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16. ABSTRACT The report describes a planning study for predicting and monitoring the hydrologic and chemical characteristics of effluent streams resulting from precipitation impacting on open storage of coal. It includes: a survey of utilities on storage habits and treatment systems for coal pile runoff, the development of a runoff model, a work plan to field test the model, and procedures for the field program. The survey includes information from 81 plants on size, shape, and handling practices for coal stocks and criteria used to design coal pile runoff treatment. The model developed in this program is in two sections: a hydraulic model TRCH20 and a quality model TRCCOAL. The model is capable of describing the surface and interior reaction in the coal pile as well as in the groundwater. A sensitivity analysis of several model parameters is also provided.

17. KEY WORDS AND DOCUMENT ANALYSIS

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Pollution	Leaching	Pollution Control	13B	07D,07A
Coal Storage	Field Tests	Stationary Sources	08I	14B
Utilities	Pyrite	Reactive Modeling		08G
Runoff		Non-point Sources	08H	
Water Treatment				
Mathematical Models			12A	

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SECTION 1

INTRODUCTION

In August 1979, TRC began Phase I of a multiphased effort to predict the quantity and quality of coal pile runoff from utility sites. The Phase I effort commenced with an intensive literature search to scope the desired model capabilities, review the most recent developments in treatment and characterization of coal pile runoff, and assess the theoretical research in quantifying and qualifying coal pile runoff. Based on the information gathered in the literature search, TRC outlined a model structure able to predict both volume and quality of drainage from any coal storage area. Along with the model format an outline was developed for the field monitoring program which will provide input as well as verification data to the model.

This report concludes the Phase I effort by presenting the following:

1. The results of the questionnaire sent to members of the Edison Electric Institute in which recent coal pile characteristics and coal pile runoff treatment systems information was gathered. Refer to Section 3.

2. A report on the Model Development task. Refer to Section 4.

3. A detailed Field Work Plan, which itemizes those tasks necessary to describe the physical, chemical and hydrologic characteristics of a coal pile, the meteorological conditions contingent to that pile, and the framework for conducting an intensive runoff sampling program. Refer to Section 5.

4. A Field Procedures Manual which details all the steps necessary to monitor coal pile runoff from the training of field personnel to the analysis of runoff samples. Refer to Section 6.

This report will be used as the foundation for Phase II of the program, the Modeling and Monitoring of Coal Pile Runoff.

SECTION 2

EXECUTIVE SUMMARY

This report on the Phase I - Planning Study to Model and Monitor Coal Pile Runoff is the culmination of approximately ten months of effort to pave the way in predicting and monitoring the hydrologic and chemical characteristics of the effluent streams resulting from precipitation impacting on open storage of coal. Through the continuing interest and technical guidance of the Utility Water Action Group (UWAG) Committee and later the Environmental Protection Agency/Edison Electric Institute Technical Advisory Committee set up to monitor this program, a plan of action has been formulated to study coal pile runoff mathematically using a recently developed predictive model and analytically through actual field testing. The recommendations, observations and criticisms of many of the researchers in previous coal pile runoff field programs have been incorporated into this plan. A major concern of previous investigators has been that individual runoff studies had failed to acquire important data on site meteorology, coal characteristics and pile characteristics so that comparisons of data between different runoff streams could be made. The plan detailed in this report synthesizes these concerns and allows for a program which, when applied nationally, will produce viable data to be used for engineering design of treatment works for coal pile runoff.

The report on Phase I is divided into four major sections, each of which may be considered a separate document in itself.

Section 3 is a Report on the questionnaire circulated to members of the Edison Electric Institute requesting information on their coal stockpiles and treatment systems for runoff. The questionnaire was strictly a voluntary document and the response was noteworthy. A total of 30 utilities representing 81 plants responded. Useful information on the size, shape, slope and handling practices for coal stocks was gathered. Ranges of values for many parameters describing each pile were collected including proximate analysis of the coals, mining origin, cleaning practices and coal storage practices. This data which comprised the first part of the questionnaire was and will be used to determine how the coal pile at a prospective utility site fits into the overall pattern of coal piles. The second part of the questionnaire dealt with the design criteria used for treatment design of coal pile runoff. The information gathered showed that the common practice of designing for the 10 year - 24 hour storm is widely used. Treatment for runoff appears to be aimed at the most economical method for neutralizing the acidity and lowering the suspended solids content. For the TVA system and many others this

means directing all runoff into the ash ponds. Few utilities treat for metals, oil and grease or organics removal. Containment for storm flows are for the majority designed according to the 'Rational Formula'. Also, less than half of the utilities monitor their coal pile runoff and those which do monitor only pH and suspended solids.

Section 4 is the report on the Model Development Task. It describes the physical/chemical phenomena and data requirements needed to describe the quantity and quality of the runoff. Examples of these include precipitation, snowmelt, percolation, pyrite oxidation and gully erosion.

A summary of the model development is included with a description of the Ohio State University Model which served as the basis for the runoff model. TRC modified the OSU model extensively to describe the surface and interior reaction in the coal pile as well as in the groundwater. The final version of this model resulted in a pair of co-models, one describing the hydraulic characteristics of the runoff, labeled TRCH20 and the other describing the quality of the runoff, labeled TRCCOAL. The hydraulic model can be used separately for predicting runoff volumes but the runoff quality model relies on the routines of the hydraulic model for predicting pollutant loadings. The most signficant algorithms of each are discussed in detail. Input requirements for the model as well as example outputs are presented.

This section discusses the sensitivity analysis of selected parameters in both co-models which was conducted to determine the data having the most impact on the results. A discussion is also presented on the model's limitations and shortcomings and its use in treatment design. Finally, the hardware and software requirements to run the model are outlined.

Section 5 presents the Field Work Plan to be used as a guidance document for carrying out the field monitoring program. It details the data requirements necessary for inputs to the model and those data requirements for comparison of runoff from coal piles of various locales and climatic regions. The plan includes the basic scope of work to be followed to collect the data, to format the data and present the data. A general schedule is outlined for conducting the field work along with manpower requirements and anticipated other direct costs. Actual costs were not calculated since these will be contingent on the location of the utility sites.

Section 6 is a Draft Field Procedures Manual that will be revised once the field program has been enacted. As closely as practicable, it itemizes those procedures necessary to carry out a field monitoring program in coal pile runoff. It is intended to be a working document which may be utilized by anyone with a certain amount of technical training in water pollution field monitoring to conduct a field program which will supply comparable data on a particular coal pile. Procedures listed include everything from training of site technicians to field analysis of solids and acidity. Illustrations are presented to assist in the visualization of a monitoring station and field laboratory.

SECTION 3

UTILITY QUESTIONNAIRE SURVEY OF COAL PILES AND RUNOFF TREATMENT SYSTEMS

INTRODUCTION

The Environmental Protection Agency (EPA) in conjunction with the Edison Electric Institute (EEI) has contracted TRC-Environmental Consultants, Inc. to conduct a program to model and monitor the quantity and quality of coal pile runoff from utility sites. As part of Phase I of this project, the planning phase, the necessary data relating to coal types, stockpile sizes, regional meteorology, and control strategies were identified as parameters important to planning a field testing program.

To compile recent (1979-1980) information necessary to both plan a field study and to be used for predictive modeling, TRC conducted an independent poll of coal-burning utilities owning plants with a generating capacity greater than 25 megawatts (MW). TRC compiled the questionnaire to include data pertinent to the design of a field sampling program. Review of the data received will be used in the selection of representative sites for the field sampling program.

In early November 1979, the Edison Electric Institute sent the TRC questionnaire to member utilities and requested a quick response. A copy of the questionnaire is found in Appendix A.

A total of thirty utilities responded to the questionnaire, five of which do not operate coal fired plants and do not store coal on-site. One utility has two plants which do not currently operate coal-fired units, however, they do store coal on-site in the event that they will be required to convert the existing units.

TRC received completed or partially completed questionnaires from eighty-one plants with 109 bituminous coal piles. The quality of completeness of the information was variable, and in data compilation, the number of questionnaires with no response to a specific question was noted.

TRC also received completed questionnaires from three plants burning lignite and one plant burning subbituminous coal. The information supplied by these plants was not used in the tabulations for this report.

TRC will report the information collected from the questionnaires back to the responding companies so that the exchange of technology on coal pile runoff treatment may be continued.

SUMMARY

A total of 81 responses to the coal pile/runoff treatment questionnaire was used in tabulating the data. From the data received, mean plant operating characteristics, coal pile characteristics, and treatment methods were determined.

Of the 81 plants, 19 plants segregate the active and reserve piles. One point of interest was the possible relationship between coal segregation and rotation of the coals. The responses indicate that segregation of the piles does not influence the practice of rotating stored coal.

Coal is compacted at 79 plants. One plant reports compaction of the reserve coal only, while the remaining 78 compact both active and reserve coals. Two plants do not compact the coal. Partial or complete coal cleaning is conducted at 42 plants; 27 plants do not clean the coal; 12 plants did not respond.

The mean reserve coal pile size is 332,400 tons (range 404-$2x10^6$ tons) compared with 249,400 tons (range 2703-$2x10^6$ tons) for the mean active pile. The coal piles at 50 plants are heterogenous mixtures of coals from several states. Coals from 12 states have been reported as sources, with Kentucky coals the most common.

Mean sulfur content of the reserve/non-segregated piles is 1.75% (range 0.45-4.1%); mean sulfur content of the active piles is 1.98% (range 0.48-4.1%). Pyritic sulfur contents of the reserve/non-segregated piles and the active piles are 0.75% (range 0.06-2.05%) and 0.96% (range 0.38-2.05%), respectively.

A variety of treatment systems is employed at the plants, including diversion to ash ponds, chemical or polymer addition, oil skimmers, and flow equalization ponds. The systems at three plants are not yet on-line. Seven plants do not treat coal pile runoff.

Total suspended solids are treated at 59 plants, pH at 53 plants, metals at 19 plants, and oil and grease at 17 plants. Flow equalization is employed at 37 plants. Twenty-eight plants monitor TSS concentrations while 42 plants monitor pH. Other parameters monitored include flow, oil and grease, iron and sulfur. Forty-five plants do not conduct any monitoring of coal pile runoff.

One utility reports that three of its plants store lignite on-site. The plants operate both active and reserve piles; the active piles are not compacted, while the reserve piles are compacted. The coal in the

piles is not cleaned and the plants do not rotate the coal. The reserve piles range in volume from 112,000 tons to 422,380 tons. The live piles range from 7,750 tons to 162,250 tons. The plants have not designed systems specifically for treating the runoff from the lignite piles, however, the coal piles are diked. The runoff from the plant areas is treated for total suspended solids by polymer addition. The three plants do conduct monitoring of runoff and groundwater pH, conductivity, precipitation, iron, total suspended solids, sulfates, chlorides, total dissolved solids and ground water level.

One utility has a plant that burns sub-bituminous coal and maintains both active and reserve piles. The coals in the active piles are not compacted while the coals in the reserve pile are compacted. The coals are cleaned in both the active and the reserve piles. The active storage piles contain 90,800 tons; the reserve pile contains 1.8×10^6 tons of sub-bituminous coal. A treatment system has been designed for coal pile runoff at this plant. It is also used to treat the runoff from ash handling areas. The piles are diked with the runoff directed through Parshall flumes. The runoff is treated for total suspended solids by liquid alum addition. The plant monitors precipitation, runoff pH, temperature, conductivity, and bacterial concentrations.

QUESTIONNAIRE TABULATIONS

General Plant Information

Thirty utilities (comprising 85 plants) responded to the questionnaire. Their regional distribution is shown in Figure 1. Of the thirty utilities, five do not operate coal-fired plants and do not store coal on-site. One utility operates two plants which do not currently burn coal, however, they have coal stockpiles on-site in anticipation of coal conversion requirements. The data from these two plants were used in the tabulations. Not used in the tabulations was the information supplied by the plants burning sub-bituminous and lignite coals.

The tables below contain the information dealing with general plant characteristics, such as generating capacity, coal usage, and coal pile base construction.

TABLE 1. DISTRIBUTION OF GENERATING CAPACITIES OF PLANTS RESPONDING TO THE QUESTIONNAIRE

Capacity (MW)	Number of Plants
<100	4
100 - 500	31
501 - 1000	20
1001 - 1500	13
1501 - 2000	8
>2000	4
No Response	1
Mean = 782 MW	
Range = 35-3160 MW	

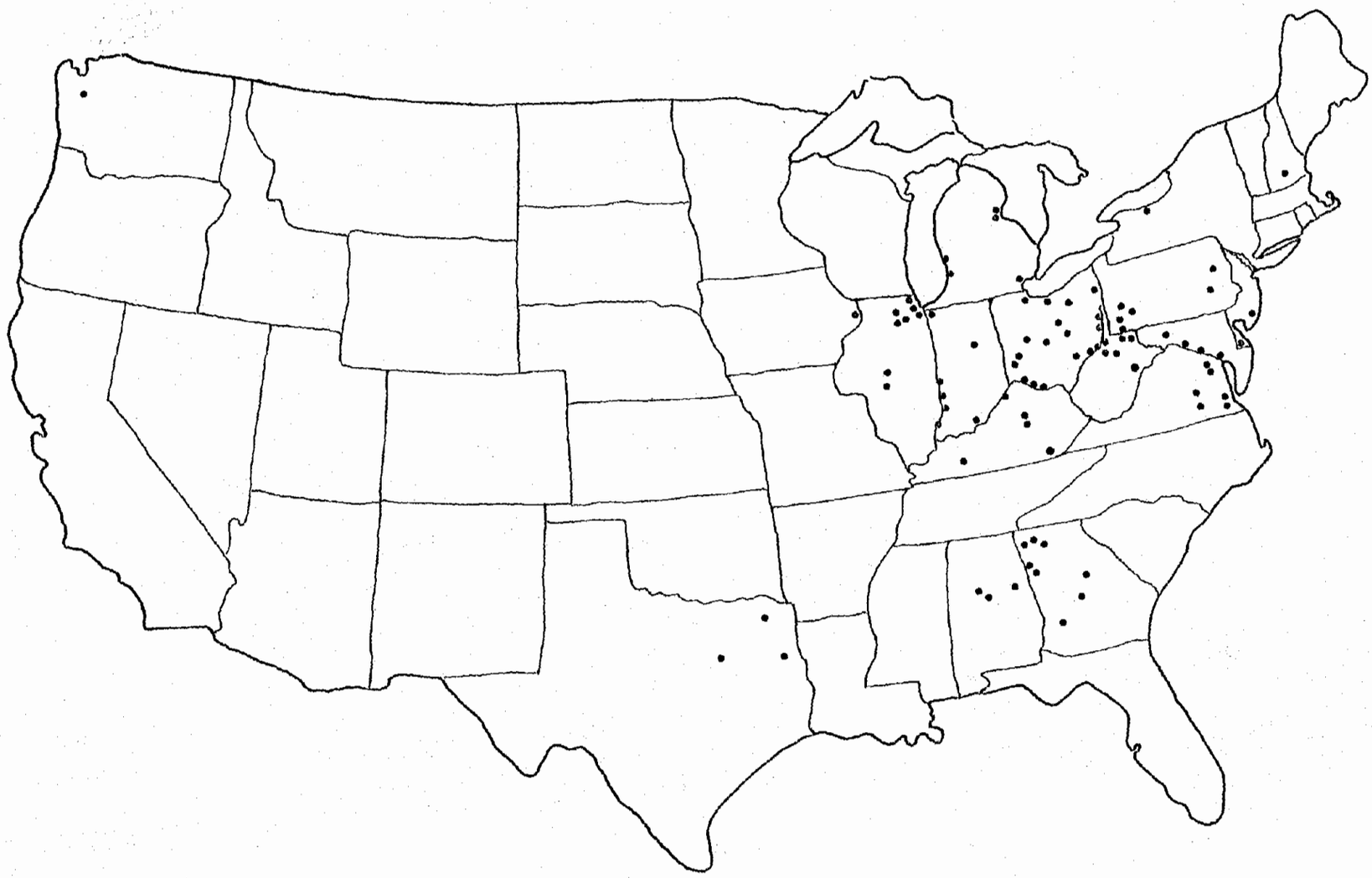

FIGURE 1: PLANTS THAT RESPONDED TO COAL PILE QUESTIONNAIRE

TABLE 2. DISTRIBUTION OF PERCENTAGES OF COAL USAGE FOR ELECTRICITY GENERATION

Coal Usage (%)	Number of Plants
100	51
80 - 99	9
60 - 79	5
40 - 59	2
20 - 39	2
0	2
Variable	1
No Response	9

TABLE 3. DISTRIBUTION OF COAL PILE CONSTRUCTION BASES

Base Type	Number of Plants
Clay	29
Compacted Coal	16
Compacted Ash, Slate, Bony, etc.	10
Compacted Ash	8
Clay & Compacted Coal	5
Compacted Slag	3
Compacted Earth	1
Sand	1
Cinders	1
Clay, Slate, Shale	1
Sand & Gob Coal	1
Gravel	1
Solid Limestone	1
No Response	3

Figure 2 shows the distribution of plant capacity vs tons of coal stored. As can be seen from the figure, the majority of the plants store less than 500,000 tons of coal and their generating capacities are less than 1000 MW.

General Coal Pile Data

Twenty-five utilities operate a total of 110 coal piles at 81 plants. Information was provided for 109 coal piles and organized into non-segregated/reserve or active pile categories. A non-segregated pile

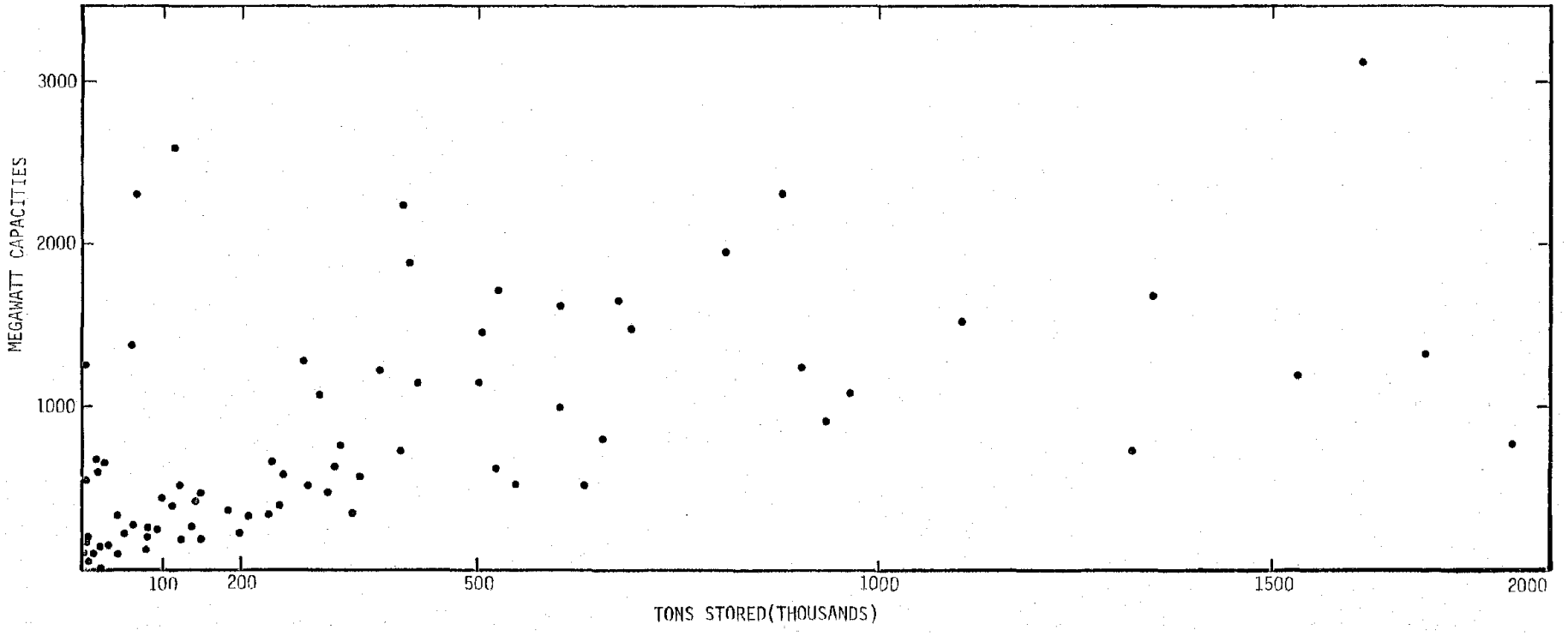

FIGURE 2: PLANT CAPACITY (MW) VS TONS COAL STORED (T)

is one in which both active usage and reserve storage occur within the same pile, rather than maintaining a separate pile for active use and a separate pile for reserve storage.

Eighty-nine piles at 62 plants are non-segregated or reserve piles. Twenty-one piles are live or active at 19 plants. The plants that do not separate active and reserve piles report lack of adequate storage space as a reason for non-segregation. For ten plants, their proximity to the source mine accounts for non-segregation.

Eighty-seven of the 89 reserve piles are compacted; 12 of 20 active piles are compacted. Compaction information was lacking for 21 active coal piles.

