines extend over an area of several square miles.

It is usually possible to accurately identify the location and seam of a particular coal coming into a preparation plant and to separate this coal source from others being handled by the same plant. But, once the coal has been prepared and shipped to a utility plant from a coal preparation facility, it already is a blend of coals, each having differences in sulfur content and other properties. In addition, pressures from regulatory agencies to reduce or maintain current sulfur oxide emission levels result in frequent blending of coals at utility power plants, even when published reports indicate that coal from a single source is being used.

Therefore, it was decided that all coals would be obtained from a single mine and/or preparation plant, in which the coal came from one documented source having a known sulfur content within the desired range.

To select prospective mine locations for coal sampling, the coal sources feeding the target utility plants chosen in the initial screening were investigated. The investigation sought to determine such information as the type of mine (surface or deep); the number of locations and seams being mined at that time; the average sulfur content of the coal (before and after washing); the variation in sulfur content of coal from different locations within the same mine; the moisture content of the coal; the volume of coal prepared and shipped; the number of customers and their sulfur content requirements; and existing sampling systems for the coal.

The sampling locations selected were:

Coal Company	Mine	Location	Coal Type	Nominal Sulfur Content (Percent)
Amax Coal Company	Belle Ayr	Gillette, WY	Sub-bituminous	0.5
Consolidation Coal Co.	Humphrey	Osage, WV	Bituminous	2.0
Amax Coal Company	Delta	Marion, IL	Bituminous	3.0
Consolidation Coal Co.	McElroy	Captina, WV	Bituminous	4.5

One final consideration was the type of sample and the sampling technique to be employed. Mine face sampling was considered, since it would be taken from a specific seam location in the mine where, presumably, the sulfur content would be known and would be quite consistent. However, obtaining a mine face or channel sample is a long, involved, and very time-consuming procedure that can only be performed by a certified specialist.

Because most commercial coals, particularly bituminous coals, are cleaned before use, it was felt that mine face or channel samples would be representative of most coals being tested in the marketplace and would contain some impurities that would be removed in the preparation plant. It was also apparent that a considerable amount of sulfur analysis data was available from washed coal samples coming out of the preparation plant, but less sulfur analysis data was available from raw coal samples taken from specific mine face locations at the sampling sites. Therefore, it was decided that all coal samples, except for the 0.5 percent coal (which required no washing), would be taken from coal preparation plants at the selected sampling locations. Sampling locations selected were based on facilities where preparation plants accepted coal from only one mine and would be able to correlate a specific location within the mine with a coal sample taken from the preparation plant during a particular time period.

3. Collection and Handling of Coals

Discussions with mine or preparation plant supervisors at each sampling location focused on resolution of logistical considerations, such as:

a. Number of locations and identifiable seams where coal is presently being mined;

b. Average sulfur content of the raw and prepared coal;

c. Consistency of the coal being supplied recently;

d. Variation in sulfur content of coal from different locations or seams within the mine;

e. Ability to obtain a 1-ton coal sample using the mine's conveying and sampling system;

f. Ability to perform sulfur analyses using available laboratory facilities at the mine;

g. Availability of personnel, equipment, and materials (such as 55-gallon drums and plastic liners) to assist in the coal sampling; and

h. Time period during which the sampling could be scheduled.

At each location, the procedures used to obtain the coal samples were essentially similar. An existing automatic coal sampling system, which obtained a full-stream conveyor belt cut, was utilized. The belt sample was passed through a crusher and one or more sample cutters and the reject material from the sample cutter (that is, the material that was not included in the coal company's composite sample) was collected at pre-determined time intervals over a several hour time period. All coal was collected in plastic-lined 55-gallon steel drums, which were sealed once the drums were filled. A 5000-to-6000 gram composite sample was also collected at random intervals during the filling of each drum for subsequent analysis of sulfur content.

The time period of sampling was selected to correspond with a particular quantity of coal, representing a desired total lot size for sampling. The lot size varied depending on the location, type, and capacity of the sampling and coal loading facilities. For example, in the case of the 0.5 percent coal, a time period for sampling was chosen to approximate the amount of coal to fill a silo for later loading of a unit train, while the sampling period for the 2.0 percent coal corresponded with the time allotted for loading of four coal barges.

A discussion of the mining and preparation plant operations at each sampling location, together with details of the sampling and handling procedures employed at each of these locations, is provided in separate Appendixes to this report. Appendix A discusses the sampling of the 0.5 percent coal; Appendix B, the 2.0 percent; Appendix C, the 3.0 percent; and Appendix D, the 4.5 percent coal.

Table 1 provides a summary of the coal sampling time periods, sampling intervals, lot sizes, and total amount of coal sampled at each sampling location.

As noted previously, samples were taken during the filling of each drum. These samples were riffle split, providing one sample for the coal company and one for NBS. Each sample was labeled with the number of the drum from which it was obtained.

All of these subsamples were analyzed as soon as possible to determine their sulfur content. The 0.5 percent subsamples were analyzed at the Amax Coal Company's Western Division laboratory located near Gillette, Wyoming. The 2.0 and 4.5 percent subsamples were analyzed by Tradet, Inc., a commercial coal laboratory located near Wheeling, West Virgina. The 3.0 percent subsamples were analyzed by the Central Illinois Public Services (CIPS) Laboratory in Newton, Illinois. Table 2 presents the results of these sulfur content tests, which are also discussed in the Appendixes in relation to the coal sampling at each location.

Table 1. Summary of coal sampling operations

Sulfur Content (Percent)	Date Sampled	Sampling Location	Total Lot Sampled	Conveyor Belt Feed Rate	Time Period of Sampling	Sampling Frequency	Total No. of Drums	Wet Wt of Coal
0.5	5/27/81	Belle Ayr Mine Crushing Plant No. 2	8,000 tons	4000 tons/hr	2 hrs 15 min	3 min	9	3150 lbs
2.0	7/29/81	Humphrey Mine Barge Loading Facility Monongahela River	2,000 tons	900 tons/hr	2 hrs 40 min	8 min	6	2100 lbs
3.0	7/22/81	Delta Mine Preparation Plant	3,500 tons	775 tons/hr	4 hrs 0 min	20 min	6	2100 lbs
4.5	7/30/81	McElroy Mine Coal Sampling Building Mitchell Power Plant	2,270 tons	1200 tons/hr	2 hrs 20 min	15 min for first 2 drums - 8 min for last 4 drums	6	2100 lbs

Table 2. Results of sulfur content tests on coal subsamples taken from individual drums during coal sampling operations

Nominal Sulfur Content (Percent)	Coal Company	Mine	Location	Number of Drums Sampled	Individual Sulfur Contents* (Percent)	Average Sulfur Contents (Percent)
0.5	Amax Coal Company	Belle Ayr	Gillette, WY	9	0.44	0.44 ± 0.02
					0.45	
					0.47	
					0.42	
					0.41	
					0.46	
					0.43	
					0.42	
					0.47	
2.0	Consolidation Coal Co.	Humphrey	Osage, WV	6	1.87	1.84 ± 0.10
					1.91	
					1.82	
					2.14**	
					1.67	
					1.92	
3.0	Amax Coal Company	Delta	Marion, IL	6	2.77**	3.00 ± 0.10
					2.85	
					3.07	
					2.94	
					3.06	
					3.07	
4.5	Consolidation Coal Co.	McElroy	Captina, WV	6	4.67	4.65 ± 0.08
					4.72	
					4.50	
					4.64	
					4.70	
					4.65	

*All sulfur content values were determined on a dry basis.
**Coal from these barrels was not included in coal processing, nor in the average sulfur content reported.

On the basis of these sulfur tests, the following decisions were made concerning the handling and processing of the respective coal samples:

a. The 0.5 percent coal appeared to be very satisfactory with respect to the uniformity of its sulfur content. Due to its relatively high moisture before drying, it was decided to oven dry and process the contents of all nine of the 55-gallon drums.

b. Two of six drums of the 2.0 percent coal appeared to have a greater deviation in sulfur content from the mean than would normally be considered acceptable. These were drums 4 and 5. Since the sulfur content from drum 4 was the only one of the six that exceeded 2.0 percent, it was decided to process all but the coal from drum 4. It was not possible to obtain over 1,000 pounds of -60 mesh coal after drying with anything less than five drums; otherwise, drum 5 would have also been withheld from processing.

c. One of the six drums of the 3.0 percent coal also appeared to have a greater deviation in sulfur content from the mean than would normally be considered acceptable. This was drum number 1. This was the lowest sulfur content of the six samples of 3.0 percent coal that were tested. Therefore, it was decided to process all but the coal from drum 1.

d. The uniformity of the sulfur content for the 4.5 percent sulfur coal samples appeared to be acceptable, except that the sulfur content of the coal in drum 3 was slightly lower and deviated a little more from the mean than the other five samples. However, this did not appear to be significant and it was decided to oven-dry and process the contents of all six of the 55-gallon drums.

4. Processing of Coal Samples

All four of the coals obtained for this program were collected in plastic-lined and sealed 55-gallon steel drums and shipped to a processing facility in Camden, New Jersey. The facility, owned and operated by Alnort Processing, Inc., is used for the custom processing of various raw materials such as minerals, pigments, and chemicals. All of the processing for each coal in this program was conducted completely and exclusively in this facility.

As shown previously in figure 1, the processing of each coal involved the following steps:

a. First stage air-drying in a temperature-controlled oven;

b. Hammermill to crush coal;

c. Screening hammermilled coal through a 60 mesh sieve;

d. Blending -60 mesh coal in a cone blender; and

e. Packaging the blended coal in individually numbered 5-gallon pails.

Each processing step is discussed in detail in the following sections.

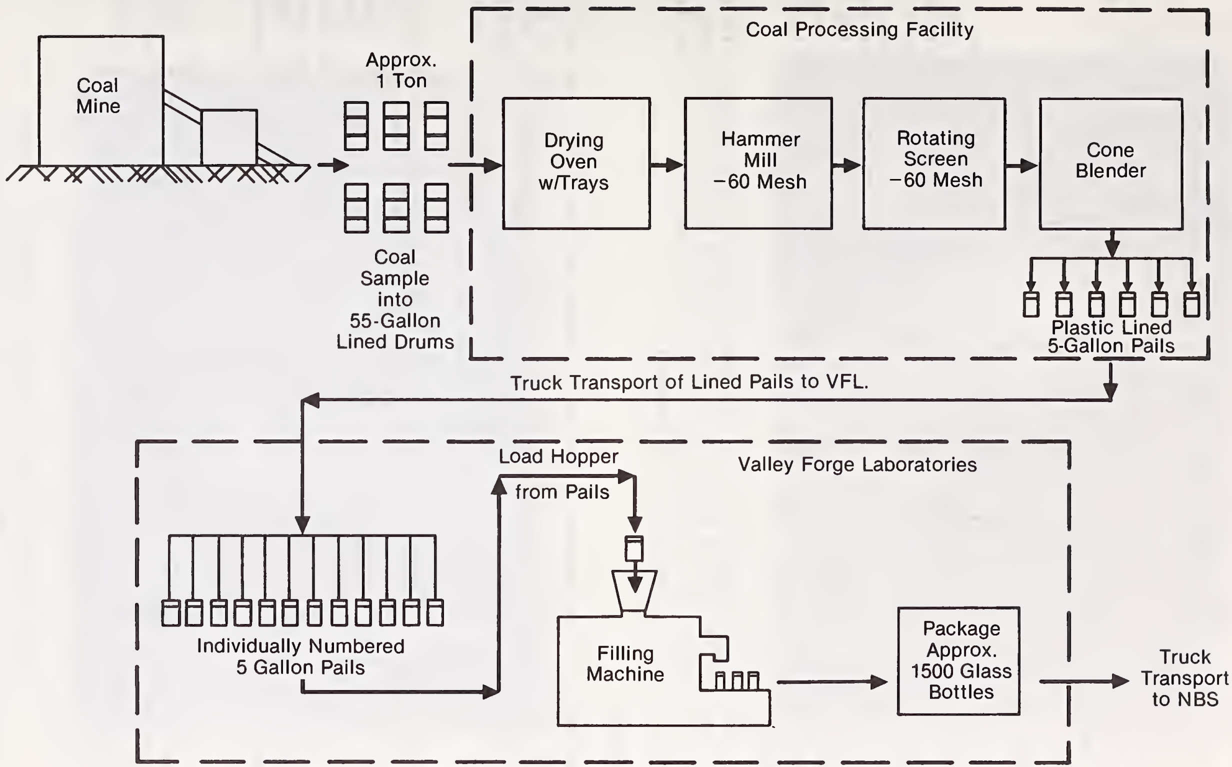

Figure 1. Material flow for preparation of standard reference coal samples

4.1 Air Drying

Air drying was accomplished by removing the coal from the drums, placing it in metal trays (approximately 3 feet square and 1 inch deep), and drying it in an oven for a period of 18 hours at an average oven temperature of 35 ^{o}C (95 ^{o}F), in accordance with procedures outlined in ASTM D2013, "Standard Method of Preparing Coal Samples for Analysis." All coals were dried at a constant oven temperature no more than 10 ^{o}C (18 ^{o}F) above ambient temperature. Figure 2 is a photograph of the oven and the tray loading of one of the coals.