The response to coal cleaning is as follows:

Coal cleaned	16 plants
Partial cleaning	26 plants
No cleaning	27 plants
No response	12 plants

Of the plants that segregate their live and reserve piles, 4 plants rotate the coal. Six plants that do not segregate the live and reserve coal practice rotation. This indicates that segregation does not influence the practice of coal rotation.

Reserve/Non-Segregated Coal Piles

Physical Characteristics--

Information relative to coal pile size, volume, and storage duration is necessary for the selection of representative plant sites for the field sampling phase of this study. In addition, coal pile slopes and degrees of compaction (density), will influence the rates and volumes of runoff generated. Tables 4 through 8 summarize the information collected on pile size, volume, density, and other physical characteristics.

Coal Characteristics--

Information on the sources of coal used and stored on-site was requested from the utilities. The degree of specificity varies widely - from no response to the exact seam, county, and state. Only the coal source states were tabulated because of the wide variability in responses.

TABLE 4. DISTRIBUTION OF RESERVE/NON-SEGREGATED COAL PILE SIZES

Size (1000 tons)	Number of Piles
<50	17
50 - 100	13
101 - 500	35
501 - 1000	20
>1000	4
Mean = 332,400 tons	
Range = 404 - $2x10^6$ tons	

TABLE 5. DISTRIBUTION OF RESERVE/NON-SEGREGATED COAL PILE DENSITIES

Density Range ($tons/yd^3$)	Number of Piles
0.1	1
0.1 - 0.49	24
0.5 - 0.89	17
0.9 - 1.19	16
<1.2	11
Mean = 0.80 $tons/yd^3$	
Range = 0.08 - 4.19 $tons/yd^3$	

TABLE 6. DISTRIBUTION OF RESERVE/NON-SEGREGATED COAL PILE HEIGHTS

Height (ft)	Number of Piles
5 - 15	10
16 - 30	29
31 - 50	21
>51 ft	5
Variable Height	11
Conical	1
No Response	13
Mean = 31 ft	
Range = 8.8 - 85 ft	

TABLE 7. DISTRIBUTION OF RESERVE/NON-SEGREGATED COAL PILE SLOPES

Slope	Number of Piles
0.025 - 0.05	11
0.06 - 0.10	5
0.11 - 0.25	5
0.26 - 0.50	8
0.51 - 0.75	6
0.76 - 1.0	16
≥1.0	4
Variable	10
No response	23
Mean = 0.63	
Range = 0.025 - 5	

TABLE 8. DISTRIBUTION OF RESERVE/NON-SEGREGATED COAL STORAGE DURATIONS

Storage Time (Month)	Number of Piles
1 month	1
1 - 1.9	25
2.0 - 2.9	23
3.0 - 3.9	6
4.0 - 4.9	2
5 - 12	1
12	12
Variable	8
No response	11
Mean = 3.9 months	
Range = 1 day to 30 months	

The plants report using coal from 12 states, with Kentucky coal the most common component of the coal piles. West Virginia and Ohio are the next most commonly reported source states. All coal piles are composed of the coal from one or more states. The distribution is as follows:

TABLE 9. DISTRIBUTION OF PLANTS WITH RESERVE/NON-SEGREGATED COAL PILES COMPOSED OF COAL FROM ONE OR MORE STATES

Number of Source States	Number of Plants
1	31
2	15
3	9
4	6
Multiple	6
No Response	14

The following tables list the distributions, ranges, and average values of the various reserve/non-segregated coal characteristics. This information was requested so that data from the future field sampling study can be used to determine whether any correlations exist between the concentrations of pollutants in the runoff and the characteristics of the coal stored.

TABLE 10. SULFUR CONTENT OF RESERVE/NON-SEGREGATED COAL PILES

% Sulfur	Number of Coals
0.45 - 1.0	39
1.1 - 2.0	37
2.1 - 3.0	31
$\geq$3.1	14
No Response	10
Mean = 1.75%	
Range 0.45 - 4.1%	

TABLE 11. PYRITIC SULFUR CONTENT OF RESERVE/NON-SEGREGATED COAL PILES

% Pyritic Sulfur	Number of Coals
0.1	2
0.1 - 0.3	5
0.31 - 0.5	3
0.51 - 1.0	3
$\geq$1.0	7
No Response	109
Mean = 0.75%	
Range 0.06 - 2.05%	

TABLE 12. DISTRIBUTION OF % ASH IN COALS COMPRISING RESERVE/NON-SEGREGATED PILES

% Ash	Number of Coals
4.0 - 9.9	40
10.0 - 14.9	67
≥15.0	14
No Response	10
Mean = 11.0%	
Range 4.3 - 19%	

TABLE 13. DISTRIBUTION OF % MOISTURE IN COALS COMPRISING RESERVE/NON-SEGREGATED PILES

% Moisture	Number of Coals
4.0 - 9.9	55
10.0 - 14.9	12
15.0 - 20.9	6
≥21.0	8
No Response	50
Mean = 10.1%	
Range 4.5 - 25%	

TABLE 14. DISTRIBUTION OF BTU VALUES OF RESERVE/NON-SEGREGATED COALS

Heat Value (BTU/lb)	Number of Coals
9,000 - 9,999	8
10,000 - 10,999	15
11,000 - 11,999	41
12,000 - 12,999	46
13,000 - 13,999	10
≥14,000	1
No Response	10
Mean = 11,700 BTU/lb	
Range 9,000 - 14,000 BTU/lb	

Active or "Live" Coal Piles

Physical Characteristics--

The following tables contain information on the physical characteristics of the active or "live" coal piles, including height, size, density, and slope.

TABLE 15. DISTRIBUTION OF ACTIVE COAL PILE SIZES

Size (1000 tons)	Number of Piles
<50	8
50 - 100	2
101 - 500	5
501 - 1000	4
1000	1
No Response	1
Mean = 249,400 tons	
Range 2703 - 2×10^6 tons	

TABLE 16. DISTRIBUTION OF ACTIVE COAL PILE DENSITIES

Density (tons/yd^3)	Number of Piles
≤0.1	1
0.1 - 0.49	5
0.5 - 0.89	7
0.9 - 1.19	2
≥1.2	-
No Response	6
Mean = 0.63 tons/yd^3	
Range 0.05 - 1.16 tons/yd^3	

TABLE 17. DISTRIBUTION OF ACTIVE COAL PILE HEIGHTS

Height (ft)	Number of Piles
5 - 15	1
16 - 30	6
31 - 50	7
51	2
Variable	-
Conical	-
No Response	5
Mean = 35.8 ft	
Range 13.5 - 80 ft	

TABLE 18. DISTRIBUTION OF ACTIVE COAL PILE SLOPES

Slope	Number of Piles
0.025 - 0.05	1
0.06 - 0.10	-
0.11 - 0.25	3
0.26 - 0.50	3
0.51 - 0.75	6
0.76 - 1.0	5
>1.0	1
Variable	1
No Response	1
Mean = 0.64	
Range = 0.04 - 1.5	

TABLE 19. DISTRIBUTION OF ACTIVE COAL PILE STORAGE DURATIONS

Storage Time (Month)	Number of Piles
1	6
1 - 2	3
3 - 4	3
5 - 9	2
10 - 12	1
No Response	6
Mean = 2.8 months	
Range = 1 day - 12 months	

Coal Characteristics--

The same types of information that were requested on the reserve/non-segregated pile coal were requested for the active or "live" pile coal. The tables that follow summarize the data on coal comprising the active coal piles.

TABLE 20. DISTRIBUTION OF PLANTS WITH ACTIVE COAL PILES COMPOSED OF COAL FROM ONE OR MORE STATES

Number of Source States	Number of Plants
1	7
2	5
3	3
4	-
Multiple	-
No Response	6

TABLE 21. SULFUR CONTENT OF ACTIVE PILE COALS

% Sulfur	Number of Coals
0.45 - 1.0	10
1.1 - 2.0	5
2.1 - 3.0	12
3.1	7
No Response	2
Mean = 1.98%	
Range = 0.48 - 4.1%	

TABLE 22. PYRITIC SULFUR CONTENT OF ACTIVE PILE COALS

% Pyrite Sulfur	Number of Coals
≤ 0.1	-
0.1 - 0.3	2
0.31 - 0.5	-
0.51 - 1.0	1
> 1.0	4
No Response	28
Mean = 0.96%	
Range = 0.38 - 2.05%	

TABLE 23. DISTRIBUTION OF % ASH IN COALS COMPRISING ACTIVE PILES

% Ash	Number of Coals
4.0 - 9.9	7
10.0 - 14.9	25
> 15.0	2
No Response	3
Mean = 14.3%	
Range = 4.6 - 17.7%	

TABLE 24. DISTRIBUTION OF % MOISTURE IN COALS COMPRISING ACTIVE PILES

% Moisture	Number of Coals
4.0 - 9.9	14
10.0 - 14.9	4
15.0 - 20.9	1
≥21.0	2
No Response	6
Mean = 10.4%	
Range = 4.6 - 23.96%	

TABLE 25. DISTRIBUTION OF BTU VALUES OF ACTIVE PILE COALS

Heat Value (BTU/lb)	Number of Coals
9,000 - 9,999	2
10,000 - 10,999	6
11,000 - 11,999	11
12,000 - 12,999	12
13,000 - 13,999	1
≥14,000	1
No Response	4
Mean = 11,640 BTU/lb	
Range = 9,000 - 14,000 BTU/lb	

Coal Pile Runoff Treatment System Design

Of the 85 plants that responded to the questionnaire, 72 plants report having some type of coal pile runoff treatment system. The tables that follow summarize the reported information on treatment system types, basis for design, date of system startup, parameters treated, etc.

As can be seen from the tables presented above, a number of systems are employed for runoff treatment. One-half of the plants that responded (41 plants) have separate treatment systems for coal pile runoff. Twenty-five plants combine coal pile runoff with other plant discharges prior to treatment. Fifteen plants did not respond to this question.

At 27 plants, one type of treatment is employed, diversion of the runoff to an ash pond. At 47 plants a combination of treatment methods are used. These combinations include chemical addition and diversion to a sedimentation pond, oil skimmer, chemical addition, and diversion to a sedimentation pond.

TABLE 26. DISTRIBUTION OF PLANTS BY TREATMENT METHODS EMPLOYED

Treatment Type	Number of Plants
Sedimentation Pond	31
Dikes or Ditches	38
Ash Pond Only	26
Chemical Addition	28
Ash Pond and Other Treatment	10
Equalization Pond	9
Polymer Addition	4
Clarifier	5
Oil Skimmer	15
Filtration	4
Percolation Pond	1
Centrifugation	1
Sump	10
Grit Chamber	4
No Treatment	7

TABLE 27. DISTRIBUTION BY YEAR OF TREATMENT SYSTEM START UP

Year	Number of Plants
1967 - 1976	6
1977	37
1978	3
1979	13
No Response	12
No Treatment	7
Not yet in Service	3

TABLE 28. DISTRIBUTION OF DESIGN STORM CRITERIA

Storm Type	Number of Plants
10 yr - 24 hr	50
100 yr	1
1 yr - 24 hr. and 10 yr - 24 hr	1
No Treatment	7
No Response	22

TABLE 29. DESIGN STORM REFERENCES

Reference	Number of Plants
National Weather Service Technical Paper #40	24
Local Data	13
Other	5
No Response	39

TABLE 30. DISTRIBUTION OF RUNOFF COEFFICIENTS (FROM RATIONAL FORMULA) USED IN TREATMENT SYSTEM DESIGN

Runoff Coefficient	Number of Plants
0.2	1
0.25	1
0.55	1
0.7	1
0.73	2
0.75	12
1.0	3
0.1 - 1.0	1
0.5 - 1.0	3

TABLE 31. NUMBER OF PLANTS TREATING SPECIFIED CHEMCIAL PARAMETERS

Parameter	Number of Plants
Total Suspended Solids	60*
pH	53**
Oil and Grease	17
Metals	19
Flow Equalization	37
Organics	0

*7 Plants treat only total suspended solids
**4 Plants treat only pH

TABLE 32. NUMBER OF PLANTS AND THE PARAMETERS MONITORED

Parameter	Number of Plants
Total Suspended Solids	25
pH	38
Flow	21
Oil and Grease	17
Other*	11
No Monitoring	45

*Other includes - bacterial populations, iron, sulfur, ground water level, ground water quality

CONCLUSIONS

It is apparent that, to meet the effluent limitations for pH and suspended solids, most utilities have incorporated some form of treatment to their runoff from coal piles. From the responses to the questionnaire, it is shown that 85% of the plants responding have some form of treatment. Recently obtained information for the 12 Tennessee Valley Authority coal fired plants is not included in the summaries.

The design criteria for collecting the coal pile runoff for the majority of plants is the 10 year - 24 hour storm taken from either local data or the National Weather Services' Technical Paper #40. By using the 'Rational Formula' the runoff coefficients for these plants has been estimated. The Rational Formula relates rainfall and peak runoff. The formula is $Q = CIA$, where Q is the peak discharge (cfs), C is a runoff coefficient depending on drainage basin characteristics, I is rainfall intensity (inches/hour), and A is drainage area (acres).

The above facts clearly indicate that most utilities are relying on unsophisticated and untested techniques to design a coal pile runoff treatment system. Once the proposed program to actually field monitor and model the quantity and quality of runoff begins, the historical criteria for runoff treatment design will be challenged. With accurate field monitoring of precipitation, runoff flow, runoff coefficients and runoff quality, the field data will be compared directly with the historic design criteria. It is anticipated that the field testing will take place at one or more sites which have been included in this survey and have treatment systems already installed. This program should begin in the fall of 1980.

SECTION 4

COAL PILE RUNOFF MODEL

INTRODUCTION

In August 1979, TRC began Phase I of an intensive two to three year effort to predict the quantity and quality of coal pile runoff from utility sites. The thrust of the program is to develop and validate mathematical modeling techniques that could be used to characterize coal pile runoff in a dynamic model; i.e., predict the quantity and quality of coal pile runoff within a storm event. This simulation can then be used to design a treatment system (Best Management Practice) based on the changing conditions of the runoff during the storm. Recent studies[1] completed by TRC indicate that during a 10-year, 24-hour design storm the initial coal runoff is "dirty" but after a few hours the runoff becomes "clean". Therefore, why not store and treat the "dirty" water and bypass the "clean" water. The model can also be modified to characterize the runoff from other material storage piles.

The program is divided into two phases. Phase I is to scope the desired model capabilities, to initiate model development, and to design a field measurements program that will acquire representative data for model modification and validation. Phase II is to conduct the field program and to modify and develop the model to reflect the field measurement program results. Phase I was initiated in August 1979.

This section of the Phase I report outlines the development of the Coal Pile Drainage (CPD) Model by TRC. Discussed in the report are:

1) Physical/chemical phenomena included in the CPD Model
2) The base model used and modifications made
3) The sensitivity analysis of model parameters
4) Shortcomings/limitations of the CPD Model
5) Field data required by the CPD Model
6) Suggestions for using the output in treatment system design

PHYSICAL/CHEMICAL PHENOMENA TO BE ADDRESSED IN THE COAL PILE DRAINAGE MODEL

In 1977 TRC completed a study of sampling and modeling of non-point sources at a coal-fired utility. In this application, TRC utilized the

Short Stormwater Management/RECEIV-II Model (SSWMM-RECEIV-II) to address sheet washoff from coal storage piles. At the completion of this modeling program TRC identified a number of shortcomings in the SSWMM-RECEIV-II Model. For example, the model could not address:

1) storm erosion of material from the coal storage pile
2) stormwater percolation through the coal pile
3) pyrite oxidation/acid production in the coal pile

As part of Phase I TRC undertook an extensive literature search of coal pile drainage. The universities, institutes, and libraries contacted are listed in Table 33; particularly valuable was the Bituminous Coal Research Library in Monroeville, Pennyslvania. In this search TRC determined what physical/chemical phenomena associated with coal piles have been researched and would be valuable in characterizing coal pile drainage in a modeling effort. The phenomena can be divided into quantitative and qualitative aspects.

Quantitative Phenomena

The quantitative pheonomena addressed by the model are:

1) Precipitation (rain/snowfall). The model should require several years of meteorological data or selection of a design storm(s) for use in runoff simulation.

2) Surface Runoff/Infiltration. The model should have mechanisms to address infiltration into the coal pile as well as runoff from the surface. While some urban runoff models consider the phenomena of exponentially decreasing infiltration, this is not thought appropriate to coal storage piles.

3) Pile Percolation/Interflow/Moisture. While many previous modeling efforts have been concerned with only the surface of a watershed, the percolation of rainfall and snowmelt through a material storage pile is also significant. This internal flow is responsible for eruptions from the sides of the coal pile as well as drainage from the base of the pile. These flows usually continue after the precipitation has ceased and usually carry a more concentrated stream of pollutants than does surface runoff. Some moisture is retained in the coal storage pile on a continual basis. TRC evaluated models to address these phenomena.

4) Snowmelt. While snowmelt is more significant in the runoff pattern of large watersheds, it should also be considered for coal storage piles in the northern climates. TRC researched

TABLE 33. LIBRARIES, UNIVERSITIES, AND OTHERS CONTACTED BY TRC FOR COAL PILE DRAINAGE STUDY

1) Appalachian Resources Project
University of Tennessee
Environmental Center
Knoxville, TN

2) Atlantic Research Corp.
Alexandria, VA

3) Canadian Institute of Mining
Montreal, Quebec, Canada

4) Coal Research Bureau
University of West Virginia
Morgantown, W. VA

5) Army Corps. of Engineers
Hydrologic Engineering Center
Davis, CA

6) National Coal Association
Washington, DC

7) National Coal Board
London, England

8) NYSEG
(New York State Electric & Gas)
Lansing, NY

9) Oak Ridge National Laboratory
Environmental Sciences Division
Oak Ridge, TN

10) Ohio State University
Water Resources Center
Columbus, OH

11) SCS (Soil Conservation Service)
Mansfield, CT

12) Tennessee Valley Authority
Chattanooga, TN

13) Tennessee Valley Authority
Kingston, TN

14) University of Kentucky
Lexington, KY

15) U.S. Bureau of Mines

16) U.S. EPA
Industrial Environmental Research Laboratory
Extraction Technology Branch
Cincinnati, OH

17) Bituminous Coal Research Library
Monroeville, PA

several snowmelt routines to be incorporated in a runoff model which would not require extensive input data.

5) Ground Water Infiltration. Some rainfall and snowmelt percolate through the coal pile into the ground water. The amount of rainfall entering the ground water depends on several factors including the permeability of the material underlining the coal pile. In addition, when the water table rises to the surface, it impacts the flow at the base of the pile. The model should, therefore, consider ground water.

Qualitative Phenomena

A number of chemical and physical reactions occur in the coal pile which affect the characteristics of the runoff. The simulation of these phenomena, as discussed below, is important:

1) Pyrite Oxidation/Acid Production

During non-rainfall periods pyrite in coal, especially framboidal pyrite, oxidizes, producing acid, iron, and sulfates. The acid further reacts, dissolving trace materials in the coal. During rainfall events or snowmelt, dissolved materials which have accumulated are distributed to the direct runoff, interflow through the pile, baseflow, and ground water.

2) Freeze-Thaw Factor

During alternating cycles of freezing and thawing coal lumps expand, contract, and subsequently break up, exposing more reactive surface area. The freeze-thaw cycles subsequently increase the production of dissolved materials which characterize the runoff.

3) Gully Erosion

Coal piles are subject to gully erosion and the subsequent transport of solids into the runoff stream. The volume of coal solids detached and deposited at the pile base is related to the slope of the coal pile, the slope length, and rainfall intensity and duration, as well as a coal erosion coefficient.

4) Coal Pile Washoff

A portion of the accumulated dissolved materials washes off immediately during rainfall by the direct runoff. Some of the base load of pollutants also remains. The model user should be able to select a linear or exponential decay function to express the washoff phenomena.

COAL PILE DRAINAGE MODEL DEVELOPMENT

TRC researched the existing runoff models to ascertain which one could best be utilized as the basis for TRC coal pile drainage simulation effort. Few models have been developed to describe industrial stormwater runoff situations and none specifically for material storage piles. A description of the more pertinent models researched is shown below:

1) Stormwater Management Model (SWMM)[2]

SWMM simulates single or multiple storm events on the basis of rainfall hyetographs and a spatial description of the catchment and sewer routing system. The model was developed under EPA to represent the quantity and quality of urban runoff. The current version of SWMM, "Release 2", includes an additional component for erosion prediction using the Universal Soil Loss equation. TRC also modified a version of SWMM to address the washoff from coal piles. Although SWMM does address the sheet washoff phenomena adequately, it cannot address percolation through a pile or the chemical reactions that take place in the pile.

2) Storage, Treatment, Overflow, and Runoff Model (STORM)[3]

This runoff model was developed by Water Resources Engineers, Inc., for the U.S. Army Corps of Engineers. Its primary purpose was to aid in the selection of storage and treatment facilities to control the quantity and quality of runoff. STORM is a continuous simulation model and has subroutines for snowmelt and land erosion, but has the same shortcomings as SWMM in relation to handling storage piles.