4.2 Hammermilling

Following air drying, each coal was size reduced using a micro-pulverizer screw-fed hammermill having a feed rate of approximately 400 pounds per hour. Figure 3 is a photograph of the unit used in this phase of the coal processing. The hammermill was thoroughly cleaned by sandblasting before use as a means of preventing contamination from other materials. The four coals were processed in order of ascending sulfur content. Between the size reduction of each of these three coals, the hammermill was thoroughly air cleaned, but not sandblasted. A small amount (approximately 100 pounds) of each coal was run through the hammermill after each cleaning to purge the equipment prior to actual hammermilling of that particular coal.

The jaw setting of the hammermill was adjusted to assure that the majority of the coal feed was reduced in size from 4 mesh top size to a nominal 60 mesh material. The nature of the hammermilling operation caused some definite dust loss despite the slow feeding of the hammermill.

4.3 Screening

Upon completion of the hammermilling, each crushed coal was repackaged and stored in the same drums originally used for transport. The screening of the four hammermilled coals was accomplished by means of a cyclone screener, Model 240, manufactured by AZO, Inc., of Memphis, Tennessee. Figure 4 is a photograph of the AZO screener with a 55-gallon drum positioned to collect the oversize material.

The screener operates by receiving material into a small hopper and feeding it into a rotating hollow shaft surrounded by a screen of the desired size, in this case a 60 mesh screen. Material of the desired size passes through the screen and falls into a collection chute centrally located beneath the screen. Oversize material passes out a discharge port at the opposite end of the screener from the receiving hopper. According to the product literature, the maximum feed rate for the screener when operating with a 60 mesh screen would be approximately 300 pounds per hour. Figure 5 is a closer view of the 60 mesh screen mounted on the shaft.

Each coal was screened separately and the AZO screener device was blown clean before each screening. The -60 mesh coal was collected and repackaged into the same drums. A separate 55-gallon drum (or drums) was used to collect, seal, and store any oversize coal resulting from the hammermilling and screening operations for each coal.

Figure 2. Tray Loading and Oven Drying of Coal Sample

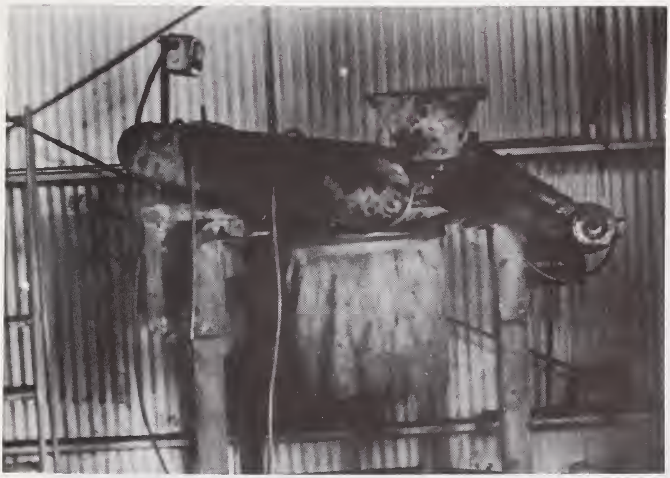

Figure 3. Hammermill Unit Used for Size Reduction of Coal Samples

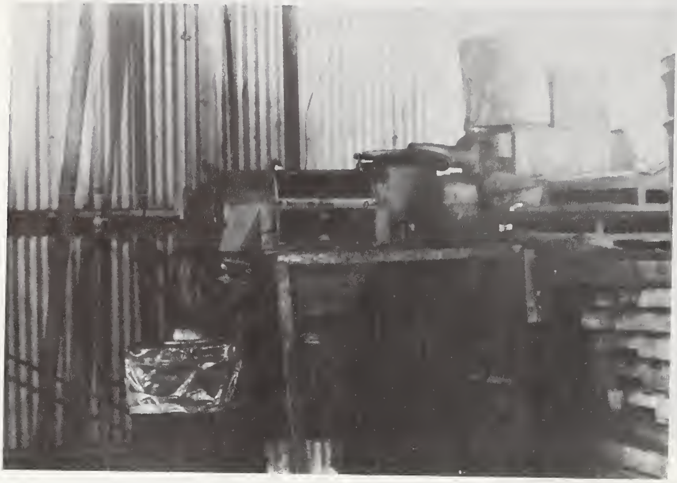

Figure 4. Cyclone Screener Used for Screening of Coal Samples

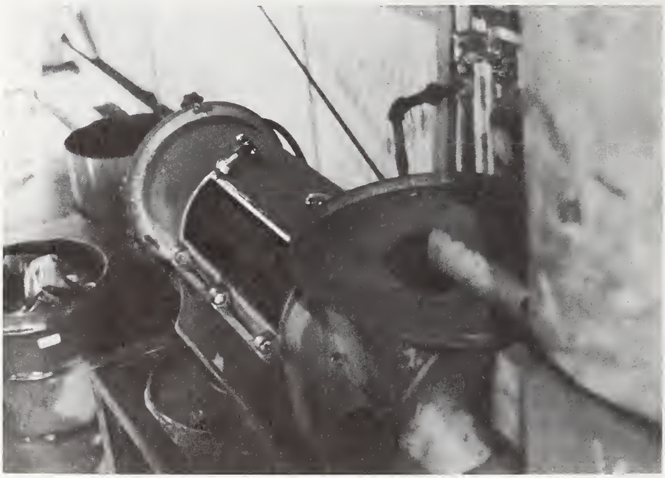

Figure 5. View of 60 Mesh Screen Mounted on Cyclone Screener Shaft

A small test sample (approximately 200 grams) was taken from each of the four screened coals prior to blending. Each of these samples was passed through a 60 mesh test screen at Valley Forge Laboratories to evaluate the percentage of material (if any) retained on the 60 mesh screen in the cyclone screener. The results of these rather cursory screen analyses are as follows:

Sulfur Content of Sample (Percent)	Percent Passing 60 mesh Screen
0.5	100
2.0	98
3.0	99
4.5	100

The coal sample weights before, during, and after processing are summarized in table 3.

4.4 Blending

Each of the screened coals was blended using a Patterson-Kelly stainless steel cone blender (approximate capacity 30 cubic feet). This blender was not equipped with an intensifier bar. The blender was of sufficient size to handle more than 1000 pounds of coal or other bulk material while still having enough room to properly blend the material. Figure 6 is a photograph of the cone blender with the discharge chute at the bottom.

All four coals were blended in the same manner. The screened coal was loaded into the top of the blender, the blender sealed, and blending accomplished by rotating the blender at 12 rpm for at least 20 minutes. The blender was blown clean and thoroughly wiped before blending of each coal.

Table 3. Summary of coal sample weights before, during, and after processing

Sulfur Level (Percent)	Weight of Coal Sample Received* (Pounds)	Weight of -60 Mesh Coal after Screening (Pounds)	Weight of -60 Mesh Coal after Blending (Pounds)
0.5	3150	1151	1139
2.0	2100	1050	988
3.0	2100	1089	976
4.5	2100	1200	1122

*Based on an estimated net weight of 350 pounds of moist coal in filled 55-gallon drum. Actual net weight will vary depending on the extent the drum was filled, the density of the crushed coal, and the moisture content of the coal at sampling.

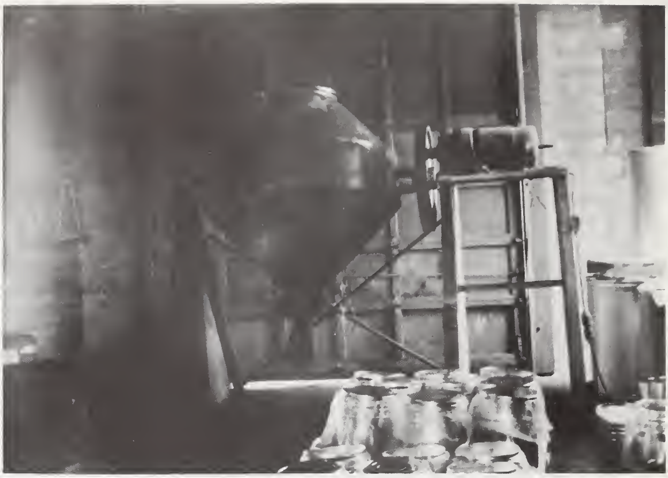

Figure 6. Cone Blender Used for Blending of Coal Samples

Figure 7. Discharging of Blended Coal into Lined 5-Gallon Pails

4.5 Packaging of Blended Coals

After blending, the discharge chute at the bottom of the cone blender was opened sufficiently to allow the coal to flow into a pail, as shown in figure 7. Each 5-gallon pail had a plastic liner for sealing. Care was taken to not fill each pail to capacity, so that at a later date, each pail could be rolled to mix the contents. Figure 8 shows the closing of a control lever to stop the flow of material from the blender when a pail is filled.

During the filling of each pail, two small samples for homogeneity testing were taken from the coal stream, approximately halfway through the filling of each pail. These samples were collected in 4 ounce glass bottles labeled with the sulfur content of the coal, the pail number, and the number of the sample taken from that pail. For example, the label "0.5, 10-2" indicates the second bottle taken from the 0.5 percent sulfur coal during filling of the tenth pail. All pails were labeled with the sulfur content of the coal and were numbered consecutively according to the order in which they were filled. At least once or twice during the unloading of each coal, the blender was rotated several times to minimize segregation of particles.

The total number of pails filled with each coal ranged from 32 to 50, depending on the total weight of the coal blended and the amount of coal placed into each pail. Table 4 summarizes the number of pails collected for each coal, the total weight of the coal, and the average amount of coal contained in each pail for that coal. Tables 5 through 8 are tabulations of individual pail weights for the 0.5, 2.0, 3.0, and 4.5 percent sulfur coals, respectively, and the sulfur concentrations of selected pails. These sulfur analyses were performed by an independent laboratory for purposes of testing the gross homogeneity and sulfur content of the processed samples. These preliminary analyses provided the basis for the decision to bottle the coals for subsequent certification.

After collecting and weighing each coal, the lined pails were shipped to Valley Forge Laboratories for eventual packaging into bottles. Individual 4 ounce bottles (two from each pail) were packaged and delivered to the National Bureau of Standards for acceptance and homogeneity testing.

5. Sample Preparation/Homogeneity Testing

Homogeneity testing is crucial in the preparation of an SRM. It is useless to perform highly accurate analyses on materials that exhibit high variability. Although homogeneity tests differ radically with various types of materials and with the various properties to be certified, all have the same common goal: to ensure that the material is sufficiently uniform to completely satisfy the end use. This helps to ensure that tests made in different laboratories will produce the same values within reasonable limits, and that test results obtained in one laboratory on one date will match those obtained at some later date. Thus, these tests are designed to ensure that candidate SRM materials meet two important criteria: homogeneity and stability.

Figure 8. Closing Flow from Blender During Filling of 5-Gallon Pails

Table 4. Summary of coal packaging operations

Nominal Sulfur Content (Percent)	Sampling Location	Total No. of Pails	Total Weight of Blended Sample* (Pounds)	Average Weight For Each Pail* (Pounds)	Weight Per Individual Pail* Maximum (Pounds)	Weight Per Individual Pail* Minimum (Pounds)
0.5	Belle Ayr Mine Gillette, Wyoming	50	1132.0	22.6	27.0	15.0
2.0	Humphrey Mine Osage, West Virginia	34	982.4	28.8	32.2	23.2
3.0	Delta Mine Marion, Illinois	32	974.8	30.5	37.0	10.2**
4.5	McElroy Mine Captina, West Virginia	36	1122.1	31.2	35.6	26.6

*Does not include tare weight of pail and liner.

**Represents weight remaining in blender during filling of last pail. Lowest weight of normally filled pail of coal was 27.4 pounds.

Table 5. Individual pail weights and sulfur content after blending of 0.5 percent sulfur coal

Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent	Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent
1	21	0.44, 0.45	26	27	
2	17		27	22	
3	15		28	19	
4	16		29	25	
5	22		30	25	
6	17		31	23	
7	22	0.46, 0.45	32	24	
8	22		33	22	
9	21		34	23	
10	22	0.45, 0.45	35	24	
11	22		36	23	
12	22		37	25	
13	22		38	25	
14	23		39	24	
15	21		40	23	
16	22		41	24	
17	22		42	25	
18	22		43	25	
19	22		44	23	
20	23		45	24	
21	22		46	26	0.45, 0.45
22	24		47	27	
23	24		48	24	
24	24		49	24	
25	23		50	23	0.45, 0.45

Sulfur Content, Weight Percent = 0.45 ± 0.01

Table 6. Individual pail weights and sulfur content after blending of 2.0 percent sulfur coal

Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent	Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent
1	31	1.76, 1.84	18	29	
2	32	1.84, 1.78	19	27	
3	30	1.82	20	31	
4	28	1.75	21	30	
5	27	1.83	22	31	
6	26		23	32	
7	26		24	31	
8	26		25	29	
9	28		26	31	1.85, 1.80
10	27		27	32	
11	32		28	28	
12	25		29	31	
13	29	1.85	30	27	
14	27		31	31	
15	27	1.87	32	29	
16	29		33	30	
17	32	1.74	34	23	1.76

Sulfur Content, Weight Percent = 1.81 ± 0.05

Table 7. Individual pail weights and sulfur content after blending of 3.0 percent sulfur coal

Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent	Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent
1	32	3.06	17	30	
2	35	3.06	18	27	
3	36	3.06	19	29	
4	30	3.04	20	28	
5	30	3.05	21	30	3.06
6	31	2.97	22	32	
7	29	2.93	23	29	
8	28		24	33	
9	32		25	34	
10	29		26	34	
11	33		27	37	3.03
12	28		28	30	
13	31	2.96, 3.01	29	31	
14	31		30	34	
15	28		31	32	
16	32		32	10	2.99

Sulfur Content, Weight Percent = 3.02 ± 0.05

Table 8. Individual pail weights and sulfur content after blending of 4.5 percent sulfur coal

Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent	Pail No.	Net Sample Weight (lbs)	Sulfur Content Weight Percent
1	33	4.64, 4.51	19	29	
2	27	4.38	20	31	4.63
3	30	4.57	21	33	
4	27	4.68	22	32	
5	29	4.51	23	33	
6	31	4.69	24	30	
7	27		25	30	
8	29		26	29	
9	33		27	32	
10	32	4.71	28	32	4.52
11	32		29	32	
12	30		30	31	
13	36		31	31	
14	33		32	30	
15	34	4.68	33	31	
16	33		34	33	
17	31		35	32	4.52
18	32		36	33	

Sulfur Content, Weight Percent = 4.59 ± 0.10

The homogeneity of each of the four coals was determined using an x-ray fluorescence wavelength spectrometer to measure the sulfur content. However, before analysis, a series of drying studies were performed to evaluate the moisture content of the blended coals. A one-gram sample of each coal was dried under vacuum at room temperature until constant weight was reached and then exposed to air at 15% relative humidity and then at 52% humidity (over saturated $Ca(NO_3)_2$ solution) to measure the sorption of moisture. The samples were then redried to measure the reversibility of this hydration. The drying and rehydration data are summarized below in table 9. All four coals reached constant weight (± 0.03%) after about 16 hours under vacuum at room temperature.

All regained much of their moisture after prolonged exposure to humid air. For the most part, this water adsorption is reversible, although the 0.5% sulfur coal shows noticeable hysteresis. The rate of moisture exchange is fairly rapid, but none of the materials is hydroscopic enough to cause significant analytical error if closed weighing bottles are used.

Table 9. Drying of coals

Conditions for Evaluating Weight Change[a]	0.5%	2.0%	3.0%	4.5%
Loss on drying, percent (Vacuum drying for 16 hours at 24 ^{o}C)	18.5	1.7	3.6	1.6
Uptake 2 1/2 hr at 15% RH, percent	0.7	0.3	0.4	0.3
+ 2 d at 15% RH, percent	2.3	0.2	0.5	0.3
+ 2 hr at 50% RH, percent	1.7	0.7	1.0	0.8
+ 5 d at 50% RH, percent	7.4	0.3	1.0	0.4
Total sorption, percent	12.1	1.5	2.9	1.8
Loss on redrying, percent	11.6	1.5	2.8	1.7

[a]The basis for calculating weight loss on initial drying is the weight as received; the basis weight for sorption and redrying is the dry weight.

A statistical evaluation of the gross homogeneity of the coal materials indicated that they were sufficiently homogeneous ($\leq$ 2 percent variation), to proceed with bottling. The bottling of each coal began with pail number 1 and proceeded in consecutive order using as many pails of coal as necessary to bottle 1500 50-g units. During the bottling process, the bottled units were sequentially numbered (1 to 1500) so that any variability that may have been caused by bottling could be detected and to assess any sample variability in the 1500 units.

In order to test the variability of sulfur in the four SRM's, two samples were removed from bottles numbered 241, 398, 410, 809, 1227, and 1500 for each of the proposed SRM's and vacuum dried for ~16 hours at 24 °C. After drying, the samples were pressed into pellets of 3-cm diameter. The relative sulfur content of each of the 12 samples of four SRM's was determined and the sulfur content in SRM's 1632a and 1635, Trace Elements in Coal, were used as controls. The results are shown in tables 10 and 10a. No correlation was found between measured values and the bottling sequence. Also, the relative sulfur content in the coals from different bottles was not significantly different from replicate samples from single bottles. Accordingly, all bottles of these proposed SRM's are homogeneous with respect to their sulfur content.

6. Conclusion

Four coals of different sulfur levels were collected, processed, bottled, and tested to determine their suitability as Standard Reference Materials. These four coals were of acceptable quality and the process of certifying them as SRM's was undertaken. These new SRM's were numbered sequentially from the lowest sulfur content to the highest as SRM's 2682, 2683, 2684, and 2685, respectively.

The details of the analytical procedures leading to the certification of these coals as SRM's are to be published in another NBS Special Publication in this "260 Series."

The success of this project was a direct result of the efforts of many people. Special acknowledgment is made by the authors to Mrs. G. P. Bowyer for her assistance in preparing this publication.

Table 10. Data for coal homogeneity samples

	Relative Sulfur Percent*			
Bottle No.	0.5	2.0	3.0	4.5
241A	0.999	0.989	1.001	1.001
241B	0.992	1.016	1.000	1.012
398A	0.990	0.988	1.007	0.978
398B	0.993	0.994	1.003	0.999
410A	0.997	0.985	0.994	1.000
410B	0.996	0.997	1.012	0.995
809A	1.017	0.997	1.000	1.010
809B	1.001	1.004	0.987	1.005
1227A	1.002	1.001	0.997	1.004
1227B	1.010	1.015	1.004	1.010
1500A	0.995	1.009	0.998	0.990
1500B	1.010	1.005	0.995	0.996
Average:	1.000	1.000	1.000	1.000
σ(n-1):	±0.008	±0.010	±0.006	±0.010

*The relative sulfur percent was determined by ratioing the average intensity of the sulfur x-ray line in all the measured bottles to that of each individual bottle.

Table 10a. Sulfur percent of controls*

Control 1632a

Analyses:	1	1.60
	2	1.57
	3	1.57
	4	1.57
Average		1.58 ± 0.02

Control 1635

Analyses:	1	0.33
	2	.33
	3	.32
	4	.33
Average		0.33 ± 0.01

*The controls, SRM's 1632a and 1635 are certified for sulfur values of 1.58 ± 0.04 and 0.33 ± 0.03 percent, respectively.

Appendix A

Sampling of 0.5 Percent Sulfur Coal From Belle Ayr Mine - Gillette, Wyoming

A low sulfur coal was obtained from the Amax Coal Company's Belle Ayr mine near Gillette, in Campbell County, Wyoming. This mine, opened for production in 1973, is an open-pit mine that produces subbituminous coal from the Wyodak-Anderson coal seam, which is part of the Powder River Coal Basin. The average seam thickness at the mine is 72 feet. The overburden depth varies, but averages 113 feet. During 1980, the Belle Ayr mine produced over 16 million tons of coal, making it the largest coal mine in the United States. This mine has a life expectancy of at least 20 years.

Coordination of sampling activities at the mine's coal preparation facility was handled through Thomas J. Lien, Manager of Preparations for the Western Division Amax, whose office is located in Gillette. The actual collection of the coal was done by Terry R. Bosecker, Chemist at Amax Western Division Laboratory, and Brian Pleuss, who also works at the Western Division Laboratory. The coal was collected in nine 55-gallon plastic-lined steel drums over a 2-1/4 hour period.

The Belle Ayr mine has two crushing plants. The smaller plant is rated at 2500 tons of coal per hour. The larger plant can handle 4000 tons of coal per hour. Each crushing plant feeds two concrete silos. All four silos are the same size and have a capacity of approximately 11,000 tons of coal. The coal from these silos is fed into unit trains comprised of 100 to 110 coal hopper cars and then shipped to coal-burning electrical utility plants in eleven states.

The coal sampled for this program was obtained from the larger plant (Plant No. 2) between 11:00 a.m. and 12:00 noon and between 1:00 p.m. and 2:15 p.m. on Tuesday, May 25. Actually, the coal sampled was a blend of coal from the upper and lower benches of the mine. During the time of sampling, the coal was being fed from the crushing plant into Silo No. 4 for eventual train loading. Since approximately 3000 tons of coal had already been fed into Silo No. 4, the coal obtained during the 2-1/4 hour sampling period was sampled from a total of approximately 8000 tons, representing most of a unit train load of coal.