3) Agricultural Runoff Management Model (ARM)[4]

The model was developed by Hydrocomp, Inc. to simulate runoff, snow accumulation and melt, sediment loss, pesticide-soil interactions, and soil nutrient transformations from agricultural lands. ARM simulates sediment transport through rill and sheet erosion but not gully erosion as may be found on coal piles.

4) Pyritic Systems[5]

The EPA pyritic systems model simulates acid mine drainage in underground mines. While the model addresses acid production and removal, it is limited in scope and has little applicability to coal pile drainage.

5) Ohio State Model[6]

The OSU version of the Stanford Watershed Model was published initially in 1972 and updated several times. The OSU watershed model simulates the hydrologic cycle of a rural area pictured in Figure 3. Features are included in the model for interception

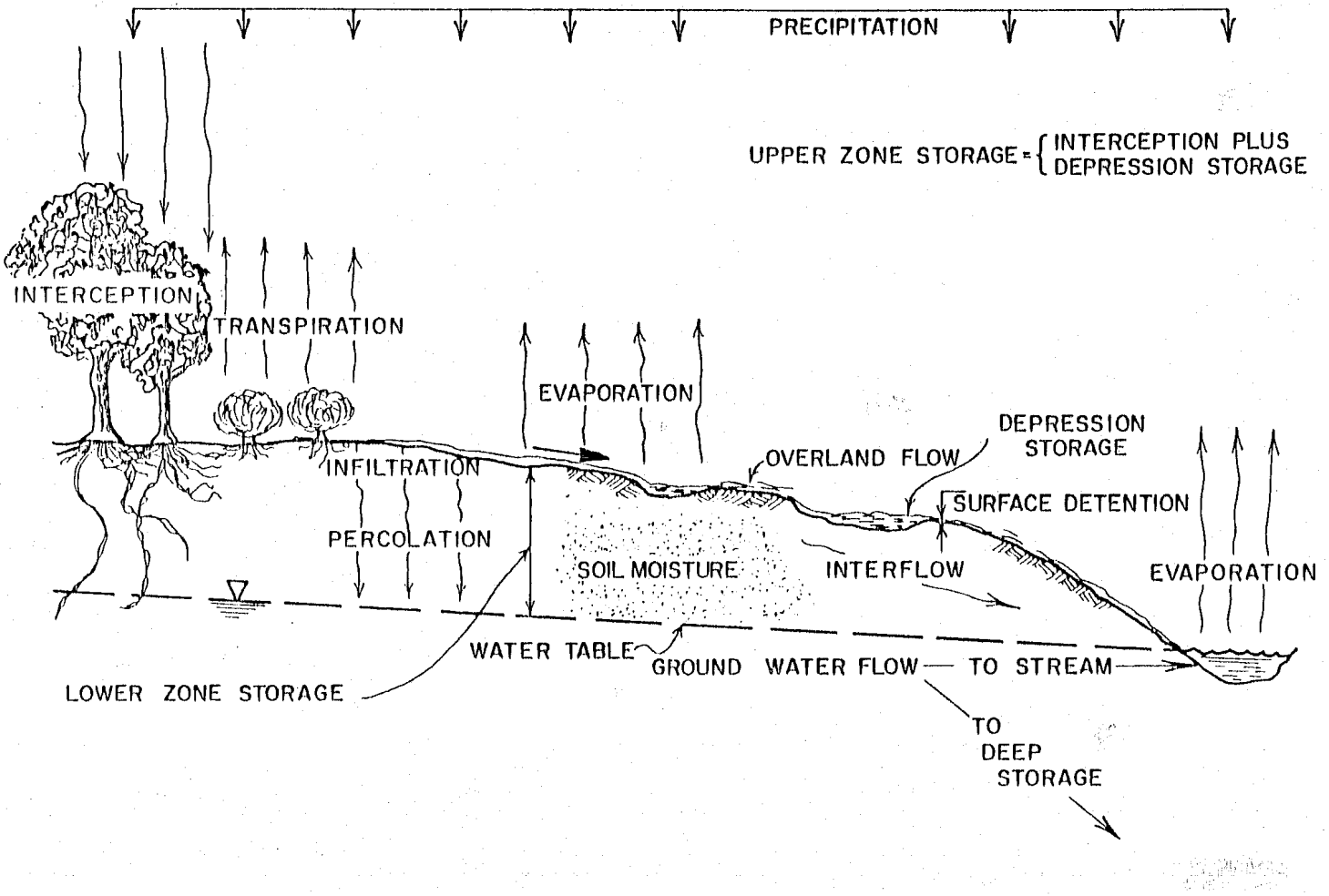

FIGURE 3: SCHEMATIC OF HYDROLOGIC CYCLE, OSU VERSION OF STANFORD WATERSHED MODEL

of rainfall by vegetation, transpiration, infiltration into the soil, percolation, evaporation, overland flow, soil moisture, depression storage, and ground water flow to deep storage. All runoff is directed to a stream channel.

While the OSU version of the Stanford Watershed Model considers hydrology, it does not simulate the chemical constituents associated with runnoff. For coal applications, OSU developed qualitative strip mine and refuse pile models to be used in conjunction with the OSU version of the Stanford Watershed Model. The surface of the pile is considered the upper zone. Rainfall percolates into a lower zone, the interior of the pile. From the lower zone the flow is routed toward deep ground water storage or to the stream channel. The only chemical parameter simulated in the OSU model is acidity.

The OSU models use a magnetic tape or data cards of several years of hourly site meteorological data as input. This input must be developed by the user.

TRC selected the Ohio State University (OSU) version of the Stanford Watershed Model and the Ohio State University co-model for coal refuse piles as a basis for TRC's coal storage pile developmental work.

TRC utilized the 1977 version, made available through the Extraction Technology Branch of the U.S. EPA.

MODIFICATIONS TO THE OSU MODELS

OSU Version of Stanford Watershed Model

While TRC utilized the OSU model as a base for its model development, a number of modifications were made. These are summarized below:

1) Deletion from the watershed model of all calculations related to the interception of rainfall by vegetation

2) Deletion of evapo-transpiration by vegetation

3) Substitution of a less complex snowmelt routine requiring less input data and using hourly temperature data

4) Deletion of lake evaporation

5) Deletion of streamflow diversion

6) Inclusion of a routine to count the number of freeze-thaw cycles

7) Deletion of routines related to impervious surfaces

8) Calculation of average daily rainfall and average daily air temperature

9) Deletion of several output options

10) Addition of the simulation of gully erosion on the coal pile

Refuse Pile Model

For the refuse pile model, the following modifications were made:

1) Addition of the simulation of pyrite oxidation and acid production with the subsequent release of dissolved iron, sulfate, and trace materials during rainfall events

2) Addition of an exponential decay function as an optional method of simulating the pollutant washoff from the surface of the coal pile

3) Use of the number of freeze-thaw cycles during the preceding dry periods to escalate the production of dissolved pollutants

4) Consideration of the acidity of rainfall and alkalinity of coals

5) Use of a variable time interval for wet weather modeling

6) Plotter output of qualitative data

As a major modification, TRC altered the model so that a standard National Climatic Center (NCC) magnetic tape #CD144 could be utilized for the input of hourly meterological data. These tapes are available for most U.S. Weather Service Stations from the NCC for under $100 (Spring 1980).

TRC's versions of the OSU coal storage pile models are termed:

1) TRCH2O - hydrologic model
2) TRCCOAL - qualitative model

They are described in detail in the following sections.

TECHNICAL ASPECTS OF THE TRCH20 HYDROLOGICAL MODEL FOR COAL PILE DRAINAGE

The coal storage pile is viewed in the model as a small unvegetated watershed with many features similar to larger watersheds. A schematic diagram of the hydrologic cycle of a coal pile is shown in Figure 4.

Coal Pile Responses to Precipatation

Surface Phenomena--

The TRCH20 model reads hourly precipitation data in the form of rain or snow from the input meteorological tape. The phenomena of direct infiltration, gully erosion, delay infiltration, direct runoff, snow accumulation, and snowmelt are hydrologic surface reactions to precipitation.

The amount of rainwater which immediately enters the pile is known as direct infiltration. It is dependent upon input factors of pile moisture and pile surface moisture storage capacity as well as time and adjustment factors. Some rainwater is retained by depressions in the coal pile surface and this infiltration is delayed. The depression storage is considered to be the upper zone of the coal pile, and the amount of depression storage on the pile surface is estimated by the model user. The entire coal pile is considered pervious.

The equations in the model for infiltration are developed below:

1) Direct Infiltration

D4F = Current peak infiltration rate

D4F = FRAC * CB/2.0**LNRATM (4-1)

FRAC = Selected routing interval (TINC) converted to fraction of an hour

CB = infiltration index

LNRATM = pile moisture index used in estimating current infiltration rate. It is calculated from ratio of pile moisture to pile moisture storage index, LNRAT

2) Delay Infiltration

PRE = Fraction of incoming moisture that is not retained in upper zone storage

PRE = (1.0/1.0 + UZI) ** UZI (4-2)

UZI = Intermediate pile surface moisture storage parameter

UZI = 2.0 * ABS (UZS/UZSN - 1.0) + 1.0 (4-3)

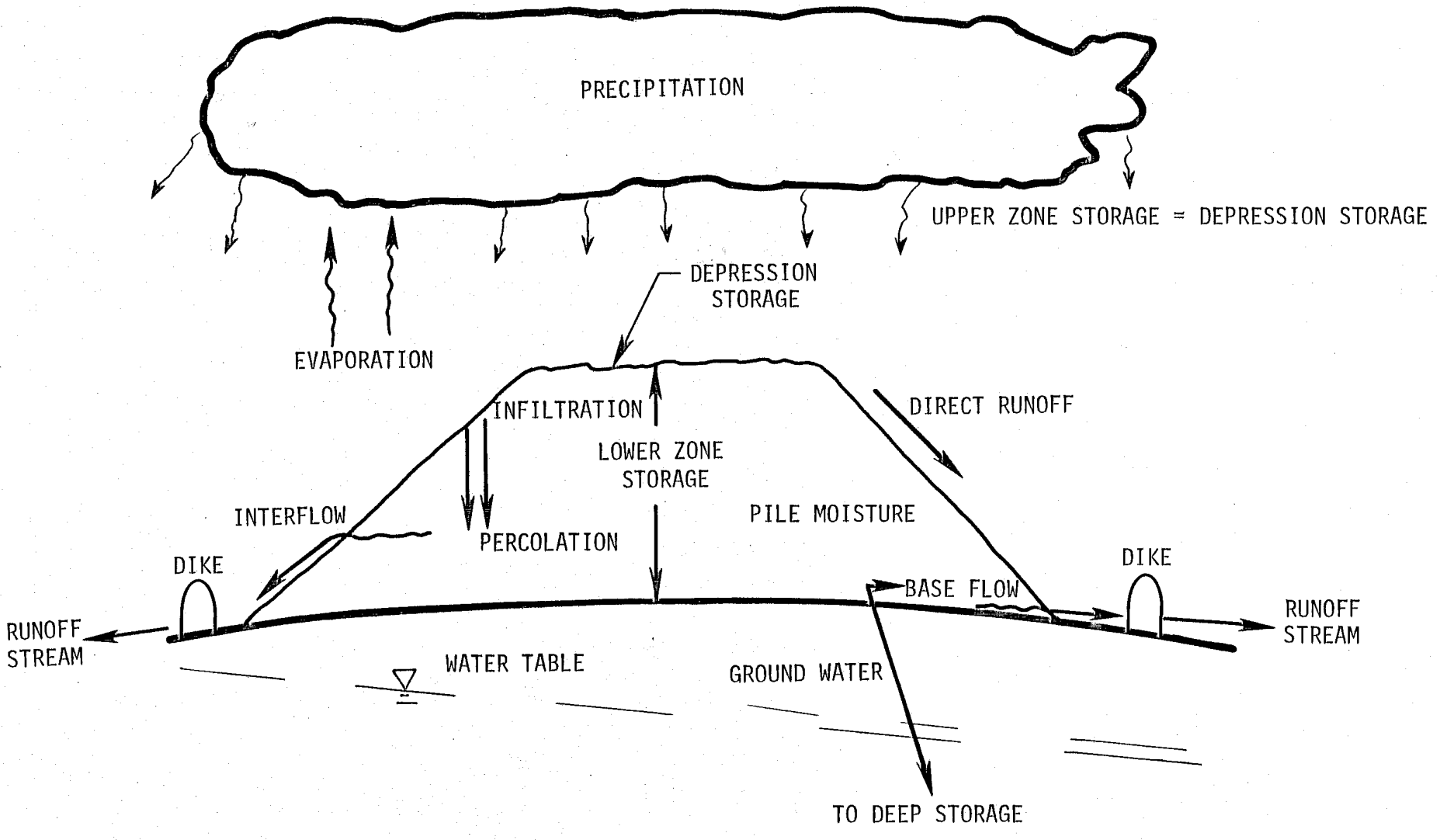

FIGURE 4: SCHEMATIC OF HYDROLOGIC CYCLE COAL STORAGE PILE

UZSN = Pile surface moisture index

UZSN = CX * EXP (-2.7 * LNRAT) (4-4)

CX = Index for estimating pile surface moisture storage

LNRAT= Current ratio of pile moisture to pile moisture storage index

LNRAT= LZS/LSZN (4-5)

LZS = Current pile moisture storage

LZSN = Pile moisture storage index

P4 = rainfall that is depleted from pile surface

P4 = PR * PRE (4-6)

PR = Current rainfall

PRE = See equation 4-2

UZS = current pile surface moisture storage

UZS = UZS + PR - P4 (4-7)

The rainwater which does not infiltrate into the coal pile becomes direct runoff. The Chezy-Manning equation for turbulent flow was utilized in the model to derive this relationship. Input variables are slope of the coal pile, length of the slopes, and a roughness coefficient.

Based on the Manning equation, the overland flow discharge off the coal pile is:

$$q = \frac{1.486}{n} y^{5/3} S^{1/2} \quad (4-8)$$

Where q is flow in $ft^3/sec/ft$, S = slope in ft/ft, and where y is the flow depth in feet at the base of the coal pile.

In addition to rainwater, the model considers the effect of snow on the coal pile. The model reads the meterological tape and determines the amount of snow which falls on the coal pile. The snow pack accumulates over time, and snow is converted to its liquid component by snowmelt. In this model snowmelt is estimated by the air temperature above freezing and a degree-day melt factor. Once snow is melted, it is handled in the model as rainwater for infiltration and runoff purposes.

The snowmelt equation developed from work by J. A. Anderson[7] and used in the model is:

SMELT = RMELT * (TEMPH-32) (4-9)

SMELT = snowmelt, inches
RMELT = degree day melt factor
TEMPH = hourly air temperature, °F

The impact of rain on the coal pile causes solids to erode and creates gullies on the side of the coal pile. Using measured data on pile slope, length, and rain intensity, as well as erosion coefficients, the Foster-Meyer equation[8] calculates the pounds of coal solids moved to the base of the pile per day during a day with rainfall.

The formulations based on the Foster-Meyer erosion equation are described below:

GFXW = lb/day of eroded coal solids at base per foot of gully width

GFXW = TRANSK*C1*(1.-(1.-L*SOILK/TRANSK*(P1SUM(I)/12**2.0)/C1)L*DETACH/
TRANSK) *EXDEC (4-10)

TRANSK = transport capacity coefficient
SOILK = coal surface constant
P1SUM = total daily rainfall, inches
DETACH = detachment coefficient
C1 = YDEPTH * SS ** 1.5 (4-11)

YDEPTH = depth of runoff flow, feet
SS = slope of coal pile, feet per foot
EXDEC = 1. - EXP (-L*DETACH/TRANSK) (4-12)

Interior Phenomena --

The moisture which percolates from the upper zone of the pile is stored in the lower zone. Some of the moisture erupts from the side of the coal pile and is termed interflow. In addition, some moisture percolates through the pile to the ground water. The amount of rainwater which emerges as interflow is proportional to the amount of rainwater which infiltrates from the surface, the current in-pile moisture storage, and adjustment coefficients. These equations for interflow and in-pile moisture are given below:

1) <u>In-Pile Moisture</u>

RECE = current rate of pile surface moisture infiltration
RECE = 0.003*CB*UZSN*DEEPL**3.0 (4-13)

CB = infiltration index
UZSN = pile surface moisture index
DEEPL = (UZS/UZSN) - (LZS/LZSN) (4-14)

UZS = current pile surface moisture storage
UZSN = pile surface moisture index
LZS = current pile moisture storage
LZSN = pile moisture storage index

The fraction of moisture that infiltrates from the upper zone and is retained in the lower zone is computed by:

PRE = fraction of incoming moisture retained in pile storage
PRE = (1.0/1.0 + LZI))**LZI (4-15)

LZI = intermediate pile moisture parameter for estimating infiltration
LZI = 1.5*ABS (LNRAT - 1.0) + 1.0 (4-16)

LNRAT = current ratio of pile moisture storage to pile moisture storage index

2) Interflow

C3 = variable controlling entry of moisture into interflow
C3 = CY*2.0**LNRAT (4-17)

CY = interflow index
LNRAT= current ratio of pile moisture storage to pile moisture storage index

Outflow from interflow storage is computed by:

INTF = current rate at which interflow is entering runoff channel
INTF = LIRC4 * SRGX (4-18)

LIRC4 = natural logarithm of interflow recession constant
SRGX = current volume of water in interflow storage

Ground Water Phenomena --

While some water is retained in the coal pile, some infiltrates to the interface that consists of the layer immediately beneath the coal pile. For the purposes of the model, moisture in this layer is termed ground water, and moisture below the interface is deep storage. The moisture into ground water can be routed to deep storage or emerge from the base of the pile as a flow stream. The amount of seepage or baseflow from the pile is dependent upon the ground water storage, the ground water slope, and flow recession constants. Infiltration into the ground water increases the slope and the resulting base flow emerging from the coal pile.

Percolation to deep, inactive ground water storage or ground water flow out of the basin is modeled by allowing a fixed portion of inflow to ground water storage to bypass the active storage that contributes to seepage. The equations for ground water flow are given below:

GWF = Baseflow
GWF = SGW * LKK4 * (1.0 + LKV4 * GWS) (4-19)

SGW = Ground water moisture storage
SGW = SGW + F1 (4-20)

F1 = infiltration reaching ground water from lower zone storage
F1 = (1.0 - PRE) * (P4 - SHRD) * (1.0 - K24L) (4-21)

PRE = see equation 4-2
P4 = see equation 4-6
SHRD = sum of current moisture entering surface runoff plus interflow
K24L = parameter indicating ground water entering deep storage

F1 = infiltration reaching ground water from the upper zone
F1 = (1.0 - PRE) * RECE * (1.0 - K24L) (4-22)

RECE = see equation 4-13
LKK4 = natural logarithm of hourly base flow recession constant
LKV4 = natural logarithm of hourly base flow recession adjustment factor
GWS = current value of ground water slope index

A block diagram of the TRCH20 model is shown in Figure 5.

Input Data Requirements of TRCH20

The data required to run the hydrologic model TRCH20 consists of three parts:

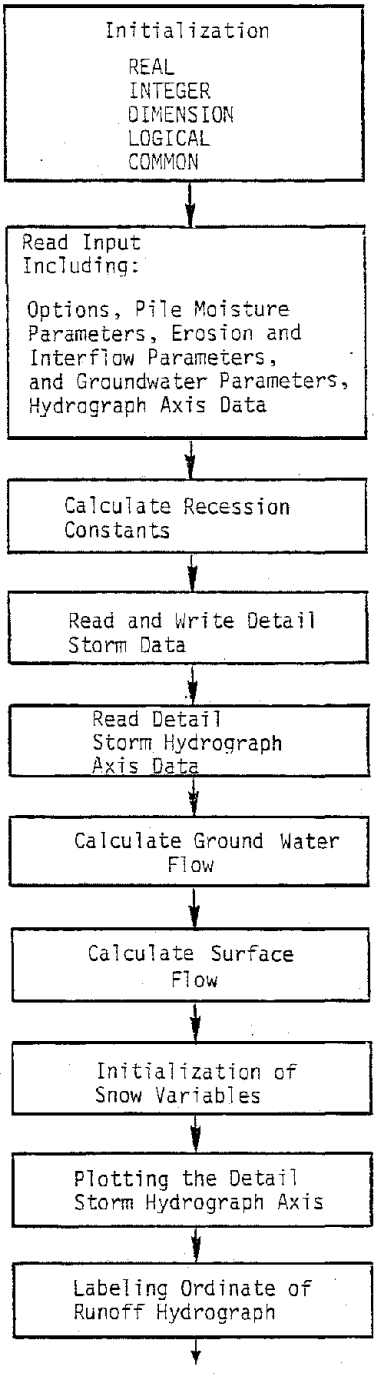

FIGURE 5: BLOCK DIAGRAM FOR TRCH20

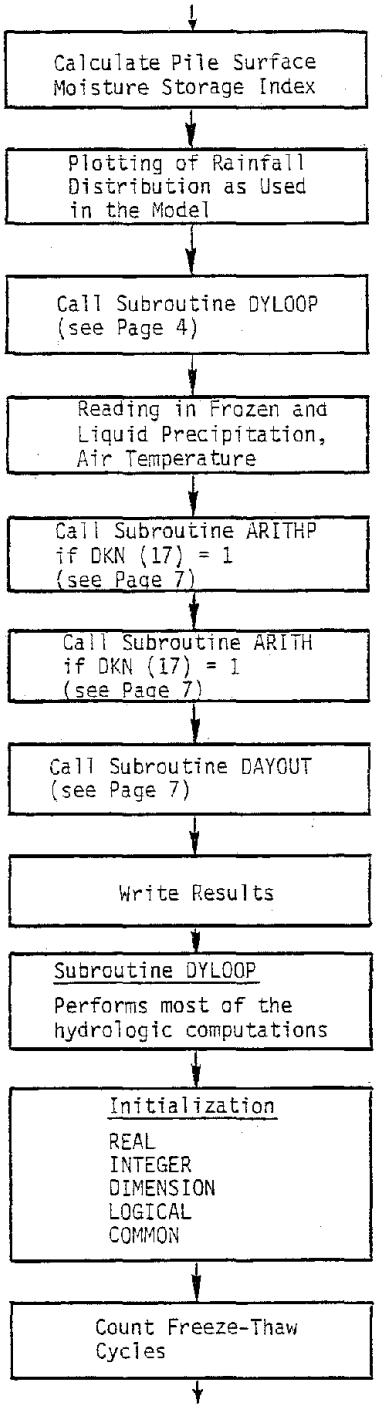

FIGURE 5: BLOCK DIAGRAM FOR TRCH20 (CONT.)

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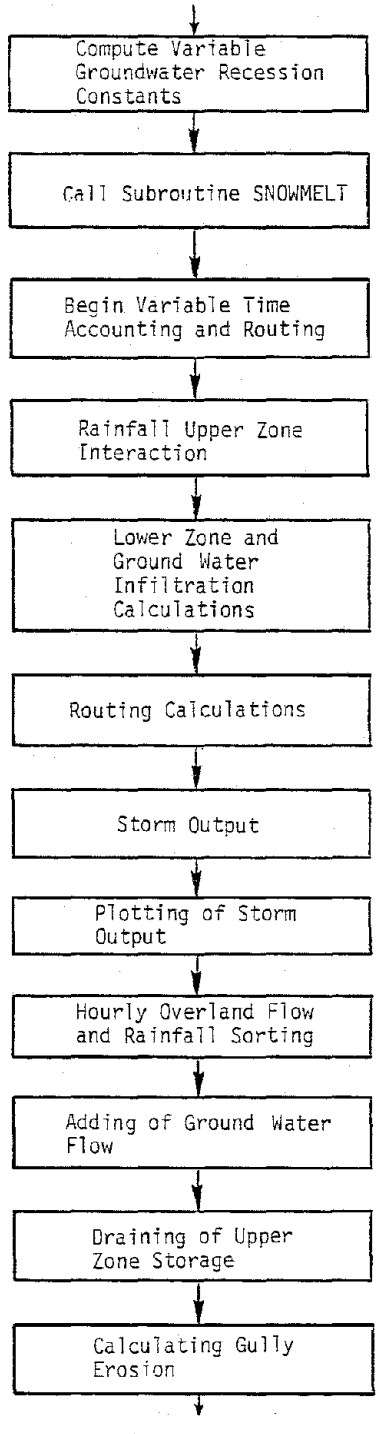


FIGURE 5: BLOCK DIAGRAM FOR TRCH20 (CONT.)

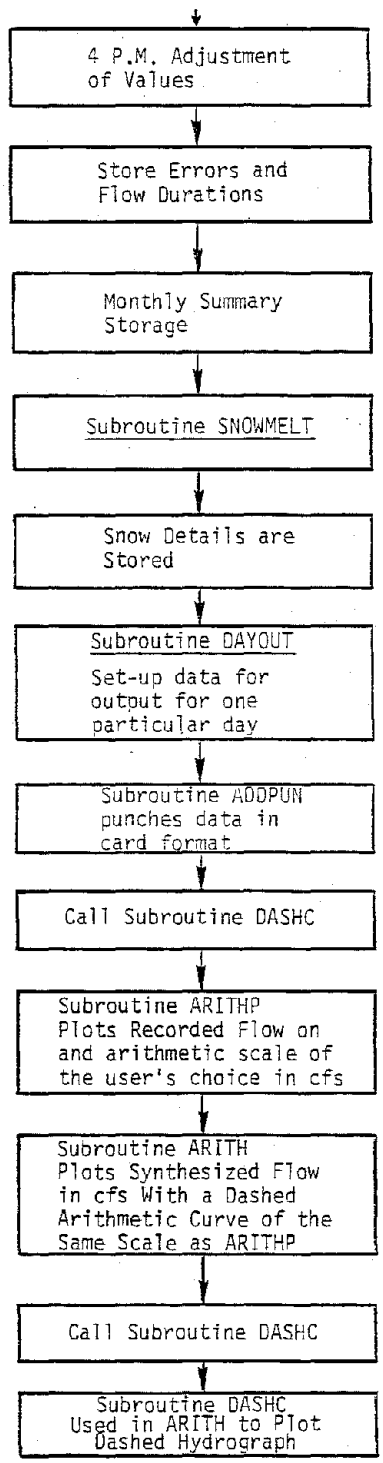


FIGURE 5: BLOCK DIAGRAM FOR TRCH20 (CONT.)

Site specific data - Card Deck

Meteorological data - NOAA magnetic tape for U.S. Weather Service Station

Plotter instructions - Card Deck

Site Specific Data --

The user of the TRCH20 model must provide coal pile data not related to meteorology. This data includes infiltration factors, flow adjustment factors and ground water information. Many of these adjustment parameters will be further defined by TRC in the user's manual developed in Phase 2 of this program. Table 34 outlines the TRCH20 site input data and the source of this data.

Meteorological Data --

TRC has designed the TRCH20 model to use a standard National Climatic Center (NCC) meteorological data tape, CD 144 format, available for U.S. Weather Services stations. In this manner, the user will not have to generate his own data tape of historical weather information. Pre-1965 data is utilized as it contains 24 observations per day.

The TRCH20 model reads values from the tape for rainfall, snowfall, and temperature for a selected number of years. The rainfall and snowfall values on the tape are given in ranges of light, medium, and heavy. The model assigns average intensity values to each. These variables are described in Table 35.

If the model user has performed a coal pile field program and has developed data on site for precipitation and temperature, this data may be used as input in lieu of the NCC tape. In addition, the user may read in actual runoff flow data and the model will plot a comparison of simulated and actual runoff flow.

Plotter Data --

The user includes data to label the axes of the hydrograph plots developed by the model for both a selected storm and an entire year of simulation. If the plotter options are not used, those cards are not included.

The plotter variables are listed in Table 36.

TABLE 34. INPUT DATA - SITE AND CALENDAR - TRCH20

1. SITE	Input Variable Name	Definition	Units	Source
	TINC	The selecting routing interval in minutes	min	From TCONC
	LZS	Current soil moisture storage	in	Initial value is estimate; checked against final simulated value
	LZSN	Soil moisture storage index	in	Developed in field program
	CY	Interflow index	---	Developed in field program
	UZS	Current soil surface moisture storage	in	Normally zero unless start of model preceded by recent precipitation
	TRANSK	Erosion transport capacity coefficient	---	Developed in field program
	DETACH	Erosion detachment capacity coefficient	---	Developed in field program
	CX	Index for estimating soil surface moisture storage	---	Developed in field program
	AREAP	Area of coal pile drainage	acres	Site map
	IRC	Daily interflow recession constant	---	Graphical techniques for hydrograph analysis
	K24L	Parameter indicating ground water flow leaving basin	---	Graphical techniques for hydrograph analysis

TABLE 34. INPUT DATA - SITE AND CALENDAR - TRCH20

1. SITE	Input Variable Name	Definition	Units	Source
	KK24	Daily base flow recession constant	---	Graphical techniques for hydrograph analysis
	KV24	Daily base flow recession adjustment factor	---	Graphical techniques for hydrograph analysis
	RMELT	Degree-day snowmelt coefficient	in/^{o}F	Developed in field program
	C	Time-area histogram (May be modified in program according to streamflow)	---	Site map and TCONC
	GULLY	Total width of gullies	ft	Measured in field
	SOILK	Coal surface constant	---	Developed in field program
	GWS	Current value of ground water slope index	in	Developed in field program
	L	Mean overland flow path length	ft	Site map
	NN	Manning's n for overland flow on pile surface	---	Developed in field program
	CB	Infiltration index	---	Developed in field program
	SGW	Ground water moisture storage	in	Developed in field program

(Continued)

TABLE 34. INPUT DATA - SITE AND CALENDAR - TRCH20

SITE	Input Variable Name	Definition	Units	Source
	TCONC	The time for water originating in the most remote region to reach the measuring station	min	Estimated from data on length and slope of pile
	Z	Number of elements in current time-area histogram	---	Site map and TCONC
	SS	Average pile slope	ft/ft	Coal pile construction
2. CALENDAR				
	YRDET	No. of years of data for the selected storm data		
	IOUT	Day of the year of current day of storm detail output being provided		
	INUM	Number of days of storm detail output		
	DKN	I.D. number of user selected options		
	QQQ	Title of computer run		
3. OTHER				
	DDYR2	Last two digits of 2nd year in water year		
	DDYR1	Last two digits of first year in water year		

(Continued)

TABLE 35. METEOROLOGICAL DATA

1) Tape Input

Variable from # NCC Tape	Definition
IR	rain - 0.1 in/hr, 0.1-0.2 in/hr, or 0.3 in/hr
IRDZL	drizzle 0.01 in/hr, 0.01-0.02 in/hr or 0.2 in/hr
IS*	snow 0.1 in/hr, 0.01-0.02 in/hr or 0.3 in/hr
ISW*	snow showers 0.1 in/hr, 0.1-0.2 in/hr or 0.3 in/hr
IP*	hail 0.1 in/hr, 0.1-0.2 in/hr or 0.3 in/hr
TEMPH	hourly temperature, ^{o}F

2) Card Input

P1	rainfall, in/hr
TSNOWH*	snow, sleet, hail, in/hr
TEMPH	hourly temperature, ^{o}F

*in water equivalents

TABLE 36. INPUT DATA - PLOTTER - TRCH20

Variable Name	Definition	Units
AXISX	Length of abscissa for plotting hydrographs	in
AXISY	Length of ordinate for plotting hydrographs	in
DDELDR	The number of cubic feet per second per inch of ordinate used in plotting the arithmetic hydrograph	cfs/in
DDX	Label of abscissa for individual storm plot	---
DDYY	Label of ordinate for individual storm plot	---
DELDR	The number of cubic feet per second per inch of ordinate used in plotting the logarithmic hydrograph	cfs/in
DELDR2	The spacing between tic marks for the ordinate of the arithmetic hydrograph	in
DELDR1	The spacing between tic marks for the ordinate of the logarithmic hydrograph	in
DL	The dash length used in plotting the synthesized hydrographs	in
DRORG	The numeric label for the minimum value of the ordinate at the axis origin for the logarithmic hydrograph	---
DRRORG	The numeric label for the minimum value of the ordinate at the axis origin for the arithmetic hydrograph	---

TABLE 36. INPUT DATA - PLOTTER - TRCH20

Variable Name	Definition	Units
QQQ	Description of gage location	---
QQY	Title of ordinate for runoff hydrograph	---
SL	The space length used in plotting the synthesized hydrographs	in
SYM	Title of absissa for runoff hydrograph	---
XAX	The length of abscissa for the individual storm plot	in
XORG	Numeric label for the minimum value of the abscissa at the axis origin for the individual storm plot	---
XTIC	The spacing between tic marks for the abscissa of the detail storm plot	in
XUNIT	The number of hours per inch of abscissa used in plotting the individual storm	hr/in
YAX	The length of ordinate for the detail storm plot	in
YORG	The numeric label for the minimum value of the ordinate at the axis origin for detail plot	---
YTIC	The spacing between tic marks for the ordinate of the storm plot	in
YUNIT	The number of cubic feet per second per inch of ordinate used in the selected storm plot	cfs/in

(Continued)

TABLE 36. INPUT DATA - PLOTTER

Variable Name	Definition	Units
ZTIC	The spacing between tic marks for the ordinate of the rainfall hyetograph plot	in
ZUNIT	The number of ordinate used in the rainfall hyetograph	in/in

(Continued)

Basic and Optional Output of TRCH20

TRCH20 provides a basic printed output of daily, monthly, and annual runoff flows, plus optional output of selected precipitation events with hourly or sub-hourly data. In addition, the program creates a tape file of hydrologic data for use in the qualitative co-model, TRCCOAL. The outputs of TRCH20 are listed below:

Basic Outputs --

1) A table of average daily rainfall in inches.

2) Summation of synthesized daily runoff rates, in cubic feet per second, for each month, followed by the annual total.

3) Synthesized monthly and annual totals for each of overland runoff, interflow, baseflow, and total flow, in inches.

4) Monthly and annual totals of liquid precipitation, in inches, developed from the meteorological input.

5) Monthly and annual totals of frozen precipitation, in water equivalent inches, developed from the meteorological input.

6) End of month values, in inches, of pile, surface, and ground water moisture.

7) End of month values for indexes of ground water slope and pile depression storage.

8) End of month values for snowpack in water equivalent inches.

9) Annual balance, in inches, of unaccounted for moisture.

Optional Outputs --

1) Echoes all input data.

2) For one selected storm per year, prints values, in inches, of rainfall deposition, moisture storage, runoff origin, and runoff outflow for each time interval.

3) Prints daily values of UZSN, UZS, GWS, SGW, SINT, SRGX, and SSGWF to give a better indication of the program interactions in the upper, lower, and deep zones of the pile.

4) Echoes recorded runoff flows, in cubic feet per second, for comparison with simulated flows.

5) Records the twenty highest clockhour rainfall events in the water year.

6) Prints daily pile moisture storage, in inches.

7) Prints daily values of the snowpack, snowmelt, and snowfall, in water equivalent inches.

8) Prints out average daily temperature, in degrees ^{o}F.

9) Prints out number of freeze-thaw cycles in preceding dry days.

10) Prints out simulated gully erosion of solids to the base of pile in pounds per day.

11) Plots an arithmetic hydrograph for water year comparing recorded and simulated flows.

12) Plots an arithmetic hydrograph and rainfall hyetograph for select detailed storm.

13) Prepares tape output file for use as input to TRCCOAL.

TECHNICAL ASPECTS OF THE TRCCOAL MODEL

During dry weather the surface of the coal pile undergoes the physical/chemical processes of pyrite oxidation; acid, iron, and sulfate production as well as the dissolving of trace materials. During wet weather these materials are washed off the surface and out of the interior of the coal pile. Seepage is generated during both wet and dry weather. These phenomena are simulated in TRCCOAL using the hydrologic balance developed in TRCH20. The block diagram for TRCCOAL is shown in Figure 6.

Dry Weather Reactions

During non-rainfall periods the coal pile is subjected to atmospheric conditions which break up the coal lumps and the moisture and oxygen in the surface of the pile cause oxidation of the pyrite in the coal. The products of pyrite oxidation are acid, sulfates, and iron. The acid further acts to dissolve trace materials.

In TRCCOAL the user inputs data on coal characteristics and reaction rates. The model simulates the total amount of pollutants in the coal and the total amount of pollutants in a dissolved state during a dry weather period. The number of freeze/thaw cycles calculated in TRCH20 is used to accelerate the break up of coal and the subsequent pyrite oxida-

1119-001

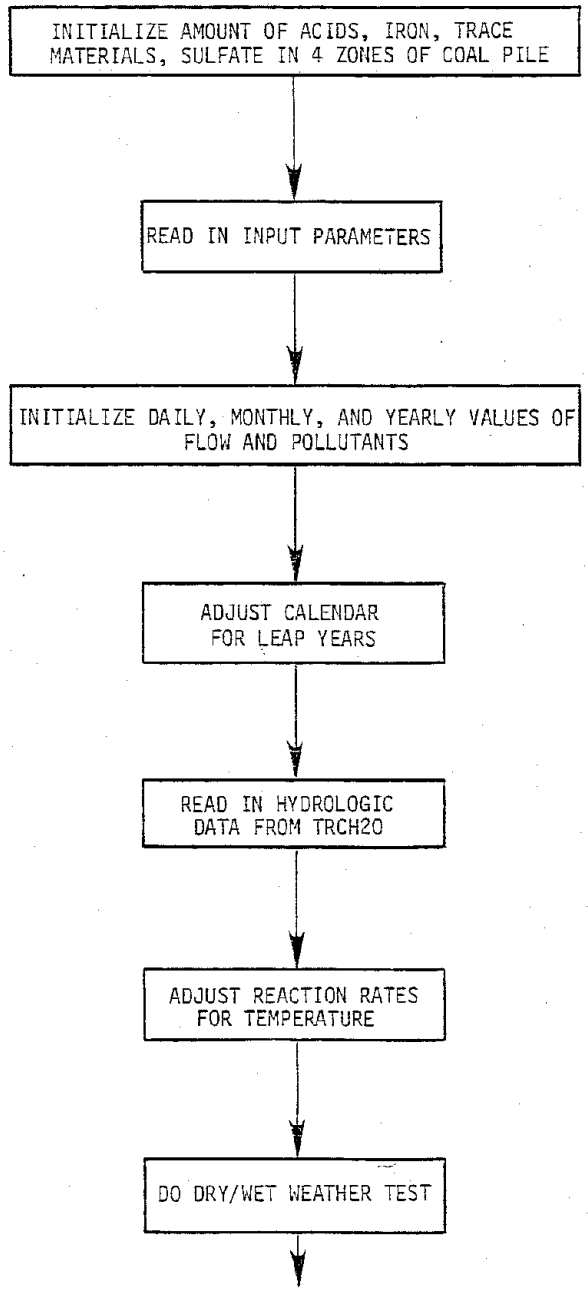


FIGURE 6: BLOCK DIAGRAM FOR TRCCOAL

1119-002

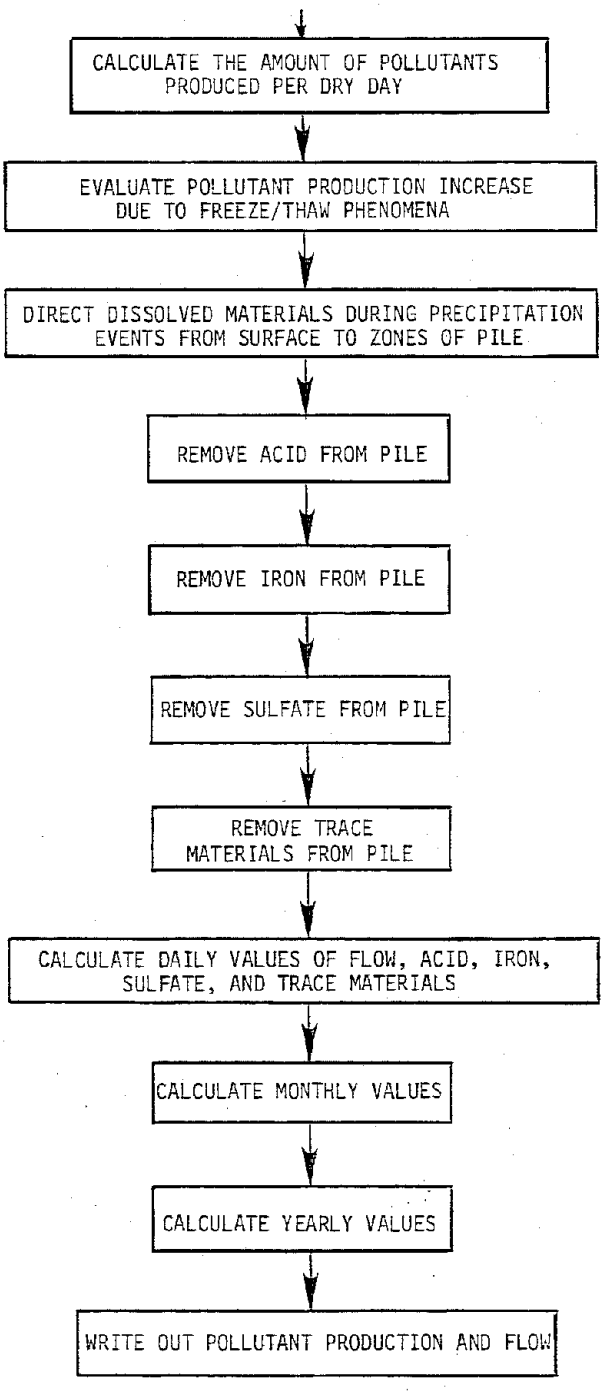


FIGURE 6: BLOCK DIAGRAM FOR TRCCOAL (CONT.)

tion. The dissolved material is then available for washoff during the next storm event. Also during dry weather moisture is emitted from the lower zone as seepage.