Figure A-1 is a layout of the mine, with its crushing plants, silos, rail facilities, laboratory, and office. This general layout provides readers with a perspective of the mine operation and where the coal was obtained with respect to locations of various mine facilities.

The coal sampling operation was conducted using the existing coal sampling facilities within the crushing plant. Incoming coal crushed to nominal 2" size is periodically removed from the conveyor belt by a primary sample cutter. A total sample of approximately 500 pounds of coal is separated from the main feed at regular intervals of approximately 2 to 3 minutes. The sampled coal is directed down a chute to a crusher, which reduces it in size to 8 mesh. The crushed coal is then passed through a series of two rotary sample cutters, each of which operates at 20 RPM. These cutters are equipped with four blades that separate a portion of the incoming coal for subsequent laboratory analysis. The balance from these sample splitters is returned to the main conveyor belt feeding the silos by means of an enclosed auger feed chute. A sampling pipe located at the downstream end of the auger chute was used to obtain the coal sample. Figure A-2 is a schematic diagram that shows the layout and function of the sampling system in the crushing plant.

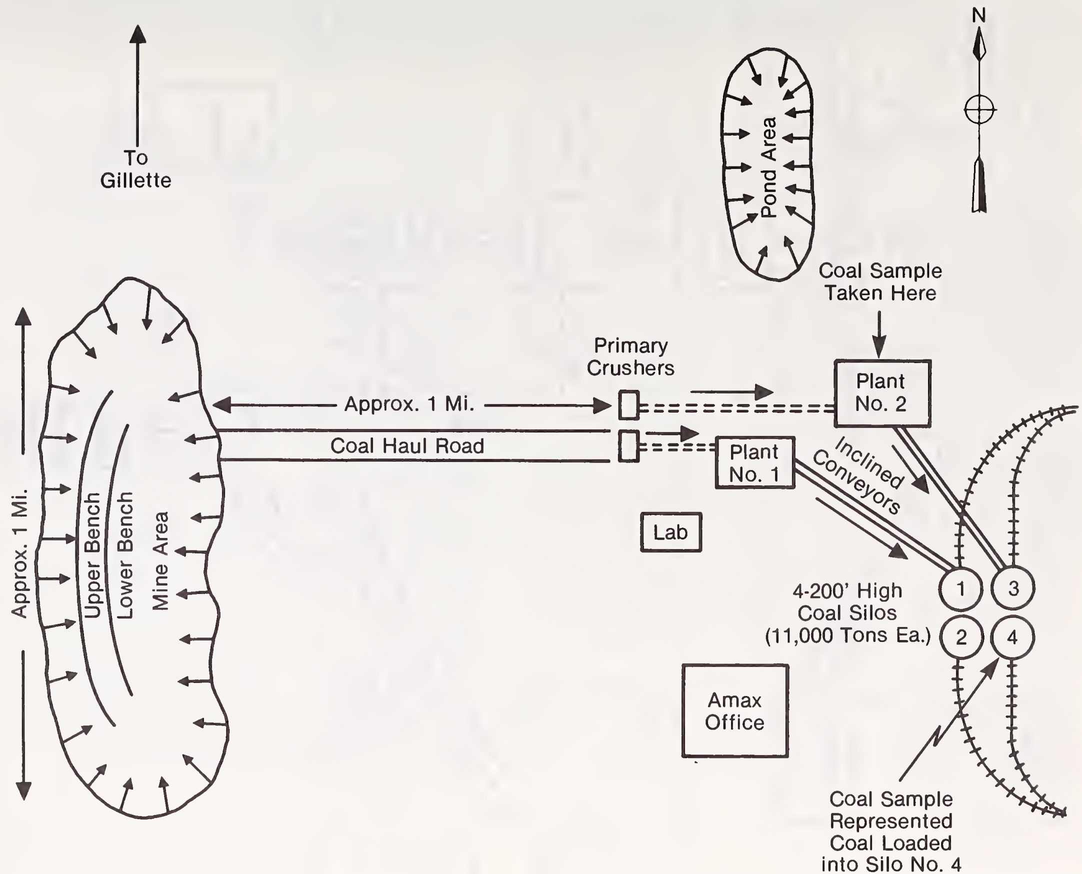

Figure A-1. Layout of Belle Ayr Mine, Gillette, Wyoming

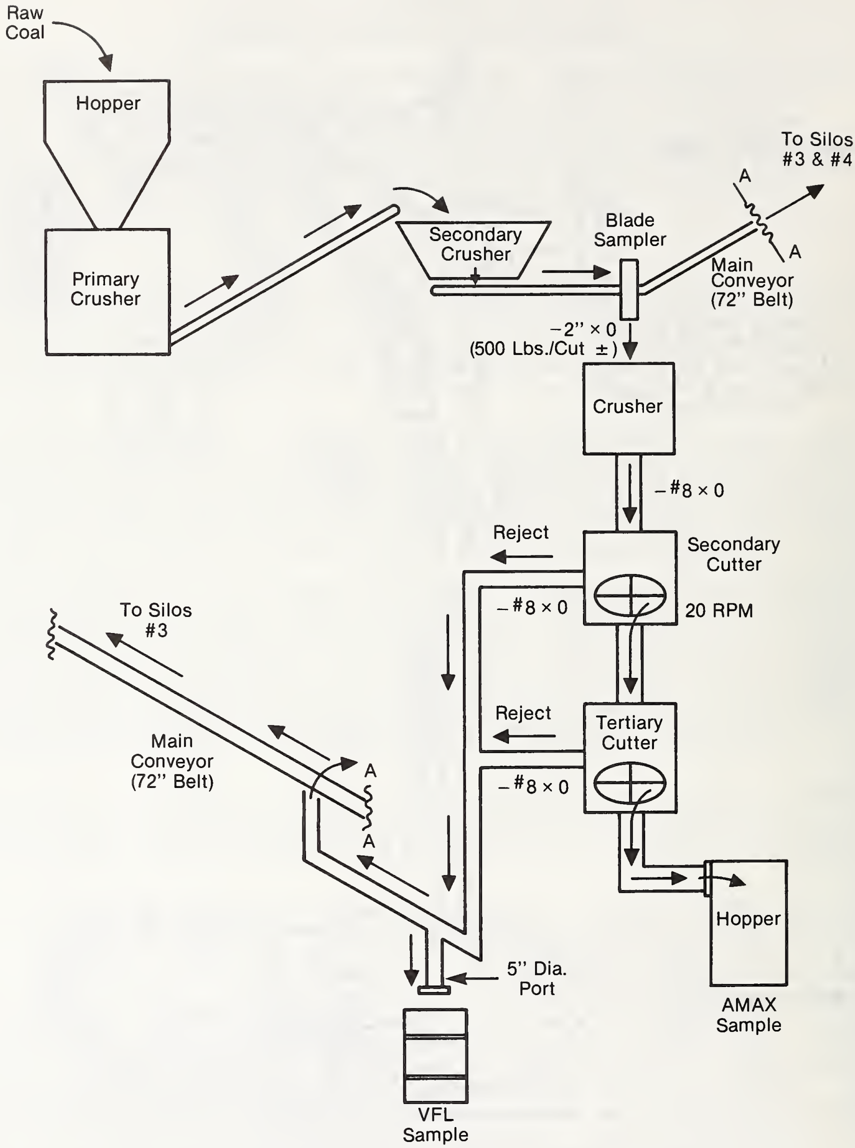

Figure A-2. Schematic of coal sampling system

The sampling interval selected was based on filling nine 55-gallon drums with coal in a time period corresponding to that required to fill one unit train (approximately 10,000 tons). Therefore, one drum was to be filled every 15 minutes.

The first drum was approximately two-thirds full after it had been filled for three minutes. It was then decided to use a 15-minute period to fill each drum by opening the sampling gate for one minute, closing it for two minutes, then alternately opening and closing the sampling gate using the same timing until the nine drums were completely filled. All drums were filled over an elapsed time of 2 hours, 15 minutes with an average of five 1-minute samples taken to fill each drum. Drums were numbered consecutively from 1 to 9 according to filling order.

Concurrently, separate small grab samples were taken directly from the coal stream at the discharge chute during each one-minute sampling period. These small grab samples were placed into individual plastic bags, one for each 55-gallon drum. Each of the plastic bags was given a number corresponding to the drum number. An estimated 6 kg of coal was placed into each of the plastic bags. Figure A-3 shows the filling of a 55-gallon drum.

All plastic liners inside the 55-gallon drums were tied closed after sample collection. Each drum contained approximately 350 pounds of coal. Therefore, the total weight of the coal sampled was approximately 3,150 pounds. The moisture content of this coal is known to range from 28 to 30 percent. Even after ASTM first-stage drying, Amax personnel advised that the average moisture content of this coal would still be somewhere around 15 percent. The plastic bags containing grab samples were tied, placed inside the proper drums, and the drums were then transported from the crushing plant to a receiving area located adjacent to the laboratory building.

Each 6 kg bag sample was taken into the laboratory, passed through an enclosed riffle splitter once or twice (depending on the sample size), and split into one 1.5 kg sample and two 750 g samples. All samples were placed into plastic bags, labeled, and sealed. The 1.5 kg sample bags were placed into their respective drums. One of each of the 750 g samples was sent to Valley Forge Laboratories. All or part of these bag samples will be made available to NBS for laboratory analysis. The remaining 750 g samples were retained by Amax for laboratory analysis of sulfur content. Figure A-4 shows a coal sample being divided with a riffle splitter.

Figure A-3. Sampling of Coal in 55-Gallon Drum at Belle Ayr Mine

Figure A-4. Riffle Splitting of 6000 Gram Coal Sample at Belle Ayr Mine

The results of sulfur and moisture determination tests performed at the Amax Western Division laboratory on each of the nine 750 g bag samples of this coal are as follows:

Grab Sample No.	Second Stage Moisture (percent)	Percent Sulfur (wet basis)	Percent Sulfur (dry basis)
1	13.72	0.38	0.44
2	15.28	0.38	0.45
3	17.13	0.39	0.47
4	17.22	0.35	0.42
5	16.32	0.35	0.41
6	16.17	0.39	0.46
7	14.76	0.37	0.43
8	16.34	0.35	0.42
9	13.99	0.41	0.47

The average sulfur content (dry basis) for the nine samples tested is 0.44 percent. The standard deviation is 0.02 percent. Therefore, the sample was considered acceptable for further processing.

The nine sealed drums were shipped to Alnort, Inc., in Camden, New Jersey, for subsequent processing.

Figures A-5 and A-6 provide some idea of the vast extent of the mined area at Belle Ayr. Figure A-5 shows the main portion of the mined area with the upper and lower benches. Figure A-6 provides some perspective of the depth of the mined area when considering that the capacity of the coal truck shown in the photograph is 100 tons.