The formulations in the model for these reactions are shown below:

AMTPY = lbs of framboidal pyrite in surface of coal pile
AMTPY = PERPY * DENCOL * DEPTH * AREA * 43560. (4-23)

PERPY = % framboidal pyrite
DENCOL = Coal density, lb/ft^3

DEPTH = depth of coal surface layer, ft
AREA = area of coal pile, acres

AMTACI = lbs of acidity produced/dry day interval
AMTACI = AMTPY * RATEPY * TIME 1/24. * ALKFAC (4-24)

RATEPY = pyrite oxidation rate, day^{-1}
TIME1 = dry day time interval
ALKFAC = factor for alkalinity of coal

AMTTM(K) = lbs of trace material, K, in coal pile surface
AMTTM(K) = PERTM(K) * DENCOL * DEPTH * AREA * 43560. (4-25)

PERTM(K) = % of trace material, K, in coal

TMDIS(K) = lbs of trace material, K, dissolved per dry day interval
TMDIS(K) = AMTTM(K) * AMTACD * FACTM (K) (4-26)

FACTM(K) = trace material, K, dissolving factor per lb. acid

SULDIS = lbs of sulfate dissolved per dry day interval
SULDIS = 19.2 * AMTACD (4-27)

TFEDIS = lbs. of T. iron dissolved per dry day interval
TFEDIS = 42. * AMTACD (4-28)

TAMTAC = lbs. of acid produced for sequential dry day
TAMTAC = TAMTAC * FRTHAW * FT (4-29)

FRTHAW = no. of freeze-thaw cycles in preceding dry days.
FT = freeze-thaw factor

The model conducts a wet/dry test to determine if the wet weather mode of washoff or the dry weather mode of acid production, and seepage will be utilized for a given day. If the total rainfall is less than 0.1 inches, then the model considers it a dry day and only acid production and seepage generation takes place. The cutoff value of 0.1 inches is arbitrarily chosen.

Wet Weather Reactions

During wet weather, rainfall entering the pile distributes a portion of the dissolved metals, sulfates, and the acid on the surface to the interior of the pile and to direct runoff. In addition, the acidity of the rain is added to the available acid in the pile. The amount of pollutants that is distributed to the upper zone (depression storage), lower zone (pile interior), direct runoff, and interflow is proportional to the solubility of the pollutants and an adjustment factor. The material stored in the lower zone is further distributed to deep storage and the baseflow. Following are example equations for acid in direct runoff. The equations for other pollutants in the other zones are similar.

ACRAIN = lbs of acid in acid rain/wet weather interval

$$ACRAIN = RAINCO * PR * AREA * 5.2\ E\text{-}6 \quad (4\text{-}30)$$

RAINCO = acidity of rain, mg/l
PR = rain (in.)/wet weather time interval

ACDDIR = lbs of acid going to direct runoff

$$ACDDIR = FACDIR * SOLACD * OVFLST * CF \quad (4\text{-}31)$$

FACDIR = factor for the amount of acid going into direct runoff
SOLACD = solubility of acid, lb/ft^3
OVFLST = inches of current rainfall entering direct runoff
CF = conversion to cubic feet of runoff

EXADIR = Excess acid in direct runoff storage

$$EXADIR = ACDDIR + EXADIR \quad (4\text{-}32)$$

The amount of acid, metals, and sulfate in storage zones that is washed out of the pile can be linearly or exponentially related to the available material. The user determines the best fit to the field data and chooses either option. The option, EXPO-1, simulates an exponential decay rate. This relationship is best demonstrated by the "first flush" effect and considers the removal of both dissolved and suspended material. Therefore, either the exponential washoff function in TRCCOAL

is used to simulate suspended material or simulate the erosion routine of TRCH20, but not both. The equations for acid are shown below as an example:

WASHA = exponential factor for acid

$$WASHA = 1. - EXP\ (-AK*TIME2) \quad (4\text{-}33)$$

AK = exponential coefficient for acid
TIME2 = wet weather time interval

AREDIR = lbs of acid being removed by direct runoff

$$AREDIR = DIRRNF * SOLACD * FACRED * CF \quad (4\text{-}34)$$

DIRRNF - Current rate, in inches, of direct runoff entering runoff channel
FACRED - factor for amount of pollutant going into runoff channel

Option EXPO --

$$AREDIR = (EXADIR + EXAUZ) * WASHA * CF \quad (4\text{-}35)$$

TRCCOAL then calculates daily, monthly, and annual totals of the acid, metals, and sulfates contained in all components of the runoff stream.

Input Data for TRCCOAL

The TRCCOAL model has two sources of input data:

1) The magnetic tape file of hydrologic data
2) Card input describing qualitative aspects of the coal pile.

The tape file created by TRCH20 contains data on rainfall, amount of rainfall directed to the pile zones, the flowrate of direct runoff, interflow, and baseflow, the total daily rainfall, the average daily air temperature, and the number of freeze-thaw cycles for each simulated time period.

The card input consists of three types: calendar data, adjustment coefficients, and site specific input.

1) The calendar input data is shown in Table 37. The user selects the time period of the simulation as well as the select storm for output. These must match the calendar data in TRCH20.

2) Table 38 lists the adjustment coefficients for the coal pile model. These parameters allow the simulated qualitative output for the distribution of pollutants within the coal pile as well as the runoff flow, to be "fine-tuned" to match actual values as found in field data. Recommended values for these parameters will be developed by TRC in the second phase of this program.

3) Values for the coal pile under consideration which can be measured are listed in Table 39. These include the solubility of acid, iron, sulfate, and trace metals, the density and area of the coal pile, and the acidity of the rain, for example.

TABLE 37. TRCCOAL - INPUT DATA CALENDAR PARAMETERS

Variable	Definition
YEARLP	Last 2 digits of next leap year divided by four
YEARST	Last 2 digits of starting year of model
YEAR1	Year of detailed output request
MONTH1	Month of detailed output request
DAY1	First day of detailed output request
NDAY	Number of consecutive days of output requested

TABLE 38. TRCCOAL - INPUT DATA

ADJUSTMENT PARAMETERS*

Variable	Definition
FACDEP	Factor for depth of outer mantle
FACDIR	Factor for amount of pollutants going into direct runoff
FACINT	Factor for the amount of pollutants going into interflow storage
FACLZ	Factor for the amount of pollutants going into lower zone
FACUZ	Factor for the amount of pollutants going into upper zone
FACRED	Factor for pollutants entering runoff stream by direct runoff
FACREI	Factor for the amount of pollutants going into the runoff stream by interflow
FACREB	Factor for the amount of pollutants going into the runoff stream by baseflow
FACRDS	Factor for the amount of pollutants going into deep storage
FACFD	Factor for the amount of runoff coming from direct runoff
FACFI	Factor for the amount of runoff coming from interflow
FACFB	Factor for amount of water coming from baseflow
TK	Coefficient for exponential washoff of trace metals
AK	Coefficient for exponential washoff of acid
FK	Coefficient for exponential washoff of iron
SK	Coefficient for exponential washoff of sulfate

* No units

TABLE 38. TRCCOAL - INPUT DATA ADJUSTMENT PARAMETERS*

Variable	Definition
FT	Freeze-thaw factor for increased production of dissolved materials
FACTM	Trace material dissolving factor per lb. of acid
RATEPY	Pyrite oxidation rate per day
ALKFAC	Factor for alkalinity of coal

* No units

(Continued)

TABLE 39. TRCCOAL - INPUT DATA SITE SPECIFIC PARAMETERS

Variable Name	Definition	Units
SOLACD	Solubility of acid	mg/l
DEPTH	Depth of outer mantle	ft
AREAP	Area of coal pile	acres
TIME2	Time interval from TRCH20	hour
PERPY	Percentage of framboidal pyrite in coal	%
DENCOL	Density of coal in pile	lb/ft^3
N	No. of trace materials in coal simulated in model (max=8)	---
TM	Name of trace materials in coal (max=8)	---
PERTM	Percentage each trace material in coal	%
PERSUL	Percentage sulfur in coal	%
PERTFE	Percentage T. iron in coal	%
RAINCO	Acidity of rain	mg/l
SOLTFE	Solubility of total iron	mg/l
SOLTM	Solubility of each trace material	mg/l
SOLSVL	Solubility of sulfate	mg/l
EXPO	Option for linear or exponential washoff	---

Output of TRCCOAL

TRCCOAL provides output concerning the pollutant loadings from the coal pile. The output products are listed below:

1) A calendar showing which days of the year in the simulation were considered wet or dry.

2) A calendar of simulated daily runoff flow volume, in cubic feet per second.

3) Daily loadings of total acid as well as in direct runoff, interflow, and seepage, in pounds.

4) Daily loadings of sulfate, iron, and trace materials in direct runoff, interflow, and seepage, in pounds.

5) A calendar of total daily rainfall, in inches.

6) A calendar of direct runoff, interflow, and seepage volume, in cubic feet per second.

7) Optional plots showing the relationship between rainfall, runoff flows, and pollutant loadings on a detailed storm basis.

SENSITIVITY ANALYSIS

TRCH20 - Quantitative Model

The input data to the coal pile drainage model will require varying degrees of accuracy. The model will be very sensitive to some parameters and their value will greatly impact the runoff stream yield and shape of flow recession curve. Other parameters are used for fine-tuning purposes only.

A sensitivity analysis was conducted on several selected parameters. Those chosen deal principally with the moisture balance of the water and are difficult to measure in the field. The values chosen were CB, LZSN, KK24, IRC, and GWS. The results of varying the parameters are summarized in Table 40.

CB - Infiltration Index --

CB is the infiltration index that controls the rate of infiltration. It functions in the model's formula for peak infiltration rate:

$$D4F = \frac{FRAC*CB}{2.0**LNRATM} \qquad \text{(see equation 4-1)}$$

TABLE 40
SUMMARY OF RESULTS OF THE SENSITIVITY STUDY - TRCH20

Key: ↑ Increased, ↓ Decreased, — Not Affected; * Slightly, ** Moderately, *** Significantly

Selected Input				Simulation Feature		
Parameter	Parameter Change	Yield	Peaks	Interflow Recession	Interflow Recession	Pile Moisture
CB	↑	↓***	↓***	↑**	↑*	↑***
LZSN	↑	↓***	↓***	↓ *	↓*	↑***
KK24	↑	—	—	—	—	—
IRC	↑	—	—	↓ *	—	—
GWS	↑	—	—	—	—	—
TINC	↑	—	—	—	—	—
TCONC						
Z						
C						

Increasing CB significantly increased infiltration and subsequently lowered runoff flow yields and peaks. As expected, pile moisture increased with increasing infiltration and the recession flows more gradually approached zero.

The impact of three values of CB on runoff flows is shown in Figure 7.

LZSN - Pile Moisture Storage Capacity --

LZSN is the pile moisture storage capacity index. It represents the volume of water which may be held in the interior of the pile.

Increases in LZSN increase the recharge capacity in the coal pile. Subsequently runoff volume and peaks decrease.

With the increase in LZSN, interflow and baseflow recession flows decrease slightly. Figure 8 shows the runoff hydrograph for three values of LZSN.

KK24 - Baseflow Recession Constant --

KK24 is the baseflow recession constant. Its effect on runoff flow yields, peaks and pile moisture is insignificant, as shown in Figure 9. Little impact can be detected in interflow and baseflow recession flows also.

IRC - Daily Interflow Recession Constant --

IRC is the daily interflow constant. Varying IRC had no impact on peaks and runoff flow yields. In addition there was only a slight variation in interflow recession flows. Like KK24, there was no significant variation in pile moisture.

Because of the nature of the exponential equation that utilizes IRC, its values must be less than and equal to 1.

Figure 10 shows two values of IRC.

GWS - Current Value of Ground Water Slope Index --

GWS is the value of the ground water slope index and represents the antecedent moisture conditions at the interface of the pile and the underlying material.