Figure A-5. Mined Area at Belle Ayr Mine Showing Two Working Benches in 72 Foot Seam

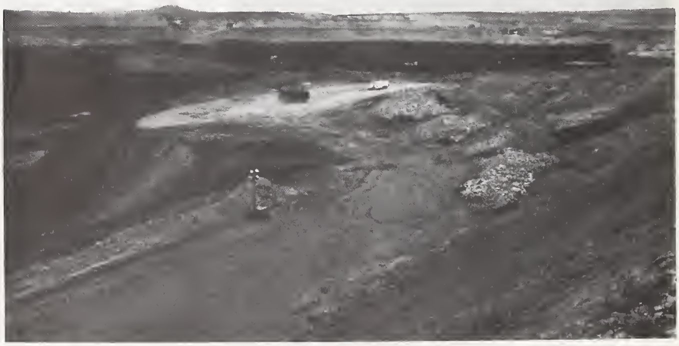

Figure A-6. Mined Area at Belle Ayr Mine Showing Depth of Excavation and Coal Truck

Appendix B

Sampling of 2.0 Percent Sulfur Coal From Humphrey Mine - Osage, West Virginia

A one-ton sample of coal was obtained from the Humphrey No. 7 Mine and coal preparation plant of the Consolidation Coal Company, Christopher Coal Company Division, located in Osage, West Virginia, near Morgantown in Monongalia County. This mine produces bituminous coal with a sulfur content of 1.8 to 1.9 percent (dry basis) after washing. This coal is obtained from an underground mine that recovers coal from the Pittsburgh seam, which is considered the single most valuable and extensive coal seam in the United States. This seam reportedly averages 7 feet in thickness. The Pittsburgh seam lies in the Northern West Virginia coal field and is geologically part of the Monongahela Group. During 1980, 2.1 million tons of coal were mined at the Humphrey No. 7 mine.

The Humphrey No. 7 mine is approximately 15 miles away from the Humphrey preparation plant, which is situated along the Monongahela River. The preparation plant and an associated blending bin were constructed some twenty years ago to supply 1.6 percent sulfur metallurgical coal for the U.S. Steel Company plants in the Pittsburgh area. Over the years, the demand for the metallurgical coal by the steel industry has diminished to the point where now all low·sulfur coal from the Humphrey plant is being shipped by barge to the West Penn Power Company's Mitchell plant in Monongahela, Pennsylvania.

Raw coal from the Humphrey No. 7 mine has a sulfur content (dry basis) of approximately 2.2 percent. After washing, the sulfur content (dry basis) has been reduced to approximately 1.8 to 1.9 percent. The raw coal is normally stored in a 10,000-ton coal silo next to the plant. The washed coal is conveyed from the plant to a blending facility, where twelve different feeders each fill four pockets, so that a total of 48 separate pockets of coal are available for blending during barge loading. One of the blending bins usually contains 2.2 percent sulfur coal in the event other coal users require a higher sulfur product. The blended coal is transported approximately a half mile to the barge loading facility along the Monongahela River by an overhead conveyor system. An automatic float-sink processing unit precedes the barge loading facility to further remove any trace impurities that may be present in the coal. The coal loaded into barges is nominal 1-1/4 inch size.

Figure B-1 is a sketch showing the location of the preparation plant, blending bins, silo, and barge unloading facilities at the Humphrey complex. Figure B-2 shows the Humphrey preparation plant, silo, and overhead conveyor system. The coal sample obtained for the Standard Reference Materials program was taken using the sampling system at the barge loading facility during the loading of four barges on the evening of July 29, 1981. Figure B-3 shows the barge loading facility at the Humphrey plant.

Arrangements for the coal sampling at Humphrey Mine were handled by James Cryster, quality control engineer for the Northern West Virginia region of the Consolidation Coal Company and by Don Jones, shift manager at the barge loading facility. During the sampling period, which lasted from 7:00 to 10:20 p.m., minor modifications were made to the coal sampling system located on the second floor of the loading building.

The existing coal sampling system consists of a primary sample cutter, which takes a full belt 240 pound cut every four minutes; a primary crusher, which reduces the size of the sample to nominal 3/8 inch; a secondary cutter, which

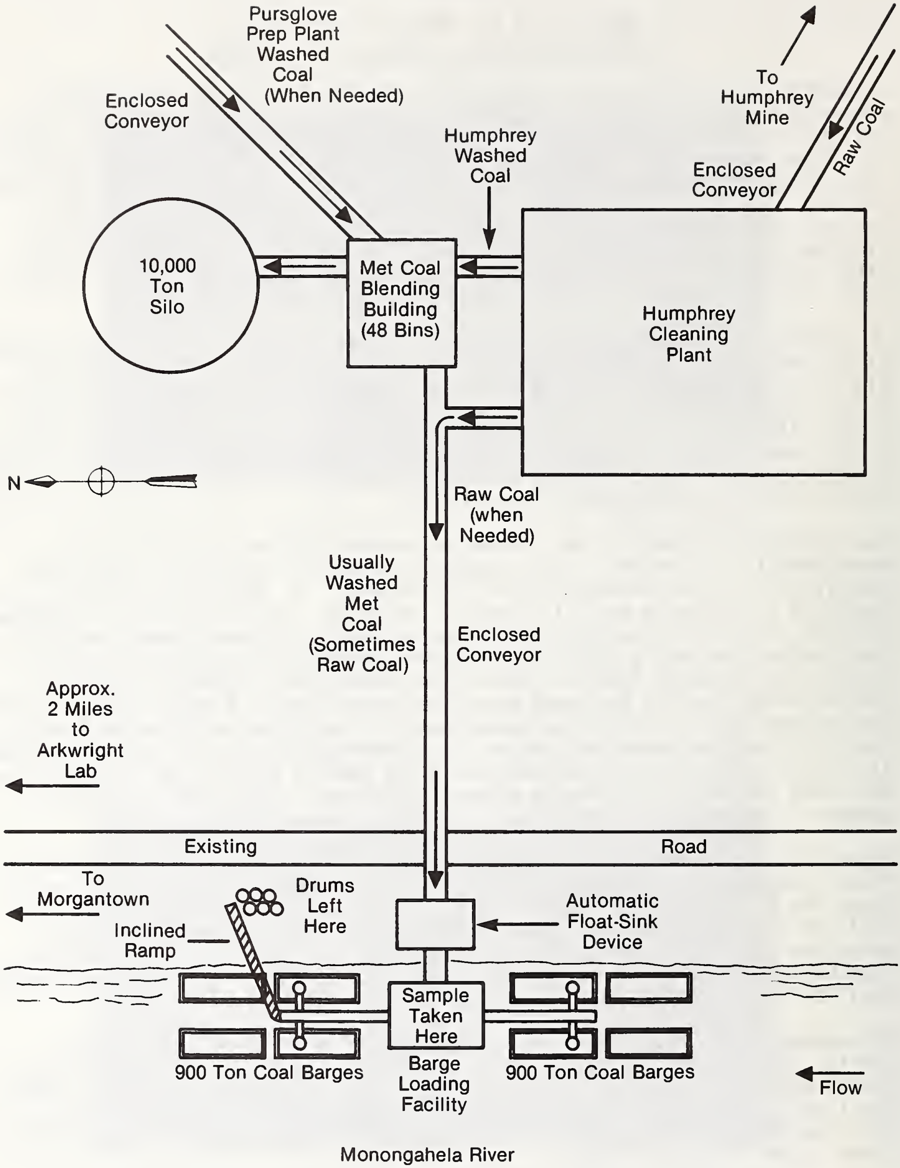

Figure B-1. Layout of Humphrey Mine and Plant Consolidation Coal Company, Osage, West Virginia

Figure B-2. Humphrey Preparation Plant and Conveyor System.

Figure B-3. Barge Loading System at Humphrey Plant.

separates a sample for Consolidation Coal Company; and a chute to convey the rejects from the sampling stream back to the barge loading chute. A sheer metal plate was inserted into an existing opening in the reject chute to divert the reject stream into lined 55-gallon steel drums to obtain the required sample of coal. Normally, the reject stream comprises about 25 percent of a full stream cut. Figure B-4 shows a schematic of the coal sampling system and the collection of the coal sample at the barge loading facility.

Coal barges loaded from the Humphrey plant have a capacity of approximately 900 tons each. Generally, these barges are loaded in sets of four during a half shift or four hour period. The coal sample was taken during a half shift and, therefore, was sampled from a total lot of 3,600 tons of barge loaded coal. Figure B-5 shows a loaded coal barge with another barge in the process of being loaded.

Because approximately 60 pounds of coal were anticipated from the reject stream for each 4 minute cut, it was initially decided to obtain a sample from each cut. During drum filling, intermittent samples of the reject stream were also sampled and placed into sealed plastic bags, one for each drum sample. During the first half hour of the sampling, one full drum and approximately 75 percent of a second drum had already been filled. At the same time, Consolidation Coal Company's foreman in the control room noted that they had been unable to obtain their usual coal sample. At the end of the first half hour, during the dinner break, it was discovered that the secondary cutter had not been operating and most of the entire cut during the sampling period had been diverted to the reject stream. An electrician was able to make the necessary repairs before resuming barge loading and the remainder of the sampling process proceeded without any problems.

However, the rate of sampling was reduced to be able to sample from a sufficiently large lot of barge loaded coal. Upon resumption of barge loading, at approximately 8:10 p.m., every other cut was sampled from the reject stream, instead of every cut, as had been done previously. The last of the six lined 55-gallon drums was loaded at 10:20 p.m. By that time, approximately 2,000 tons of coal had been loaded into the four barges. The six loaded drums were later transported by Valley Forge Laboratories directly to the Alnort coal processing facility in Camden, New Jersey.

Sulfur analyses of the Consolidation Coal Company's Humphrey samples were performed at the company's nearby Arkwright laboratory. Typically, only four samples are analyzed during a half shift loading period. These samples correspond to quarter, half, three-quarter, and fully loaded conditions. The results of the sulfur tests from the Arkwright lab are:

Loading Condition	As Determined* Percent Sulfur
Quarter	1.91
Half	1.93
Three-quarter	1.81
Full	1.85
	1.88 Average

*"As Determined" refers to the sulfur level at time of test, with an approximate moisture of 0.5 percent for each sample.