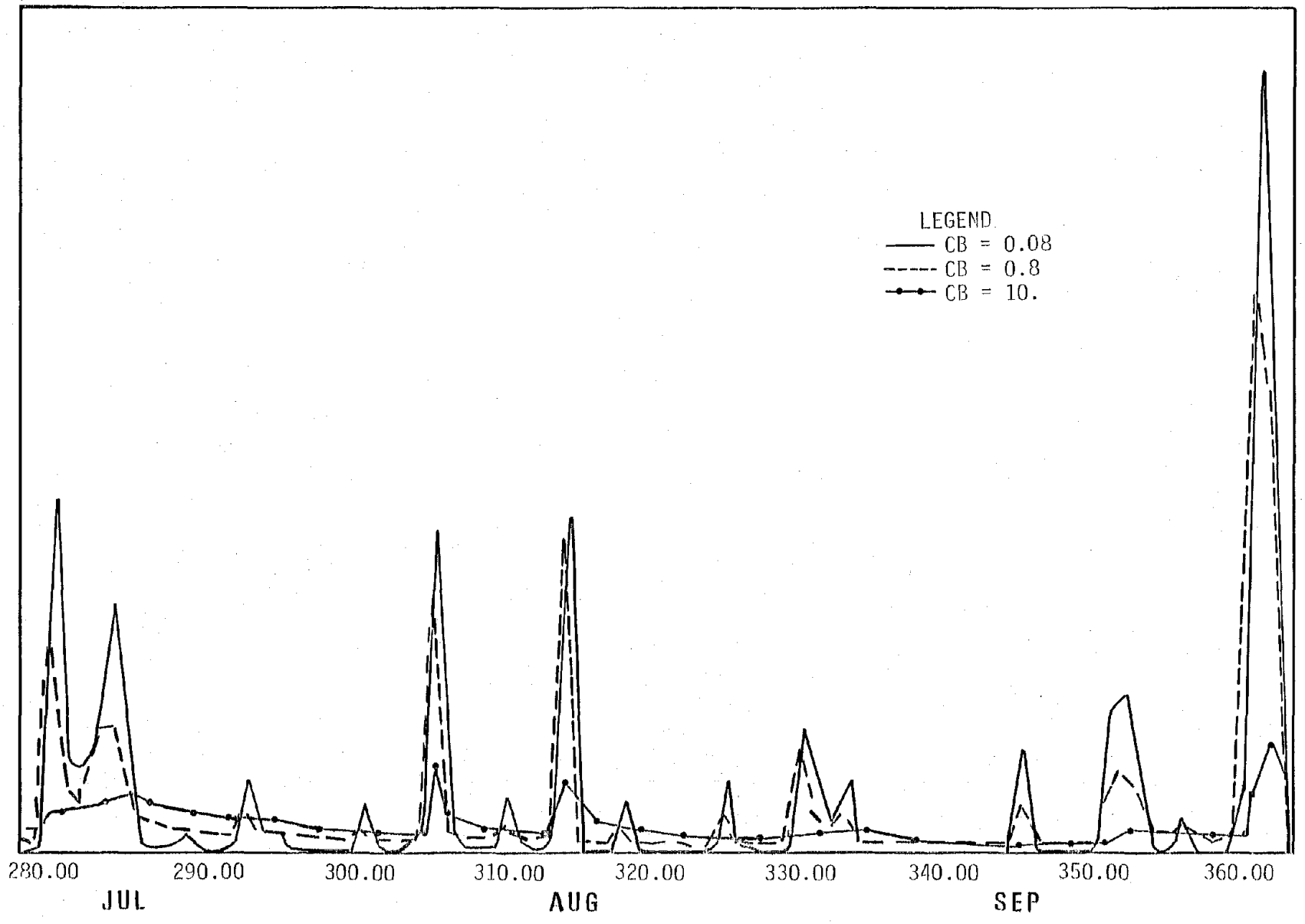


FIGURE 7: EXAMPLE SENSITIVITY ANALYSIS, CB-INFILTRATION INDEX

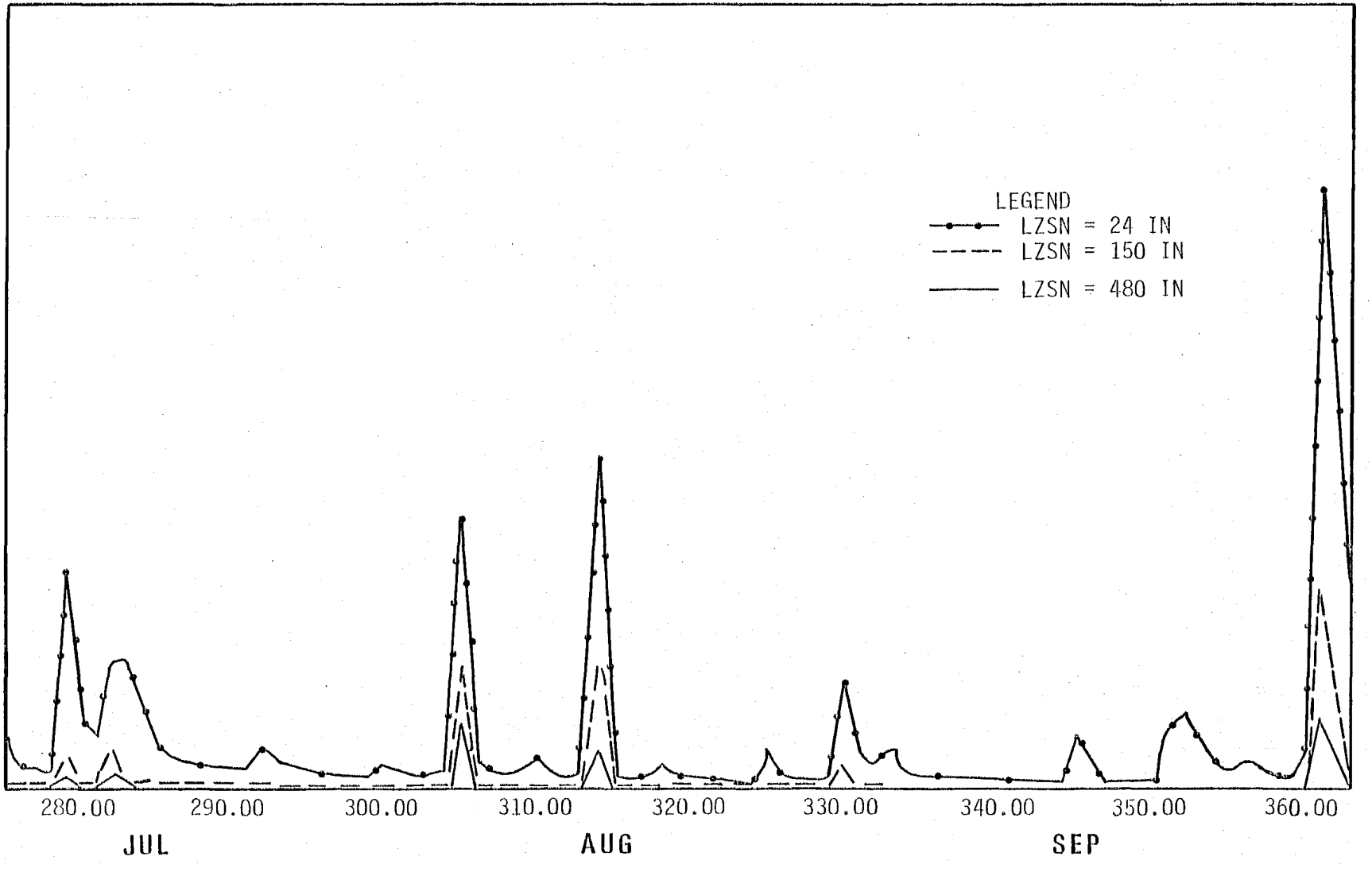


FIGURE 8: EXAMPLE SENSITIVITY ANALYSIS, LZSN-PILE MOISTURE STORAGE

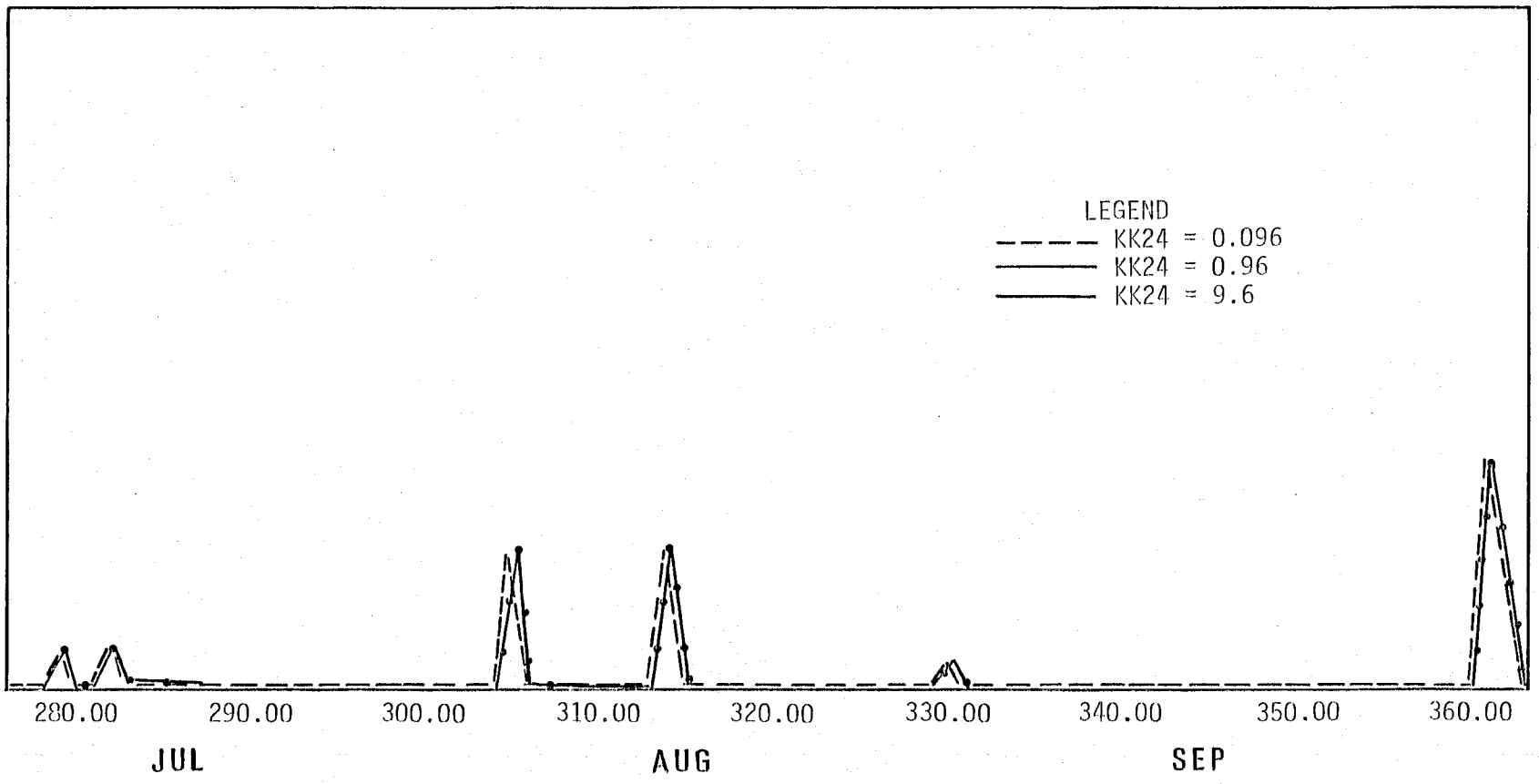


FIGURE 9: EXAMPLE SENSITIVITY ANALYSIS, KK24 - BASEFLOW RECESSION

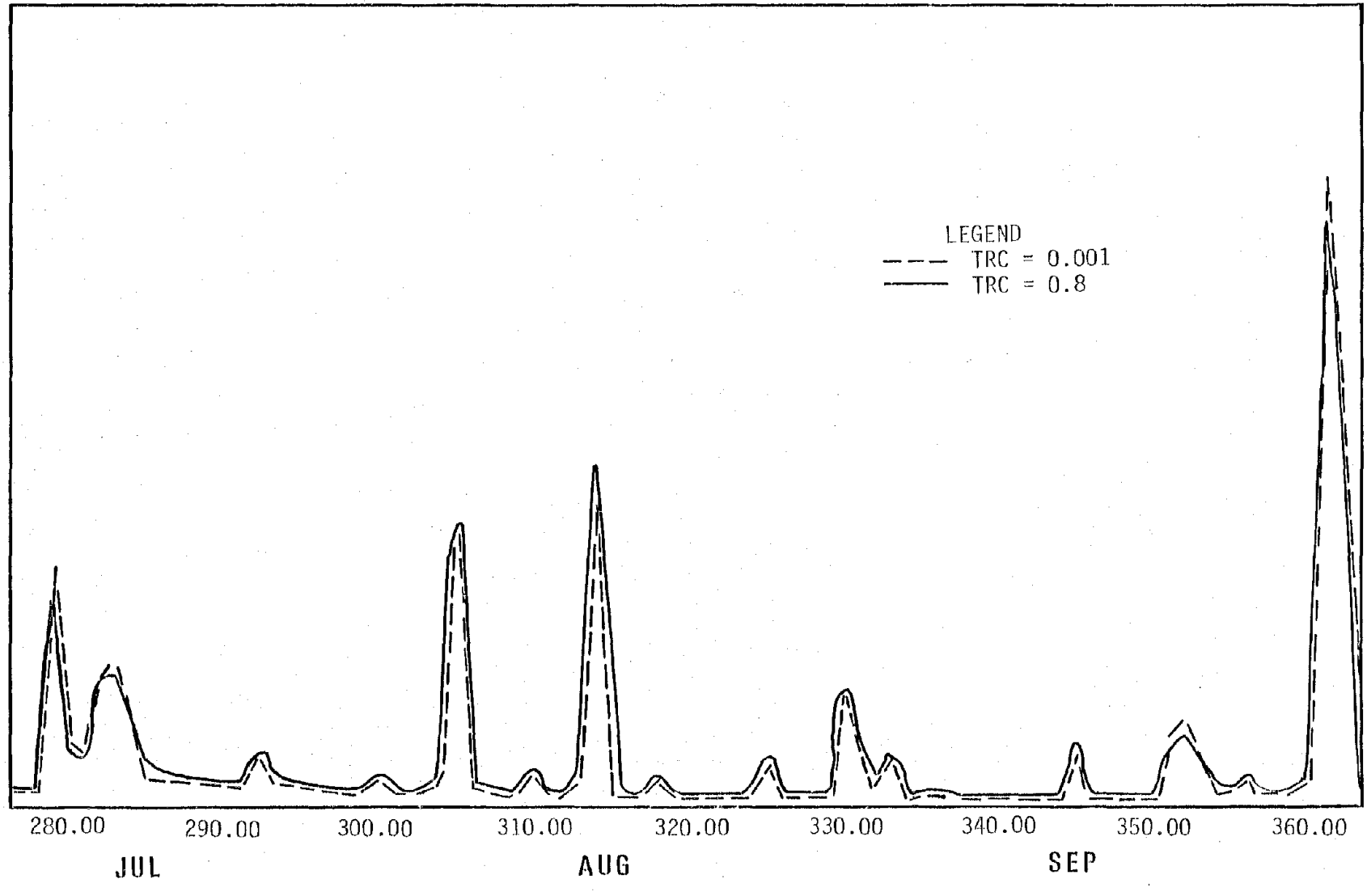


FIGURE 10: SENSITIVITY ANALYSIS, TRC - DAILY INTERFLOW RECESSION CONSTANT

GWS is used in the equation:

GWF = S6W*LKK4*(1.0+LKV4*GWS) (see equation 4-19)

As expected, it had little impact on runoff flow volume and peaks. However, it also had no impact on ground water flow. It should be noted that GWS did not reach steady state in these sensitivity tests and for this example of an 11 acre coal pile, an initial value of greater than 21 was warranted.

Figure 11 shows the impact of variations in GWS.

TINC - Selected Time Routing Interval --

In the OSU model decreasing the time interval of simulation shifted the peak flow earlier in time and increased synthesized peak flows. However, because of the small watersheds involved in coal piles and the short time of travel, these changes were not seen in decreasing the routing interval time, from 30 minutes to 5 minutes.

As seen in Figure 12, the routing interval had no impact on runoff flow and peaks.

TRCCOAL - Qualitative Model

Five parameters associated with the production and washout of acid from the coal pile were chosen for the sensitivity analysis of TRCCOAL. These are DEPTH, FACDIR, RATEPY, PERPY, and AK. All sensitivity analyses were plotted for a two month period and the graph lines are offset slightly to show the three parameters evaluated. The model output utilized was the acid loading in the coal pile drainage stream.

DEPTH - Depth of Reactive Surface of Coal Pile --

DEPTH is the parameter which reflects the depth of the reactive surface of the coal pile, in feet, in which pyrite oxidation take place. It is used in the equation to calculate the pounds of framboidal pyrite in coal.

AMTPY = PERPY * DENCOL * DEPTH * AREA * 43560 (see equation 4-23)

It is also used in similar calculations to calculate the amount of sulfur, iron, and trace metals in the surface.

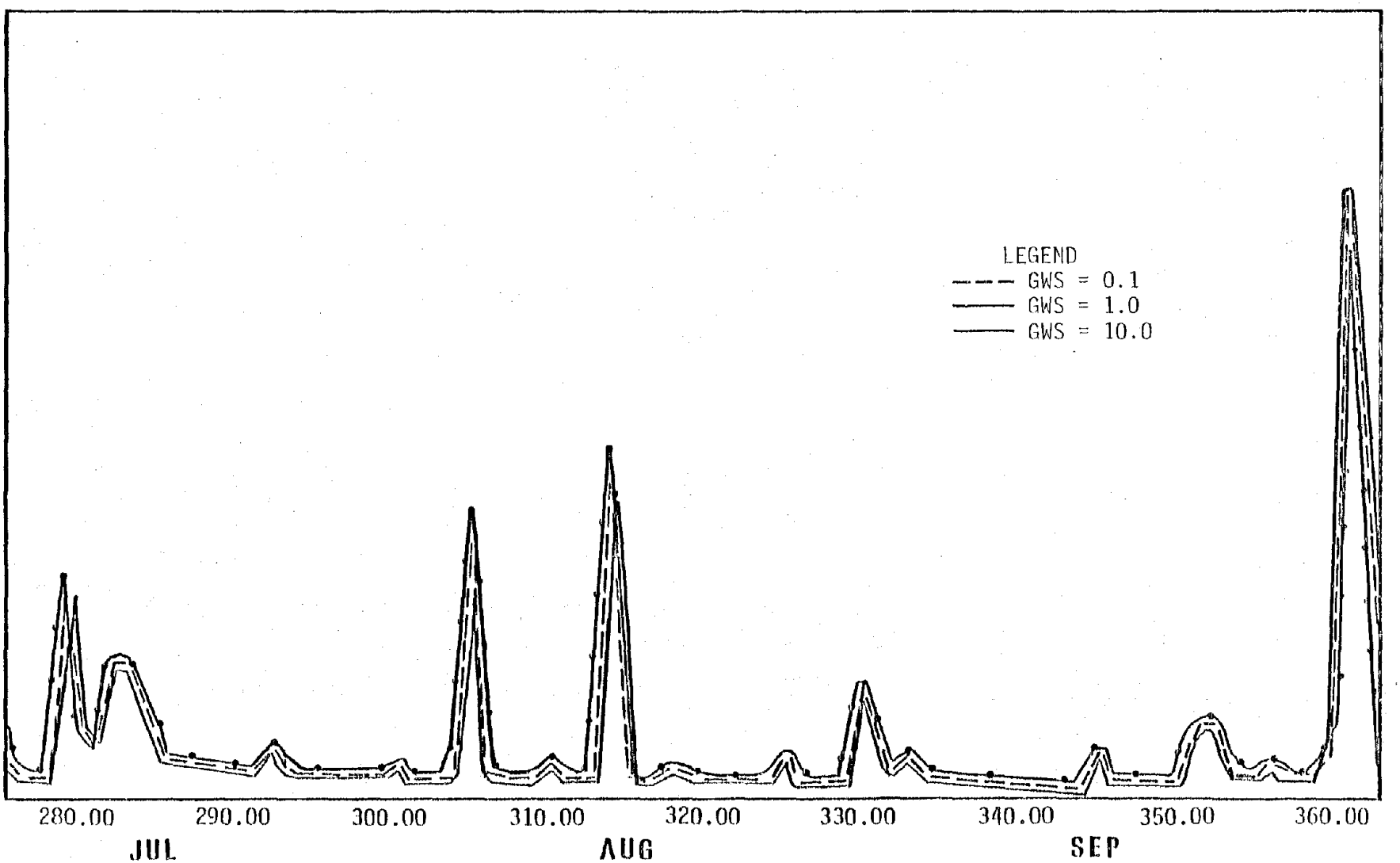


FIGURE 11: SENSITIVITY ANALYSIS GWS - GROUND WATER SLOPE

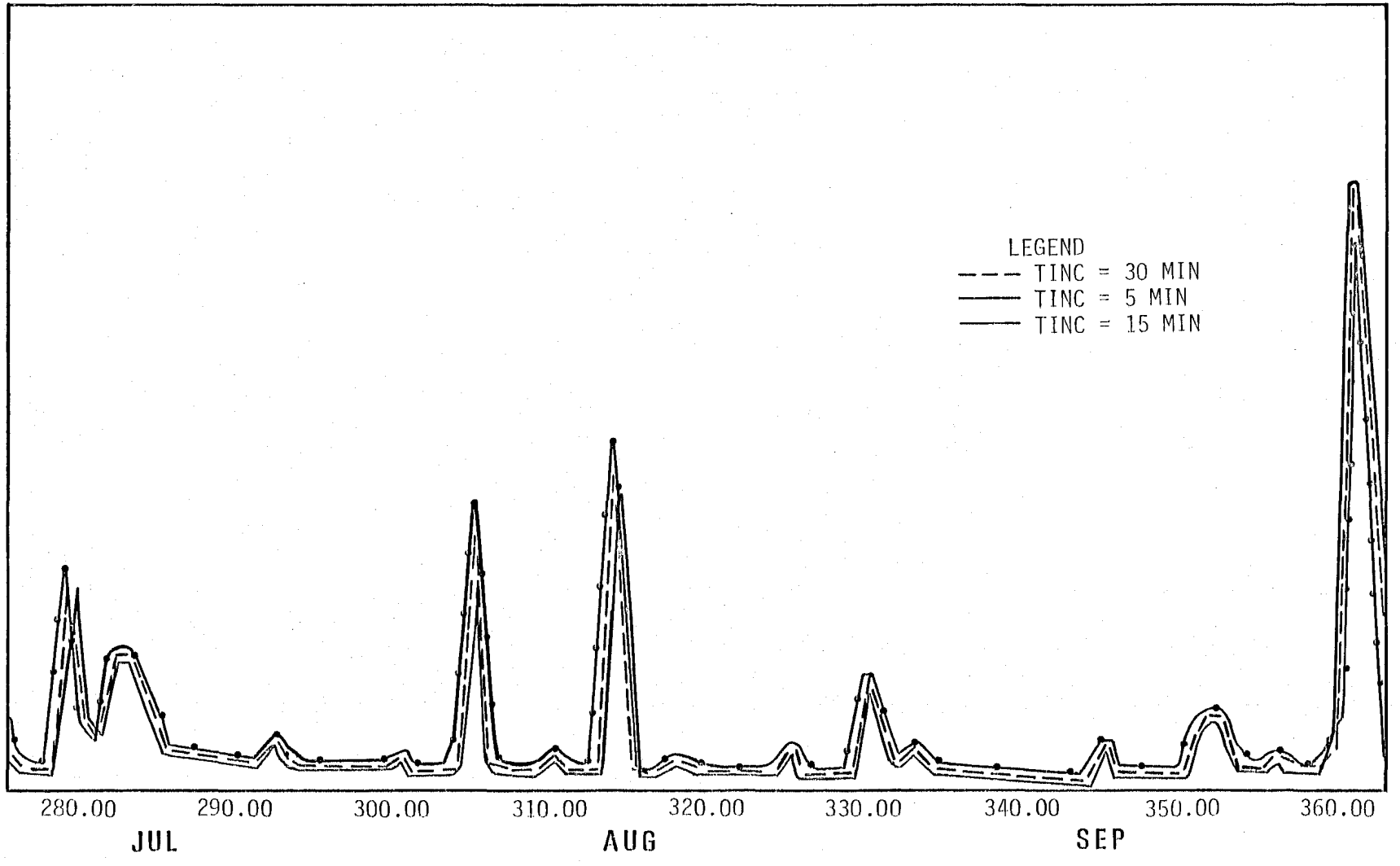


FIGURE 12: SENSITIVITY ANALYSIS SIMULATION TIME INTERVALS

The depth to which oxygen, which controls the reaction, penetrates the pile is difficult to estimate. Values of 0.01', 0.1', and 0.25' were evaluated. The loading of acid in the coal pile drainage stream was determined to be very insensitive to the depth values in this range.

The sensitivity analysis for DEPTH is shown in Figure 13.

RATEPY - Rate of Pyrite Oxidation --

While the model showed no sensitivity to the depth of the reactive surface, there was slight sensitivity to the rate of pyrite oxidation, RATEPY. RATEPY is used in the equation:

AMTACI = AMTPY * RATEPY * TIME 1/24. * AIKFAC (see equation 4-24)

Three values of the pyrite oxidation rate were evaluated in the sensitivity analysis, 0.01, 1., 100, as shown in Figure 14. While increasing the pyrite oxidation rate from 0.01 to 1. is reflected in an increase in the peak acid loading in the outflow stream, an increase from 1. to 100. does not produce a similar increase. While increased pyrite oxidation increases acid loadings in the runoff stream at low levels, after a certain point the washoff parameters are the limiting factors, despite an increase of acid in the coal pile.

FACDIR - Factor for the Amount of Pollutants Going Into Direct Runoff --

FACDIR is the adjustment factor for the amount of pollutants going from the surface to the direct runoff zone of the pile. It is used in the equation for acid, for example:

ACDDIR = acid in direct runoff zone
ACCDIR = FACDIR * SOLACD * OVFLST * CF (see equation 4-31)

There are similar adjustment values for the amount of pollutants directed to the lower zone, upper zone, and interflow zone of the coal pile as well as adjustment factors for the washout of pollutants from these zones to the outflow channel.

Three values were selected for FACDIR - 0.00001, 0.0001, 0.0002. As shown in Figure 15, increasing FACDIR caused a moderate increase in the acid loading in the runoff stream. It is one of the more sensitive parameters in the qualitative model.