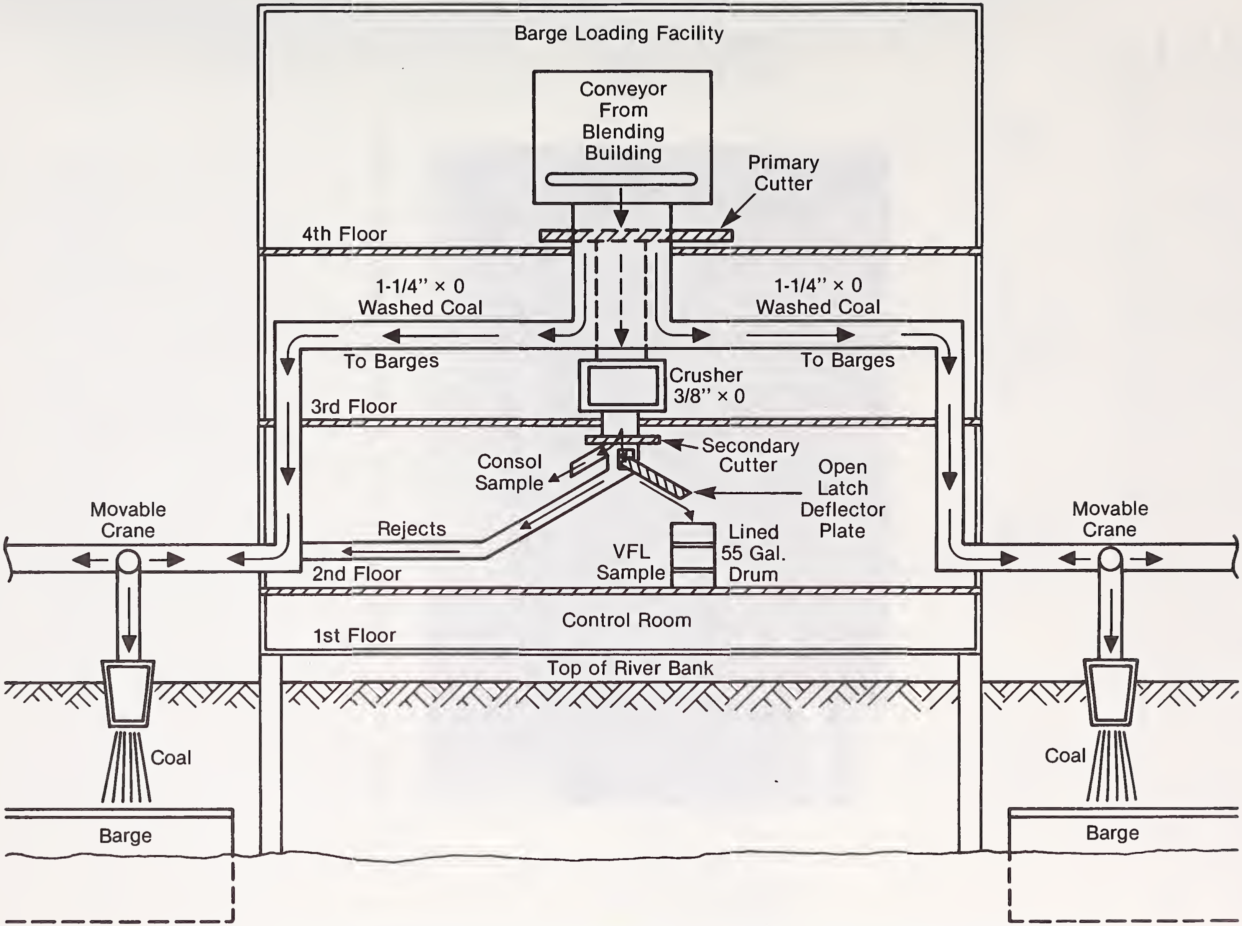

Figure B-4. Schematic diagram of coal sampling of Humphrey plant barge loading facility

Figure B-5. Loading of Coal Barge at Humphrey Plant

The six sealed plastic bag samples were later riffle split, with one portion of each sample given to Valley Forge Laboratories in a sealed plastic bag and part of the other portion being placed in individual 4 ounce plastic bottles for analysis by Tradet, Inc., an independent coal testing laboratory from Wheeling, West Virginia. The samples analyzed by Tradet were pulverized to -60 mesh size and tested for sulfur content using a Leco furnace model number SC37. The results of the Tradet sulfur analysis tests are:

Sample No.	Percent Sulfur (as received)	Moisture Content (percent)	Percent Sulfur (dry basis)
1	1.80	3.94	1.87
2	1.85	3.27	1.91
3	1.74	3.92	1.82
4	2.07	3.49	2.14
5	1.62	3.10	1.67
6	1.85	3.65	1.92

The average sulfur content of the six samples is 1.89 percent. However, it was noted that samples 4 and 5 have a greater deviation from the mean than the other samples. Tradet was asked to recheck the sulfur level of these two samples and after retest, reported dry basis sulfur levels of 2.18 and 1.65 percent, respectively. Because these values essentially confirm the initial analyses, it was suggested that all the coal, except for drum number 4, (the only sample with a sulfur level in excess of 2 percent) be processed if at least 1000 pounds of -60 mesh coal could be obtained from the remaining five drums. All barrels of the Humphrey coal, except for number 4, were processed.

Appendix C

Sampling of 3.0 Percent Sulfur Coal From Delta Mine - Marion, Illinois

A one-ton sample of coal was obtained from the Delta mine of the Amax Coal Company. The mine, which was expanded and reopened in 1978, is a surface mine located near the small town of Crab Orchard, roughly six miles east of Marion, Illinois. This mine originally began operation in 1934 and produces bituminous coal with a sulfur content after washing of 2.8 to 2.9 percent (dry basis). It is situated in Williamson County in southern Illinois. The coal mined at the Delta mine is obtained from the Illinois No. 6 Herrin seam, which is the most extensively mined coal in Illinois. In the district comprising Franklin, Jefferson, and Williamson Counties, the Illinois No. 6 Herrin seam has a reported average thickness of 8 feet in these counties, according to the Keystone Coal Industry Manual. At the Delta mine, this coal seam is 5-1/2 feet thick and lies below 81 feet of overburden.

During 1980, the Delta mine produced more than 2 million tons of steam grade coal. Nearly all of this coal is shipped to coal-fired power plants, with several unit train loads each week going to a Central Illinois Public Service (CIPS) plant in Newton, Illinois. At least one unit train each week goes to Consumers Power in Wisconsin for loading onto lake boats. A certain amount of coal is also trucked away from the mine and taken to barges on the Ohio River for shipment to Florida Light and Power Company. Some coal is also taken by barge to New Orleans for export to Japan.

Coal produced at the Delta mine is currently being obtained from two operating open pits. These pits are located several miles away from and on opposite sides of the mine buildings and preparation plant. Figure C-1 is a sketch showing the layout of the mine and the two open pits. These two pits are referred to as the 1450 pit and the 3270 pit. The pits are so named because of the size of the respective draglines that are being used to excavate each pit. The 1450-W is a smaller dragline than the 3270-W, which is the second largest dragline ever put into operation. The coal from the 1450 pit is considered to be of better quality than coal from the 3270 pit because a rock fault exists through part of the coal seam in the 3270 pit. During the time that the coal sample was being taken, all of the coal fed into the preparation plant was taken from the 1450 pit. Figure C-2 is a view of this open pit with the 1450-W dragline in the background.

The raw coal from the pit is first crushed to a maximum 5 inch particle size by a rotary breaker, then fed into the preparation plant. Figure C-3 shows an overall view of the preparation plant. The coal is first separated from rock and other impurities by heavy media separation in the flotation cells or wash boxes. The coal product is then split into several sieve fractions and further processed by washing, froth flotation, or further size reduction, depending on the sieve fraction being prepared. After final preparation, all sieve fractions are dewatered and combined into a single feed stream. The top size of the washed coal coming out of the preparation plant is 2 inches.

Coordination of sampling activities at the Delta mine was handled by Dick Lawrence, preparation plant manager at the mine. The actual collection of the coal sample was done by three Amax employees who were members of the

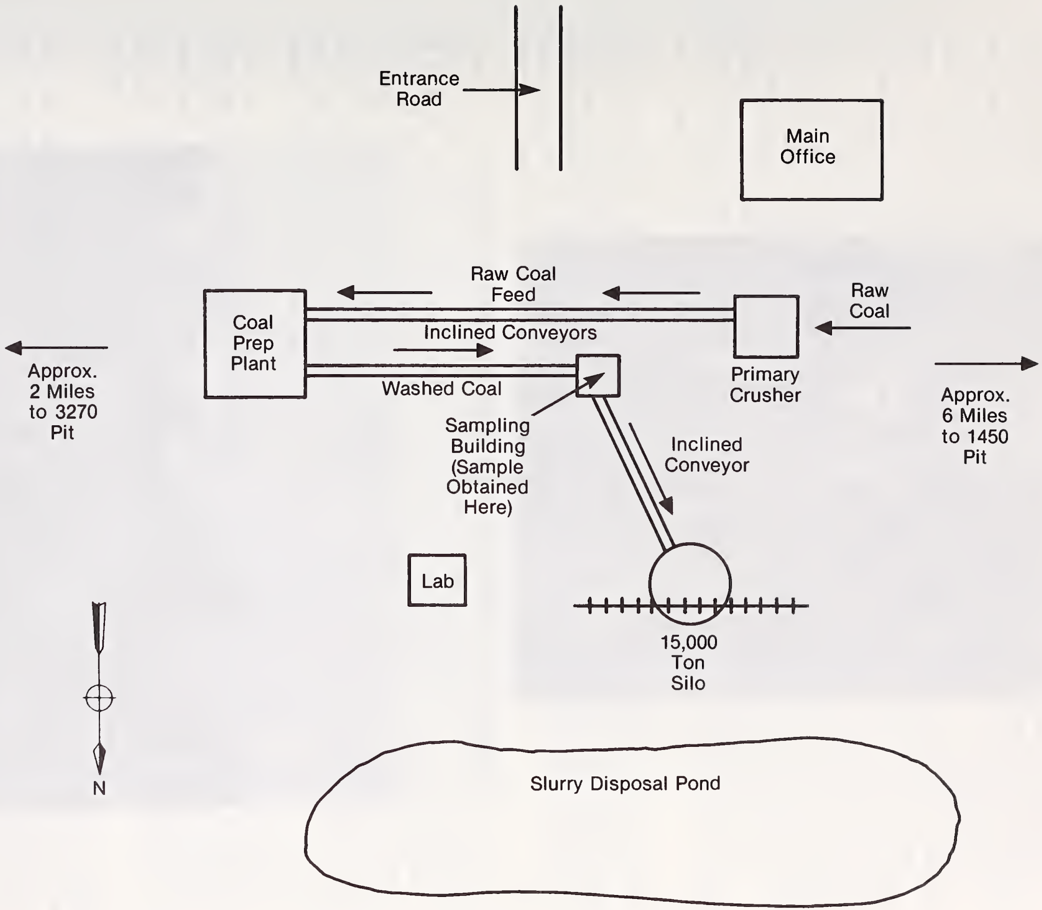

Figure C-1. Layout of Delta Mine, Marion, Illinois

Figure C-2. View of 1450 Open Pit and Dragline at Delta Mine

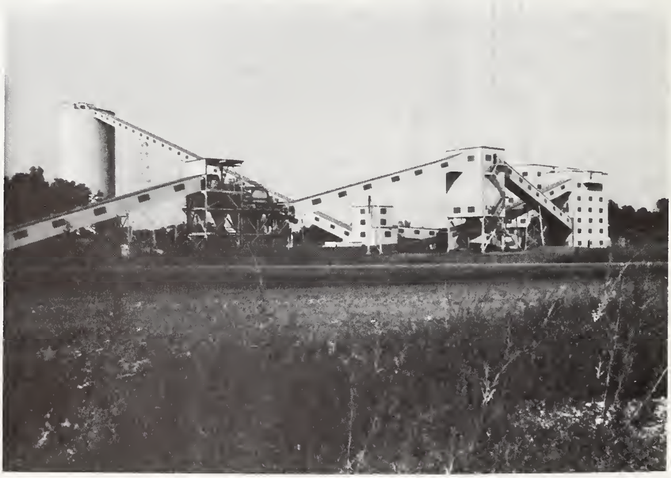

Figure C-3. Overall View of Coal Preparation Plant at Delta Mine

United Mine Workers Union. Sample collection activities were conducted in the sampling building, where washed coal is fed from the preparation plant to a 15,000 ton coal silo. Samples are ordinarily collected for analysis by Amax Coal Company at this location.

The existing coal sampling facilities were modified slightly and utilized for the collection of the coal sample for the Standard Reference Materials program. The sampling system consists of a primary sample cutter, which takes a full-belt cut of approximately 358 pounds every four minutes. The coal is conveyed by elevator and screw auger to a crusher, which reduces the top size of coal to 4 mesh. This crushed coal is fed into a secondary rotary sample splitter, where a bag sampler on the second floor collects a coal sample for later use by Amax personnel. Normally, the reject stream from the secondary cutter is discharged into a rectangular chute and then onto the main conveyor belt feeding the coal silo. To obtain a coal sample for this program, an adjustable metal deflector plate was installed into the rectangular discharge chute for the reject stream and the reject coal was diverted at selected intervals onto an open loading area on the second floor. From the second floor, the reject coal was shoveled into a circular chute passing into the ground floor area of the sampling building, where the flow was captured in lined 55-gallon steel drums. Figure C-4 shows the collection of the coal sample. Figure C-5 is a schematic diagram that shows the layout of the coal sampling system utilized at the Delta mine.