1119-024

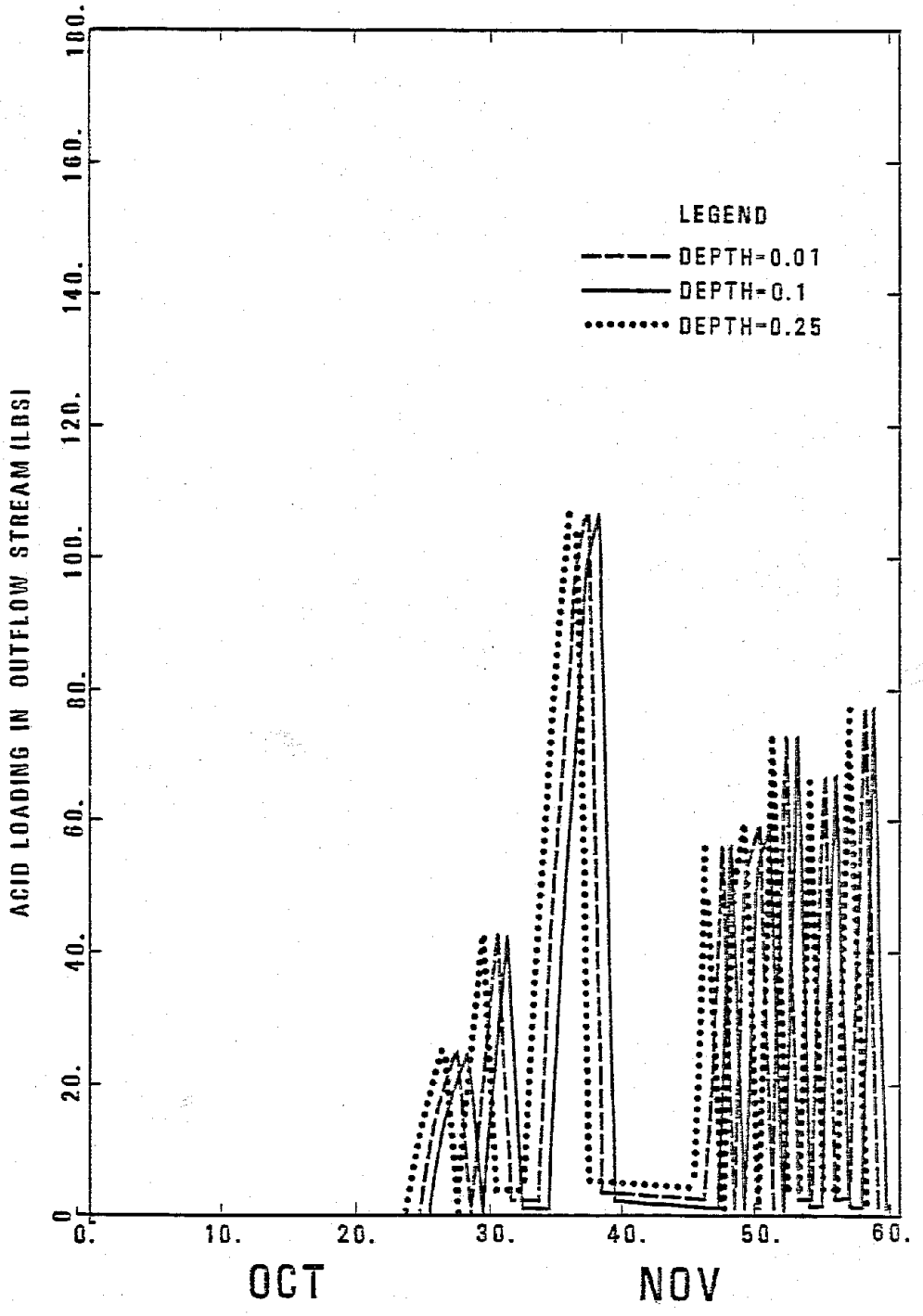


FIGURE 13: EXAMPLE SENSITIVITY ANALYSIS DEPTH

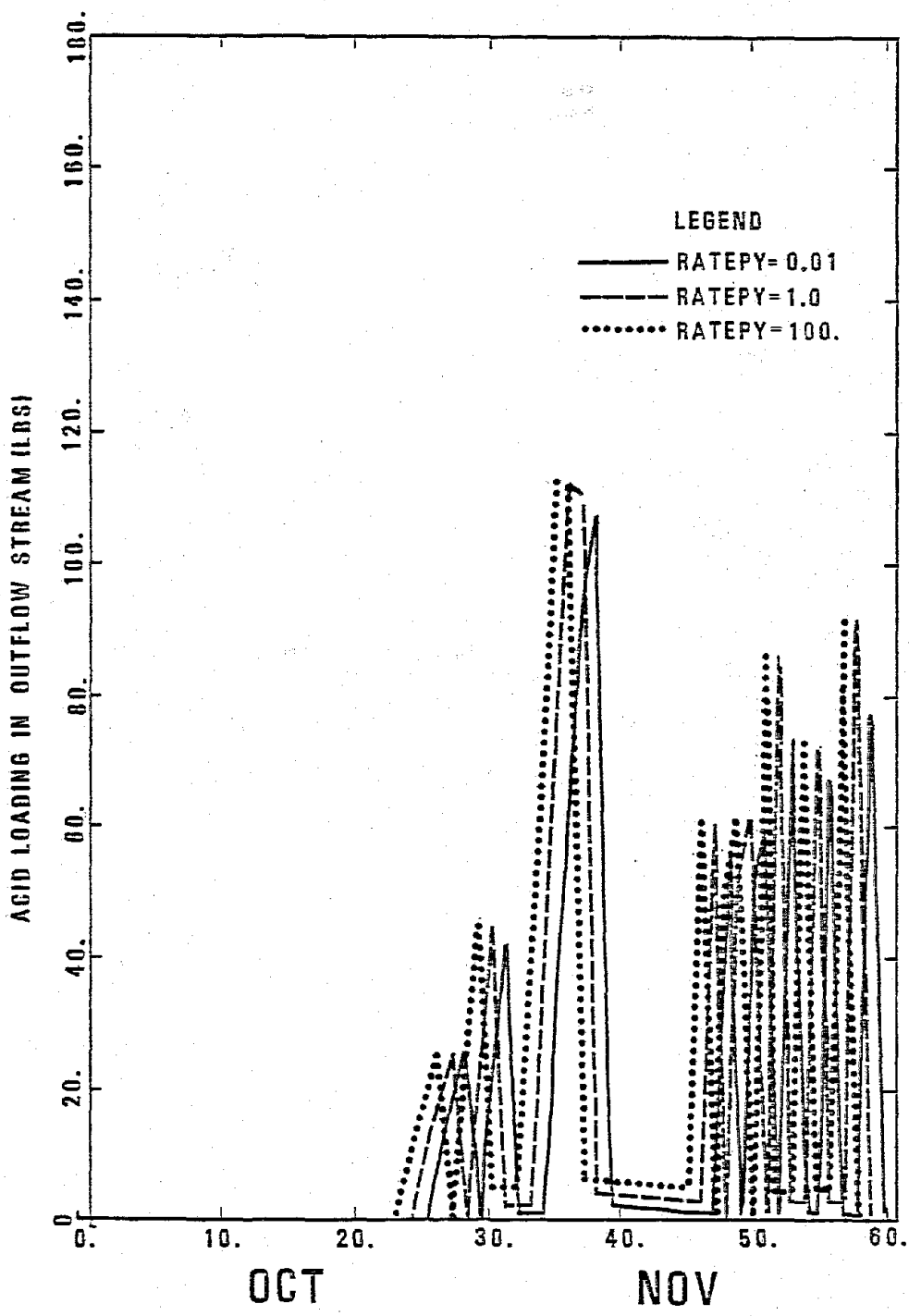


FIGURE 14: EXAMPLE SENSITIVITY ANALYSIS RATEPY

1119-027

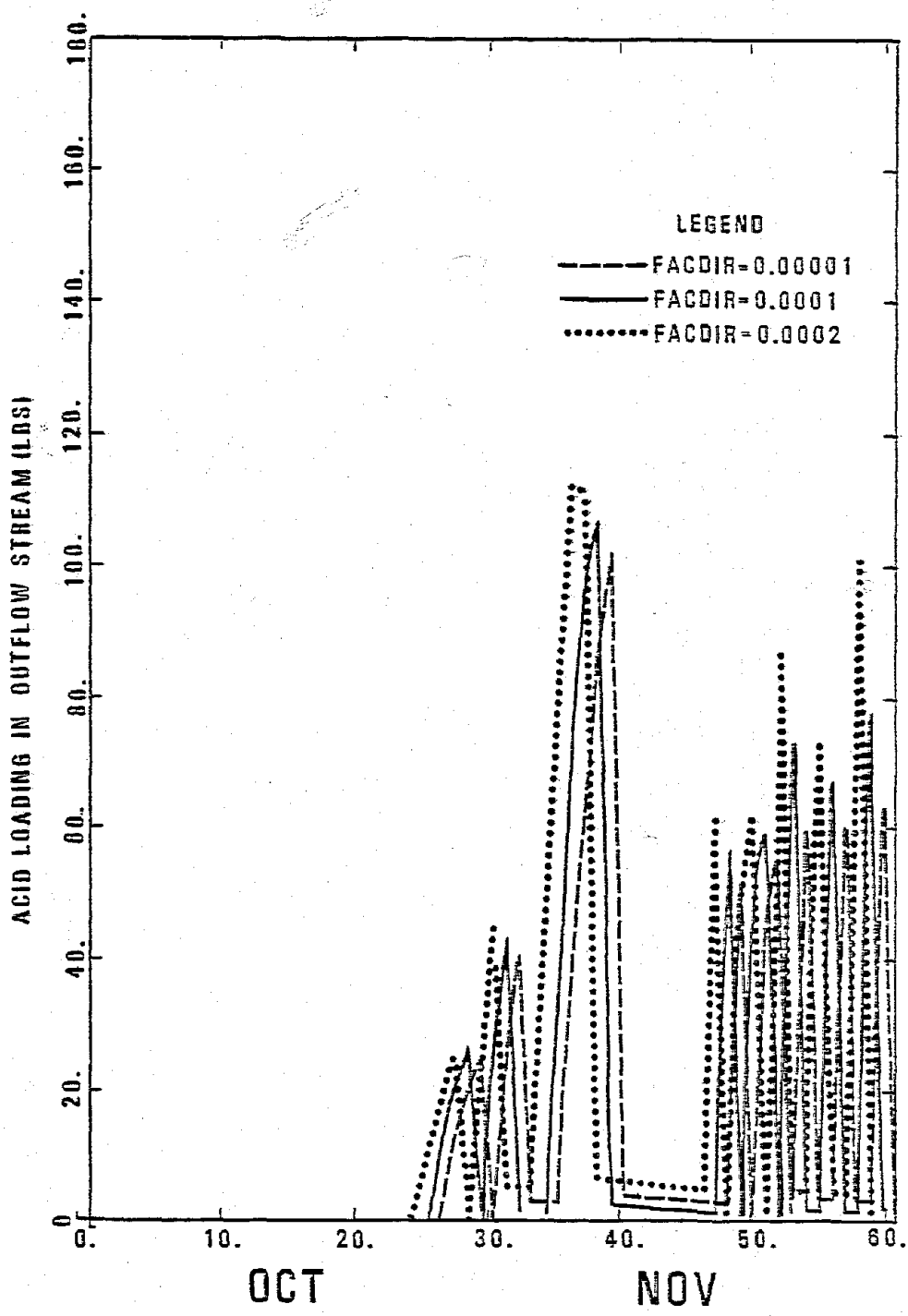


FIGURE 15: EXAMPLE SENSITIVITY ANALYSIS FACDIR

PERPY - Percentage of Framboidal Pyrite in Coal --

The amount of acid production is related to the degree of framboidal pyrite in coal, PERPY. However, a coal pile will represent several coal seams and sources, therefore the sensitivity of the model to an estimate of the percentage of framboidal pyrite is important.

PERPY is used in the equation:

AMTPY = PERPY * DENCOL * DEPTH * AREA * 43560 (see equation 4-23)

Three values were chosen for PERPY - 1%, 3%, and 5%. Like the parameter DEPTH, the amount of pollutants washed out of the coal pile is not sensitive to PERPY. This is shown in Figure 16.

AK - Exponential Washoff Factor for Acid --

The model has the option of considering the linear washoff of the pollutants from the coal pile or a "first flush" or exponential washoff. The variable AK is the exponential coefficient for acid in the latter algorithm.

WASHA = 1. - EXP (-AK * TIME 2) (see equations 4-33
AREDIR = (EXADIR + EXAUZ) * WASHA * CF & 4-34)

Three values were chosen for AK - 0.06, 0.6, and 6. As shown in Figure 17, the acid loading removed from the pile is somewhat sensitive to AK, especially with the lower range of values 0.06 - 0.6.

SHORTCOMINGS AND LIMITATIONS OF CPD MODEL

Mathematical models are, by definition, simplifications of complex physical and chemical reactions. Within each model are assumptions which make the model a workable tool. By understanding the shortcomings and limitations of the coal pile drainage model, the user can better use the results of the model in engineering applications and can better design the input data for the model.

1119-028

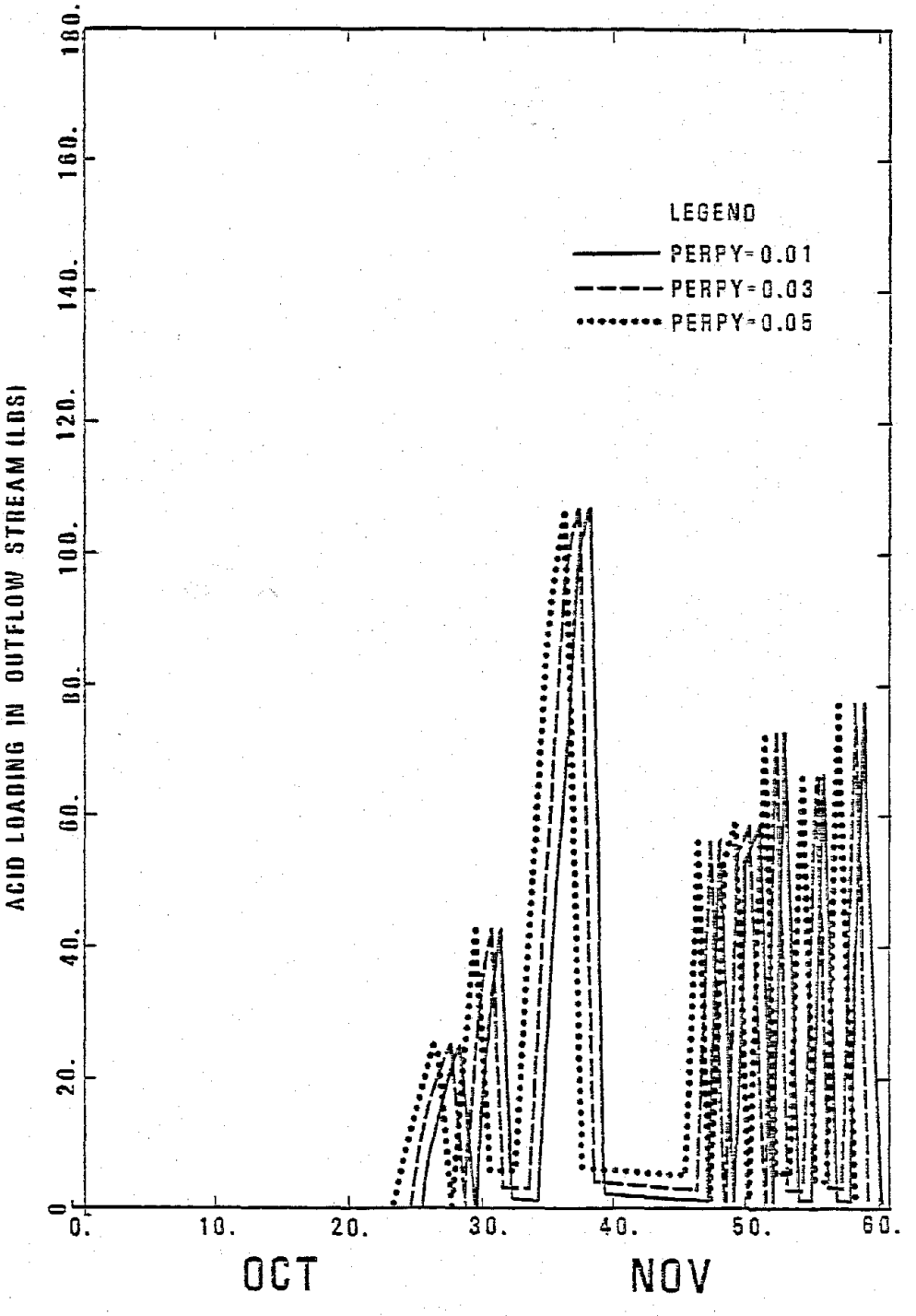


FIGURE 16: EXAMPLE SENSITIVITY ANALYSIS PERPY

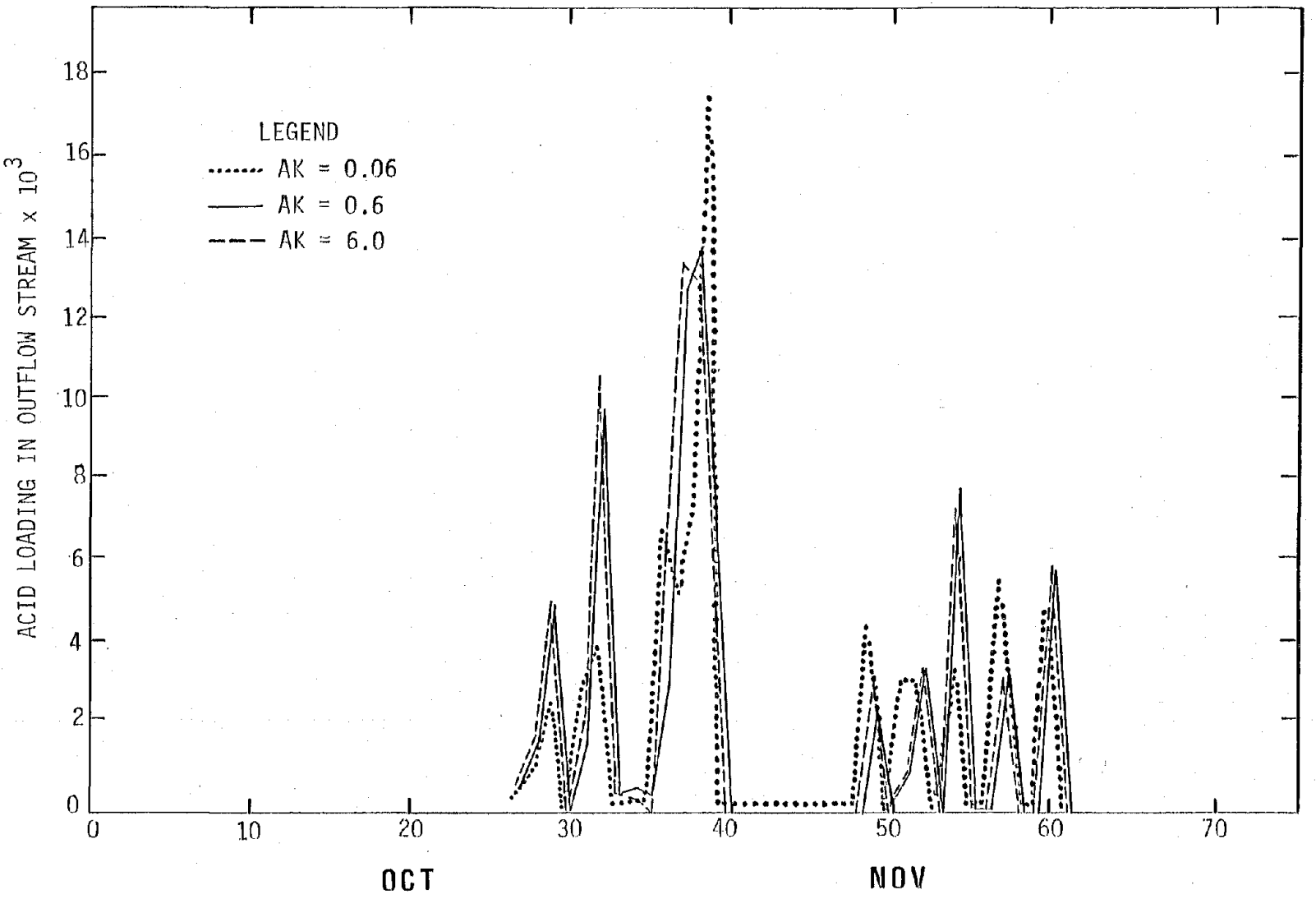


FIGURE 17: EXAMPLE SENSITIVITY ANALYSIS AK

TRCH20

Coal Pile Hydrology --

The hydrologic model, TRCH20, concerns the flow from one runoff drainage point from the coal pile under consideration. It assumes that the runoff stream is channeled and can be measured. It also assumes one runoff stream per coal pile.

Therefore the model should be used with contained coal piles only. When there is more than one runoff outlet per coal pile, the coal pile should be considered as several piles. The input variable for the area of the coal pile will then reflect only the fraction of the pile in the runoff drainage area for that model run.

Snowmelt --

Snowmelt is addressed by TRCH20, but in a more simplified form than the previous Ohio State Model. In reality snowmelt is the result of solar radiation, ground temperature, and snow density. TRCH20 considers only air temperature to generate snowmelt.

The snowmelt is expressed in water equivalent inches of snow. Water equivalent inches are 5%-15% of the total snowfall. The snowmeltsnowpack values generated by sample runs produced values which correlated reasonably well with recorded data.

Infiltration --

In coal piles there are working faces as well as inactive sections of the pile where no coal has been recently removed. In reality these areas probably have different surface characteristics, with the working face much more pervious than the inactive portions of the coal pile. However, in the model, there is only one average surface infiltration rate for the coal pile and one interior percolation rate, with no differentiation between active and inactive faces. However, if the coal pile drains to several different runoff points, then the program run which includes the working face of the coal pile, can have modified infiltration parameters.

Evaporation --

Most models which consider evaporation in the hydrologic balance are, in reality, evaluating evapotranspiration by vegetation on the watershed or lake evaporation off water surfaces. Little work has been done concerning pan evaporation from unvegetated surfaces, including coal piles. Since the coal pile is pervious and unvegetated, evapotrans-

piration should have little impact on the moisture balance. One exception is the depression storage on top of the coal pile. In these areas, puddles could occur which would be subject to evaporation effects.

TRC has not found a totally satisfactory simulation of daily evaporation which can be used for coal piles and therefore has not included evaporation in the CPD model at this time. However, a system of algorithms relating evapotranspiration from short vegetation to average daily air temperature has been developed. During the field program pan evaporation will be measured and if it appears that evaporation from the depression storage is very significant, the algorithms on evapotranspiration will be included in the model to attempt to simulate this phenomena.

Precipitation --

The NCC magnetic tape which is used as meteorological input to the program expresses the precipitation each hour as light, medium, and heavy and an average value is assigned to each. These model values underestimate actual precipitation when there is heavy rainfall greater than 0.4"/hr and overestimate trace rainfall.

In the average year these approximations cancel out. For example, in the test water year 1963-1964 the total simulated rain and snow was 43.9 water equivalent inches and the actual was 42.0 inches.

However, for detailed storms more exact data may be needed. Therefore the model has the option to replace the magnetic tape input with card input of exact hourly precipitation data.

TRCCOAL

The qualitative model, TRCCOAL, also makes certain simplifying assumptions to simulate coal pile drainage.

For example, the model views each day as a wet day or a dry day. On wet days, previously dissolved materials are washed out of the coal pile. On dry days, pyrite oxidation and dissolved materials production takes place. In reality, only a part of each day will the coal pile be wet enough to preclude pyrite oxidation. Therefore the model has this simplification inherent to its structure. The model could be modified to do the wet/dry test on an hourly basis, but this would make the cost of computing prohibitive.

The TRCCOAL model also makes assumptions that certain values are average values for the year and do not fluctuate by day or hour. These are listed below:

1) the percentage of framboidal pyrite in the coal pile
2) the alkalinity factor of the coal
3) the pyrite oxidation rate (modified by air temperature)
4) the percentage of trace metals, iron, and sulfur in coal pile
5) the production rate of dissolved iron and sulfate
6) the acidity of rainfall

USES OF THE CPD MODEL IN TREATMENT DESIGN

The ultimate purpose of simulating coal pile drainage is for use in the design of systems to treat the acid drainage before discharge.

To design treatment facilities, data is needed on both the quantity of runoff that can be expected and its average and worst case qualitative characteristics.

On the initial level, the model uses meteorological data tapes to simulate "average" conditions. From a run of several years worth of data, the user can simulate a range of values for the volume of runoff expected and the pollutant loadings.

However, an examination of worst case conditions must also be included in design engineering for collection basins and treatment works. Often treatment facilities are designed to contain the 10 year - 24 hour storm without any actual knowledge if this storm frequency is appropriate. Only the first hours of a heavy storm may contain a pollutant loading requiring treatment and the runoff flow from the remainder of the storm can be safely bypassed without treatment. In addition, the actual worst case may be several sequential storms during a short period with a continuous stream of pollutants washing out of the coal pile.