The flow rate of the washed coal on the main conveyor belt feeding the silo was initially estimated at 775 tons per hour. A four-hour sampling period was employed, between 8:20 a.m. and 12:20 p.m., representing a sampling from approximately 3100 tons of coal. Actually, belt recording equipment later indicated that a total of 3500 tons of coal passed over the main feed belt during the four-hour sampling period.

A total of six 55-gallon plastic-lined drums of coal were collected in four hours. The reject coal from the one sample cut was enough to fill at least half of a drum. Therefore, every fifth cut was taken and half a drum was filled every 20 minutes, and one drum was filled every 40 minutes. During the drum filling, small full-stream cuts were also taken at different times and placed into plastic sample bags. Each plastic bag of coal represented a sample from one of the six drums. Approximately 5 kg of coal were collected in each plastic bag. Both the 55-gallon drums and the plastic sample bags were numbered consecutively from one to six according to filling order.

Once the coal samples were collected in 55-gallon drums, the drum liners were tied and the drums were stored outside the sampling building. Each drum had an estimated weight of approximately 350 pounds of coal, so that the total sample comprised a little over one ton of coal. This coal is reported to have a moisture content between 8 and 10 percent with an ash content between 10 and 12 percent. On July 28, the six drums were shipped to Alnort, Inc., in Camden, New Jersey, for further processing.

Each 5 kg bag was taken to a processing area inside the preparation plant, where the coal was twice passed through an enclosed riffle splitter. One portion from each bag was placed back into its respective plastic bag, sealed, and given to Valley Forge Laboratories for later use by NBS. Six small plastic bottles, one from each bag, were also filled with the as-received coal and were also given to Valley Forge Laboratories for later use by NBS.

Figure C-4. Collection of Coal Sample and Companion 5000 Gram Sulfur Analysis Sample at Delta Mine

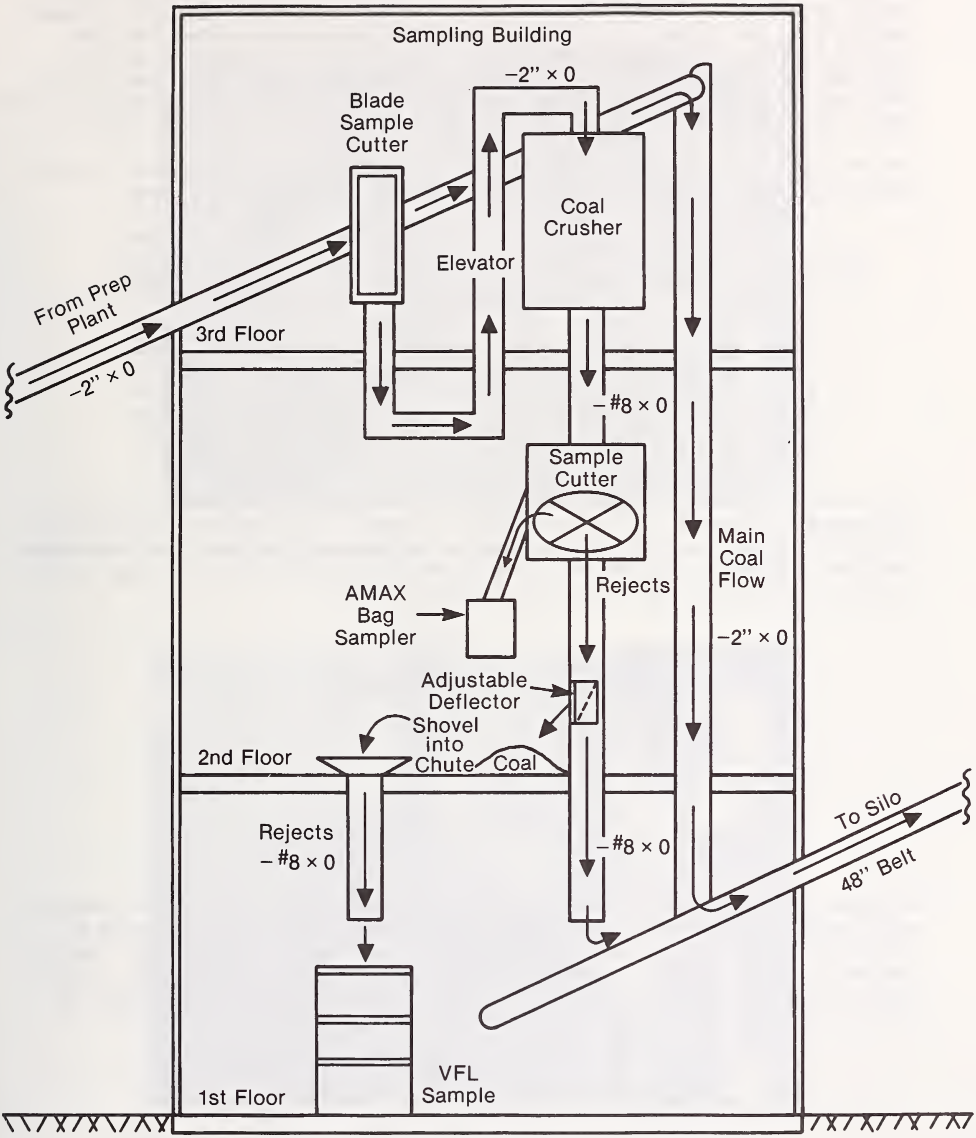

Figure C-5. Schematic of coal sampling system, Delta Mine

The other portion from each bag was reduced to -60 mesh size in a laboratory model pulverizer. Figure C-6 shows the riffle splitter and laboratory pulverizer equipment. Figure C-7 shows the air cleaning of this equipment between samples from each drum. The -60 mesh coal from the bag was placed into two plastic bottles, one of which was given to Valley Forge Laboratories and one retained for sulfur analysis in the laboratory at the Delta mine. The six -60 mesh samples given to Valley Forge Laboratories were taken to the Central Illinois Public Service (CIPS) laboratory in Springfield, Illinois, for sulfur analysis.

The results of the sulfur and moisture determination tests performed at the CIPS laboratory on each of the -60 mesh samples of this coal are as follows:

Sample No.	Percent Sulfur (as received)	Moisture Content (percent)	Percent Sulfur (dry basis)
1	2.57	7.84	2.77
2	2.67	6.70	2.85
3	2.87	6.93	3.07
4	2.75	6.98	2.94
5	2.87	6.67	3.06
6	2.88	6.74	3.07

The average sulfur content (dry basis) for the six samples tested at CIPS is 2.96 percent, although the sulfur content of sample 1 is somewhat lower than the average. Without sample 1, the average sulfur content (dry basis) of the remaining five samples is 3.00 percent. The as-received sulfur content values for the six samples tested at the Delta mine laboratory are:

Sample No.	Percent Sulfur (as received)
1	2.43
2	2.49
3	2.61
4	2.51
5	2.67
6	2.72

Although no direct comparison is made between the percent sulfur values from the two laboratories, it is significant to note that each laboratory reported the sulfur level of sample 1 as the lowest of all six samples. Therefore, a decision was made to process five of the six drums of 3.0 percent coal and, with NBS concurrence, to withhold drum 1 if a sufficient quantity of -60 coal could be obtained from the remaining drums.

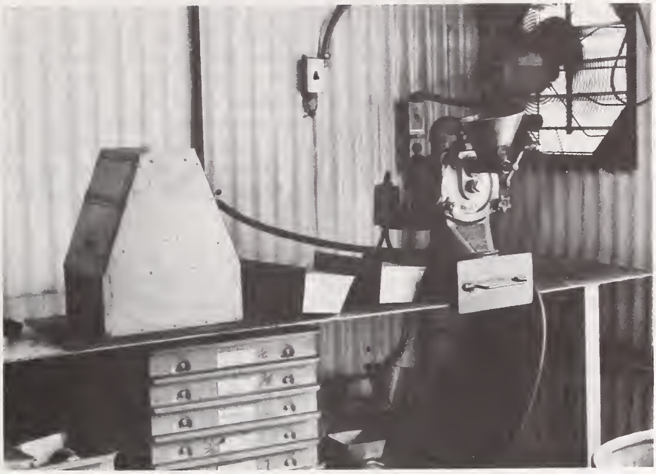

Figure C-6. Riffle Splitter and Laboratory Pulverizer Equipment at Delta Mine.

Figure C-7. Air Cleaning of Laboratory Pulverizer.

Appendix D

Sampling of 4.5 Percent Sulfur Coal From McElroy Mine - Captina, West Virginia

A one ton sample of coal was obtained from the McElroy mine and coal preparation plant of the Consolidation Coal Company, Ohio Valley Division, located in Captina, West Virginia, some 20 miles south of Wheeling along the Ohio River in Marshall County. This mine produces bituminous coal with a sulfur content of approximately 4.5 percent (dry basis) after washing. This coal is obtained from an underground mine that is a part of the Pittsburgh No. 8 coal seam, which is in the northern West Virginia coal field. The seam thickness at the McElroy mine is 5-1/2 feet. The mine is 275 feet underground at the portal and, during 1980, produced 1.4 million tons of coal.

The McElroy mine is located 3 to 4 miles east of the preparation plant, which is on the east side of West Virginia Route 2 along the Ohio River. The mine and preparation plant were constructed sometime in the late 1960's to provide coal for the Mitchell power station of the Ohio Power Company in Captina, West Virginia. All of the coal for Unit No. 1 of the Mitchell plant is obtained from the McElroy mine. Coal from the preparation plant goes across Route 2 on an enclosed overhead conveyor to a coal sampling building owned by Consolidation Coal Company on the power plant property, where samples are obtained for analysis by both the coal and the power company. The layout of the mine, preparation plant, and the Mitchell power plant are shown in Figure D-1. Figure D-2 shows the McElroy preparation plant. Figure D-3 shows the Mitchell Power Plant.

During the past year, a series of ten channel samples were taken from within the McElroy mine. These samples were all taken at different locations, in some instances as much as one or two miles apart from each other. The channel samples were often taken from portions of the coal seam where few impurities were present, resulting many times in raw coal samples that are roughly the equivalent of washed coal. The average sulfur content (dry basis) of the ten channel samples was 4.52 percent with sulfur levels varying from 4.12 to 4.98 percent. Washed coal from the McElroy preparation plant has been averaging from 4.2 to 4.5 percent sulfur. The most recently available data, from February, 1980, indicates that the average sulfur level was running at 4.38 percent (dry basis) during that time. The average moisture content of this coal is 4.7 percent.

The coal sample collected for the Standard Reference Materials program was obtained at the coal sampling building (referred to at the plant as sampling station number 1). The sample was obtained between 9:45 a.m. and 12:05 p.m. on July 30, 1981. Originally, the coal sample was to have been collected earlier in the week, but repair of a broken screen in the preparation plant and mechanical difficulties with the longwall miner in the mine forced postponement of the coal sampling.

Arrangements for the sampling of McElroy coal at sampling station number 1 were coordinated by Bob Knaul, Chief Chemist at the Mitchell power station. Bert Shalek of Consolidation Coal Company, who works in the nearby laboratory shared by the McElroy and Ireland mines, was responsible for making the necessary arrangements with the preparation plant.

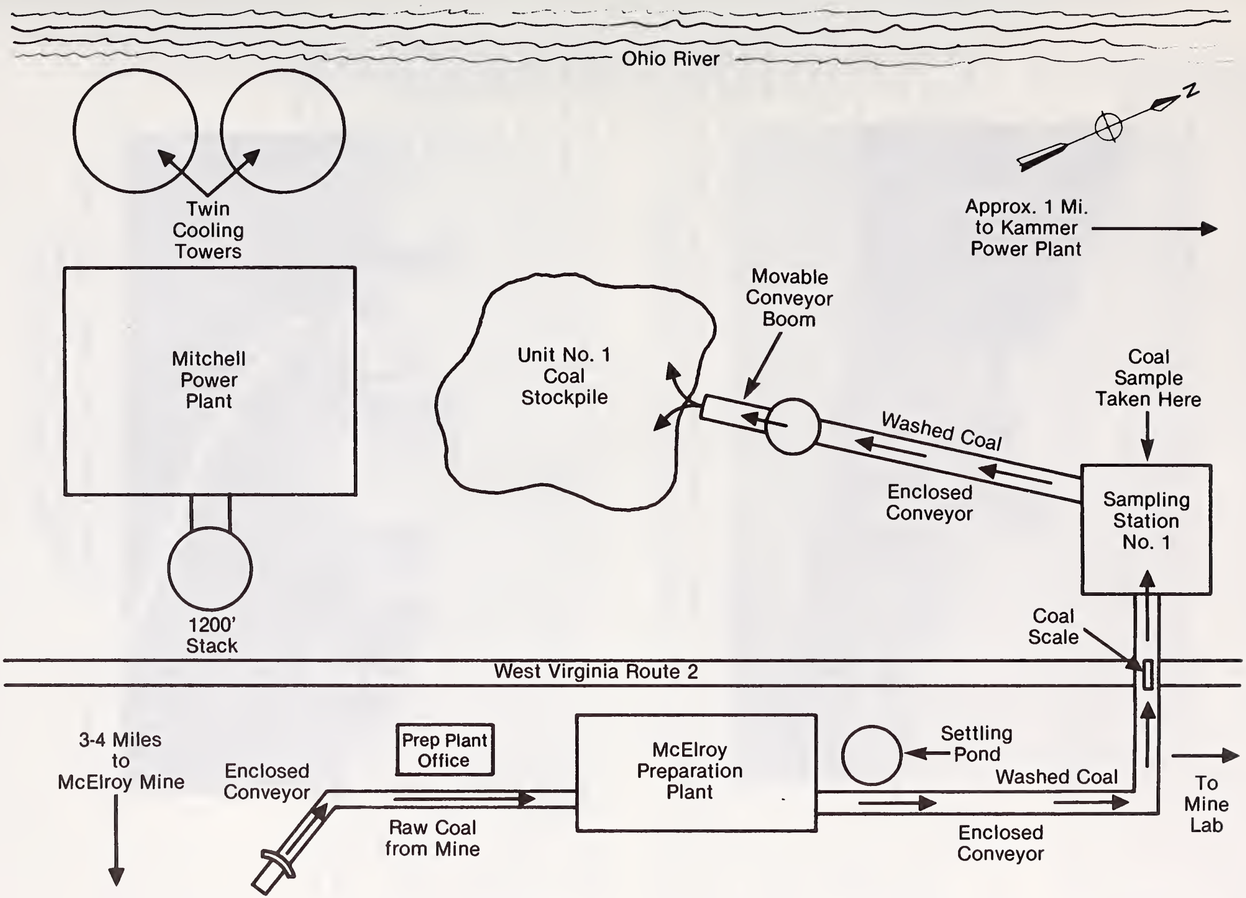

Figure D-1. Layout of Mitchell-Captina Power Plant and Consolidation Coal Company's McElroy Mine, Moundsville, West Virginia

Figure D-2. McElroy Mine Preparation Plant with Coal Storage Silo in Foreground

Figure D-3. Mitchell-Captina Power Plant with Cooling Towers and Conveyor from Sampling Building

The existing coal sampling system consists of a primary sample cutter, which normally takes a full-belt cut every 7-1/2 minutes; a crusher, which reduces the coal from -2" to -4 mesh; a rectangular feed chute with a removable plate for sampling; and a secondary sample cutter for the collection of coal samples for the Mitchell plant and for Consolidation Coal Company. To obtain a sample of the McElroy coal, the removable plate was opened along the bottom of the feed chute during alternate sample cuts and the coal stream was diverted directly by gravity into lined 55-gallon steel drums. The drum samples were collected on the second floor of the sampling station building. Figure D-4 shows a schematic of the existing coal sampling system and how the coal sample was collected at the sampling station.

The normal flow rate of washed coal from the McElroy preparation plant through sampling station number 1 to the stockpile location for Unit No. 1 on the Mitchell plant property is 20 tons per minute. On the day the sample was collected, the longwall miner was still being repaired, but enough coal had been mined during the previous day by two continuous miners to allow the preparation plant to be run at full capacity. Although the continuous miners were also working during the time of sampling, Bert Shalek noted that only a four-hour supply of coal was available. The initial reading of the coal scale at the conveyor before entry into the sampling station was recorded at 61,372 tons when sampling began. At the outset of sampling, a full stream cut was being taken every 7-1/2 minutes and a sample was being collected every 15 minutes. During each sampling interval, approximately half of a 55-gallon drum was filled. Therefore, two samplings were needed to fill a 55-gallon drum and the first two drums required an elapsed time of an hour for sample collection. During drum filling, intermittent samples of the coal stream were also taken and placed into sealed plastic bags, one for each drum.

After the first two drums were filled, concern existed that the coal stream could be unexpectedly cut off before completing coal sampling because of the limited amount of coal available for that day's production. Therefore, an electrician from the mine was summoned and the timing interval on the sample cutter was changed from 7-1/2 minutes to 4 minutes. The coal sampling was still conducted on every other cut so that the mine and the plant could also obtain their samples, but at this point it required only 16 minutes to obtain a full drum sample, compared to 30 minutes each for the first two drums. The last 55-gallon drum was filled at 12:05 p.m. and approximately 15 minutes later the coal supply ran out. The reading on the coal scale immediately after filling the last drum was 63,645 tons. Therefore, a total of 2,273 tons of coals were fed into the sampling station during the time that the six drums were filled. Sulfur analyses were not performed on mine samples of that day's coal by the Consolidation Coal Company's mine laboratory because the Leco furnace at that lab was not operating at the time.

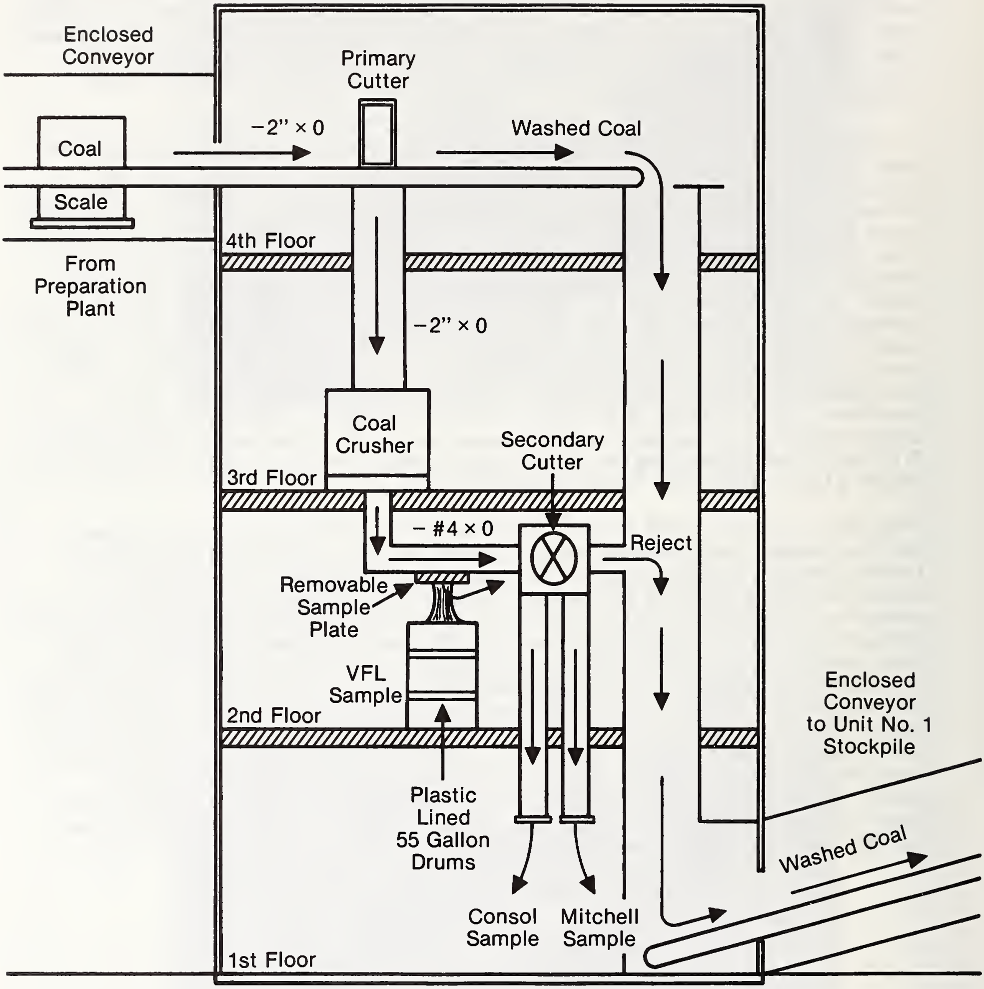

Figure D-4. Schematic Diagram of Sampling Station No. 1 at Mitchell Power Station

The six sealed plastic bag samples were riffle split in the power plant laboratory. One portion of each sample was given to Valley Forge Laboratories in a sealed plastic bag, and part of the other portion was placed in individual 4 ounce plastic bottles for analysis by Tradet, Inc., an independent coal testing laboratory from Wheeling, West Virginia. The samples analyzed by Tradet were pulverized to a -60 mesh size and tested for sulfur content using a Leco furnace model number SC37. The results of the Tradet sulfur analysis tests are:

Sample No.	Percent Sulfur (as received)	Moisture Content (percent)	Percent Sulfur (dry basis)
1	4.50	3.59	4.67
2	4.53	4.02	4.72
3	4.35	3.46	4.50
4	4.48	3.47	4.64
5	4.53	3.52	4.70
6	4.50	3.20	4.65

The average sulfur content of the six samples is 4.65 percent. As very little deviation exists between the sulfur levels of samples taken from any of the six drums, all of these drums were processed in preparation of the -60 mesh Standard Reference Materials.

Following sample collection, the six drums were removed from the second floor of the sampling station and transported (by a Valley Forge Laboratories employee) directly to the coal processing facilities of Alnort, Inc., Camden, New Jersey.

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This publication describes in detail the performance of a grant given to Valley Forge Laboratories, Inc., by the National Bureau of Standards, to obtain and prepare four standard reference coals, with nominal sulfur contents of 0.5, 2.0, 3.0, and 4.5 percent to be issued as SRM's 2682, 2683, 2684, and 2685, respectively. All activities pertaining to the sampling, preparation, packaging, and homogeneity testing of the coal SRM's are documented in this report, including a separate description of each of the four coal sampling activities. Protocols used in the development of these Standard Reference Materials are similar to those used in other NBS SRM preparation procedures to ensure that materials used for SRM's have the highest possible homogeneity and stability.

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