The model is structured to allow the user to input simulated meteorological data instead of actual historical values. The user then creates different combinations of storm events and evaluates the simulated runoff output.

The plotter output of the model for both detailed storms and an entire year presents the most salient data in an easy to use form. Plots of hydrologic and pollutant data for the same time period can be superimposed for comparison purposes. From these plots, the volume and duration of pollutant loadings can be determined.

COMPUTING REQUIREMENTS

TRC developed the combined coal pile drainage models (TRCH20, TRCCOAL) utilizing time-sharing capabilities. TRC performed time-sharing

on the Apex system of CDC computers through United Computing Service centered in Kansas City and serving 150 metropolitan areas.

The costs below for running the model are for one year of output. Of course, initial runs will be performed on small time spans and costs will be significantly less.

Estimated Cost (spring 1980) for running the model for one year are given:

1)	TRCH20 with no options	$200
2)	TRCH20 with all options	$300
3)	TRCCOAL with no plotter output	$ 80
4)	TRCCOAL with plotter output	$ 90

Most utilities will be able to access the models utilizing a small portable keyboard terminal and telephone hook-up through a local telephone exchange or by submitting input data to TRC. Meteorological information can be obtained from the National Climatic Center's tape library of data from U.S. Weather Service Stations. The tapes will then be forwarded to the UCS data center in Kansas City where they are mounted for model use.

The card input deck is very small and can be typed in at the terminal or read in by card reader by the user at a local UCS office.

Printout and plotter output are mailed directly to the user.

SECTION 5

FIELD WORK PLAN

INTRODUCTION

The primary objectives of a phased field program for coal pile runoff from utility sites are as follows:

1) To obtain representative data to fine tune and modify the coal pile runoff model.

2) To test the runoff model for a variety of climatic regions, coal pile configurations, and coal characteristics.

3) To generate a substantial data base on the quantity and quality of coal pile runoff from various utility sites throughout the country.

The end results of these objectives will be:

1) A field proven mathematical model for predicting the quantity and quality of coal pile runoff. This model will be a useful tool for the design of runoff treatment systems. The primary emphasis of the model will be in predicting the volume of storm-water runoff from a coal pile so that accurate estimates of runoff coefficients can be compared against the coefficients used in the Rational Formula. This information can then be used as design data.

2) Simpler tools, such as nomograms, charts, and/or tables to assist those without computer resources in designing treatment for coal pile runoff.

In addition, the phased field measurement program has the following secondary objectives, all of which will be useful in the design of runoff treatment systems. These include:

1) Determination as to whether the quality of coal pile runoff exhibits either a "first flush" effect or a "flow dependent" effect. The "first flush" effect is a condition whereby the

maximum pollutant loading occurs at the start of a storm event; i.e., the pollutants are flushed from the pile at the start of a storm event. The "flow dependent" effect is a condition in which the quantity and quality of coal pile runoff varies directly with storm duration and intensity, i.e., peak pollutant concentrations and runoff flow occur during peak rainfall intensity. This will be valuable information in the design of runoff treatment systems.

2) Establishment of a water balance for coal piles. The hydrologic balance would consider runoff flow, base flow, and interflow from the coal piles, as well as water retained by the piles and water which infiltrates into ground water beneath the piles.

3) Performance of correlation analyses for various pollutants in runoff from coal piles. Correlation analyses will be used to preclude the need for extensive analyses of various pollutants and also to establish important trends in runoff characteristics. Past research has shown that the degree of framboidal pyrite oxidation can be correlated with the acidity of the runoff. In addition, temperature and oxygen availability may be correlated with the degree of pyrite oxidation. Other research has shown that conductivity may be correlated with total dissolved solids (TDS) and sulfate. In turn, TDS may be correlated with sulfate. TDS and total suspended solids (TSS) may also be correlated with the dissolved and suspended fractions of metals.

4) Initial screening of coal samples for metals potentially available for washoff. This will determine which metals should be analyzed during the field program. These metals will then be analyzed for both dissolved and suspended fractions to determine speciation according to the acidity/alkalinity of the runoff.

The overall field program will be designed to attain these primary and secondary objectives. The field program will be conducted at 12 different utility sites throughout the country over a three year period.

The field program will begin with two "test" sites to gather information to be used to fine tune and modify the coal pile runoff model. In addition, the field testing procedures will be refined and modified as necessary at these two sites. The "test" sites will also be used to satisfy the secondary objectives of the program. As such, these sites will serve to refine the field program for the remaining ten sites. The sampling program at each test site will be approximately of 10 weeks duration.

The remaining ten sites will be used to test the runoff model for a variety of climatic regimes, coal pile configurations, and coal characteristics, as well as to generate a substantial data base on coal pile runoff. These sampling programs will vary in duration from one month to nine months.

All data collected in the field will be put in a form which is compatible for use in the runoff model. In addition, this data will be digitized and made easily accessible through magnetic tape. Thus, the user, who may wish to design a treatment system for coal pile runoff for a coal pile similar in characteristics to one of the studied sites, has only to acquire the magnetic tape and review the data.

This data will incorporate the following:

1) Site description data, including:

   a) General plant information
   b) Coal pile characteristics
   c) Coal characteristics

2) Meteorological conditions (historical and concurrent with field program);

3) Daily runoff flows and pollutant loadings;

4) Single storm hyetographs, hydrographs, and pollutographs; and

5) Statistical summaries of storm events indicating worst case conditions encountered during the field program.

The field work plan to be discussed will serve as a guide to planning the overall field program. The work plan addresses the following components necessary in the design of a field program of this magnitude:

1) Selection of 12 utility sites.

2) Initial site visit including background data acquisition.

3) Preliminary work, including acquisition of coal samples, and coal pile and ground water testing.

4) Laboratory testing of the coal samples.

5) Choice of pollutant parameters for analysis.

6) Determination of sampling frequency.

7) Selection of sampling, flow monitoring and meteorological equipment.

8) Development of a plan for shipping samples for analysis.

9) Laboratory analysis of the runoff samples.

In addition, data reduction, analysis, and presentation procedures, manpower requirements, and the overall program schedule and budget are considered.

SELECTION OF REPRESENTATIVE PLANT SITES

The identification of plant sites to be used in the study of coal pile runoff is the first step in preparing a detailed work plan for a monitoring program. The study of selection criteria describing representative coal pile characteristics and site conditions resulted in a listing of parameters that can be divided between macro-parameters and micro-parameters. Macro-parameters include generating capacity of the plant, percent sulfur content of the coals, general region where the coal is mined and annual total precipitation for the power plant locations. Micro-parameters include actual coal pile sizes that are representative of a certain group of plants, seasonal precipitation patterns, amount of snow accumulation, and plant operating conditions which may influence the characteristics of the runoff such as lined piles, coal cleaning, etc. For the purposes of the field work plan, only the macro-parameters are used in the selection. Once the individual monitoring sites are identified, they will be distinguished according to the micro-parameters during the preliminary site setup task.

The field work plan is based on monitoring the coal pile runoff from 12 different coal fired utility plants. Two of the 12 sites are identified as "test" sites where the runoff will be monitored from a low sulfur medium size pile and a high sulfur medium size pile for a 10 week period. Medium size plants will be selected for scale purposes so that it will be easy to adjust follow-on monitoring at small plants and large plants. High and low sulfur containing coals were selected for test sites because there is agreement in the literature that sulfur, expecially pyritic sulfur content of the coals, is the major determinant in the quality of the runoff water.

Table 41 presents the site selection matrix to be used to identify the category in which a prospective site may fall. Referring to the last row of plant categories, only one category is marked for plants burning coal mined in the southwestern region because there are very few plants in this category. An additional medium size plant burning Appalachian coal is included because this represents a very large category of older plants and will include many plants reconverting from other fuels back to coal. An additional large size plant burning Western and Great Northern Plains coal is included because this represents those future plants using coal from states with the largest reserves of coal, i.e., Wyoming and Montana. Once the prospective monitoring sites have been identified, only those plants with different micro-parameters will be selected for the medium size Appalachian and large size Western coal burning plants.

TABLE 41. SITE SELECTION MATRIX FOR THE MONITORING OF COAL PILE RUNOFF

Small Plant W/GNP Coal	Medium Plant W/GNP Coal*	Large Plant W/GNP Coal
Small Plant IE/IW Coal	Medium Plant IE/IW Coal	Large Plant IE/IW Coal
Small Plant AP Coal	Medium Plant AP Coal*	Large Plant AP Coal
Medium Plant AP Coal	Medium Plant SW Coal	Large Plant W/GNP Coal

Plant Size: Small 25-100 MW
Medium 100-800 MW
Large 800 MW

Coal Source Region:	Mean % Sulfur by Weight
W/GNP coal - Western & Great Northern Plains	0.83
IE/IW coal - Interior Eastern & Inter. Western	2.86
AP coal - Appalachian	1.83
SW coal - Southwestern	0.57

* = Test Sites to be identified with medium size plants, one using low sulfur coal and one using high sulfur coal.

INITIAL SITE VISIT

The field survey at each of the 12 utility sites will be specific to each site. This will necessitate a visit to each site as the first step in the design of the field program. The site visit should accomplish the following tasks:

1) Selection of suitable locations for runoff and base flow sampling.

2) Selection of suitable locations for installation of the meteorological station and the field laboratory trailer.

3) Acquisition of necessary background information as input to the runoff model.

Each of these tasks must be addressed in the development of test plans for each site. The on-site visit and discussions with plant personnel should be sufficient to accomplish these tasks.

Selection of Sampling Sites

A visual survey will be used to select suitable locations for storm-water runoff and base flow sampling. Coal piles typically have several runoff and base flow streams which, due to the local topography, do not flow in the same direction. This will necessitate treating each stream as an individual sampling site. For instance, a particular coal pile may have as many as 2-5 streams which must be considered individually. Attempts will be made to schedule the initial on-site visit so that at least one storm event with post-storm baseflow conditions is observed to determine the number of streams to be sampled and the best location for sampling each stream.

Some of the sites may already have flow measurement devices, such as weirs and flumes, for monitoring coal pile runoff. These devices will be incorporated into the test plan for the site.

Selection of Locations for Installation of Meteorological Station and Field Laboratory Trailer

Continuous meteorological data will be collected throughout the field program. This data will serve as necessary inputs to the coal pile runoff model and also as a data base on meteorological conditions at each site. The meteorological information to be collected includes:

a) Precipitation
b) Air temperature/relative humidity
c) Solar radiation
d) Evaporation

The data will be obtained with automated monitoring equipment. All measurements will be recorded on continuous strip charts. The equipment will be housed on site in a louvered shelter. A suitable location will be selected for the installation of the meteorological station during the initial on-site visit. This location should be in an open, flat area free from obstructions but in close proximity to a power source.

In addition, a suitable location for setting up the field laboratory trailer will be selected. This trailer will be used for performing on-site laboratory analyses, for containing the continuous monitoring equipment and preserving samples for other analyses. The trailer will

also contain a drying oven for solids and moisture content analyses. A central location will be chosen close to the coal piles with easy access but away from the flow of plant traffic.

Acquisition of Background Data

Another objective of the on-site visit will be the acquisition of necessary background data which is recorded at the site. These data will satisfy several of the data requirements at the individual sites. Figure 18 shows the major categories of data requirements. These data will serve as inputs to the runoff model and also to generate a data base for each site. The data to be collected during the on-site visit includes:

1) Site description data

   a) General plant information
   b) Coal data
   c) Coal pile data
   d) Ground water data

2) Historical site meteorological data (if available)

The remaining data requirements for each site, including meteorological and runoff data, will be satisfied during the actual field testing program.

The majority of the site description data will be gathered from plant operating records, plant correspondence, coal delivery records, and from discussions with plant personnel. These data are listed below by major category.

The general plant information to be gathered will include the following elements:

a) Plant name or station
b) Utility name
c) Location (city, county, state)
d) Type of plant (base load or peaking)
e) Number of coal-fired units
f) Generating capacity

The following information will be collected for each type of coal burned at the site:

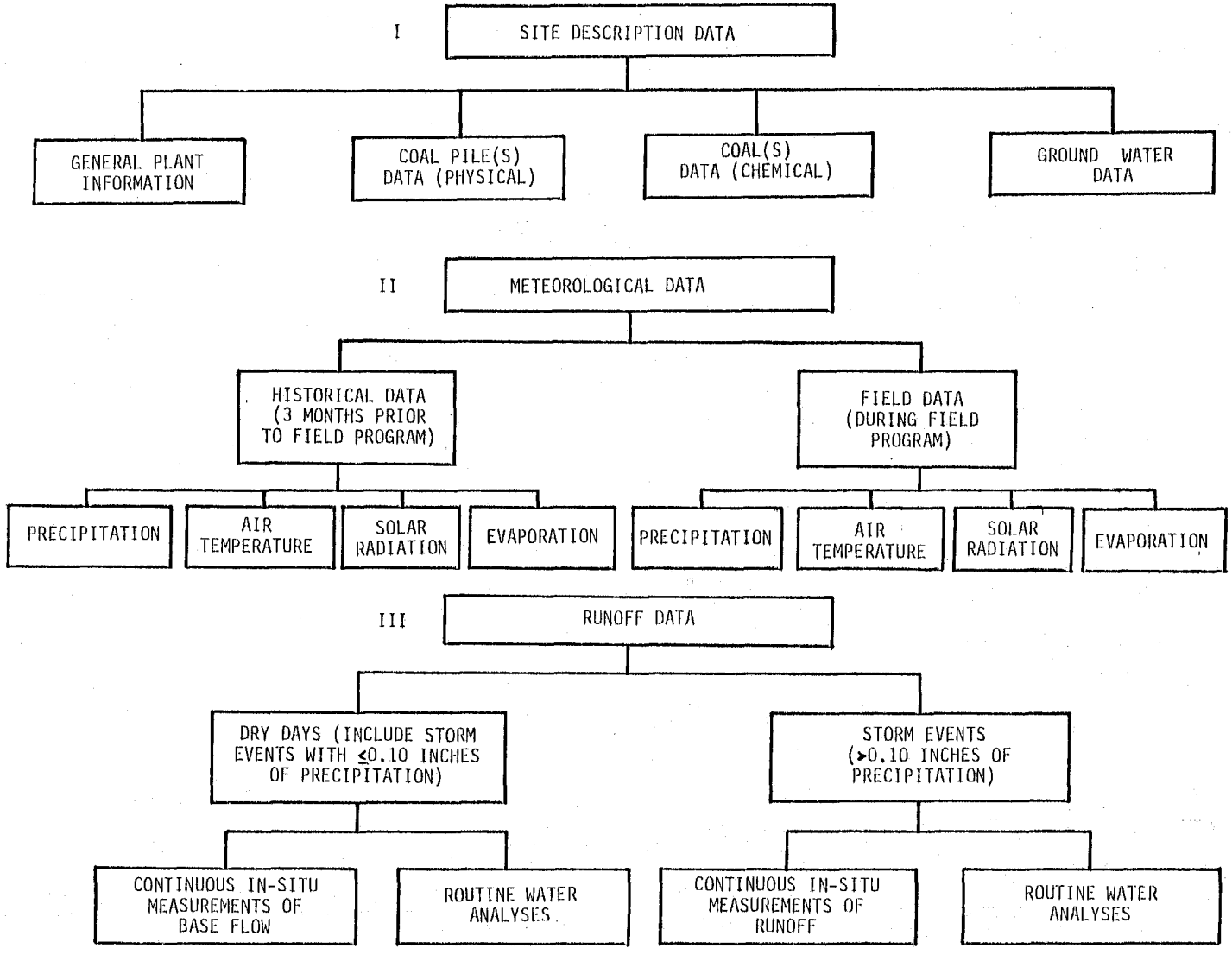


FIGURE 18: COAL PILE RUNOFF DATA REQUIREMENTS AT INDIVIDUAL SITES

a) Coal rank
b) Coal source:
   1) County, state
   2) Seam
c) Method of coal cleaning
d) Coal characteristics (if available):
   1) Bulk density
   2) Lump size (by sieve analysis)
   3) Average BTU content
   4) Average percent sulfur
   5) Average percent pyritic sulfur
   6) Average percent framboidal pyrite
   7) Average percent ash

The following data will be gathered for each individual coal pile at each site:

a) Type of pile (active or reserve)
b) Drainage basin area
c) Description of pile construction
d) Description of pile compaction
e) Pile storage time

The following recorded data on ground water in the vicinity of the coal piles will be collected (if available):

Local geology description:

a) Types of soil beneath the coal piles
b) Average percent soil moisture
c) Soil infiltration rates
d) Ground water elevations

It is possible that soils survey maps of the sites may be available which will provide some of this information.

If available, the following historical meteorological data will be gathered at the site for the three-month period prior to the start of the field survey:

a) Total daily precipitation
b) Average daily air temperature
c) Average daily solar radiation
d) Average daily evaporation
e) Average daily relative humidity

Local airport and/or National Weather Service (NWS) records for the general plant vicinity will be sought for this information. If the utility maintains its own meteorological records, then the NWS information will be collected to supplement the plant data.

PRELIMINARY SITE WORK

The preliminary site work (prior to the field survey) will include the following tasks:

(1) Acquisition of representative coal samples for laboratory testing.

(2) Field testing of coal pile characteristics and ground water in the vicinity of the coal pile.

A second site visit will be necessary to accomplish both of these tasks.

Acquisition of Representative Coal Samples

Coal sampling is difficult because of the high variability of the coal itself. In sampling from a storage pile the difficulties are compounded by having a mixed source of coals and size segregation during handling. Therefore, acquisition of representative coal samples will be conducted in two phases: the first to determine overall variability based on ash content; the second to collect a sufficient number of samples to give a representative characterization of the parameters of concern.

Phase I - Sampling for Determination of Coal Variability--

The traverse method will be utilized to determine coal variability in each coal pile. This method involves obtaining samples systematically along a traverse of the entire pile. The methodology for Phase I is described in Appendix A of ASTM procedure D-2234 (refer to Appendix B of this report). Variability determinations will be based on gross ash content of the samples.

Phase II - Sampling of Coal for Precision--

The methodology as described in Section 7 of ASTM procedure D-2234 will be used to obtain samples of coal for precise results. The number of coal samples per coal pile will be determined by the results of Phase I.

Field Testing of Coal Pile Characteristics

The field tests to be performed on the coal piles at each site can be grouped into two major categories:

a) Physical measurements and visual observations of the coal piles
b) Coal pile hydrology testing
    1) Average percent moisture content of pile
    2) Water infiltration rates

Physical Measurements and Visual Observations

The physical measurements and visual observations listed below will be recorded for each coal pile. These data will serve primarily as inputs to the runoff model.

a) Average pile volume
    1) Average length
    2) Average width
    3) Average depth
b) Average pile side slope
c) Average depth of outer mantle
d) Average bulk density and porosity
e) Average depth of depression storage on top of pile
f) Average depth of ice and snow on top of pile
g) Gully erosion observations
    1) Approximate number of gullies
    2) Average width
    3) Approximate volume of displaced material
h) Number of visible springs from side of pile.
i) Typical particle size distribution of surficial coal

Coal Pile Hydrology Testing

Coal pile hydrology tests to be performed during the on-site visit include:

a) Average percent moisture content of each pile
b) Water infiltration rates

These data will be important both as inputs to the runoff model and also in water balance calculations for the coal piles.

Two types of moisture content measurements are necessary in the evaluation of coal pile hydrology. These include the measurements of the moisture content of the unsaturated zones and the dimensions of the saturated zones within the pile.

Standard drilling techniques will be used to determine the moisture content of these zones on a preliminary basis. Five to ten hollow stem auger borings, depending on the size of the coal piles, and split spoon samples taken every five feet will be used to obtain discrete samples for the determination of the variation of moisture content with depth in the unsaturated zone. Piezometers consisting of slotted PVC pipe with Ottawa sand packed in the lower part of the annulus and a bentonite seal above the zone of interest will be used both as a monitoring well for depth to the saturated zone and a sampling point for water quality.

Surface resistivity techniques will also be used to extend the data base. Changes in moisture content with depth should be correlative with the electrical resistivity of the pile as measured at the surface of the pile. In particular, the depth of the saturated layer will be determined. Using these data, as well as the data from the drilling program, a contour map of the top of the saturated zone in each pile will be prepared.

The moisture content of the unsaturated zone will be determined from the discrete samples obtained during the drilling program. ASTM procedure D3302, refer to Appendix C, will be employed to determine the total moisture content of these coal samples. The moisture content will be correlated with the resistivity information to extend this data base.

Water infiltration rates will be determined for each coal pile. These rates are to be used in developing the overall water balance for the piles and in determining erosion potential.

ASTM procedure D3385 (see Appendix D) will be used in the determination of infiltration rates. A double ring permeameter or infiltrometer will be used to measure these rates. Infiltration rates are time variable and reflect the change of permeability with saturation.

Ground Water Field Tests

In the same manner as the coal pile tests, average percent soil moisture and soil infiltration rates will be determined in the vicinity of the coal piles. Also, the depth to ground water, or ground water elevations, will be determined. However, the resistivity survey will not be performed as part of these tests.

Laboratory Testing of Coal Samples

Laboratory testing of the coal samples will include:

Physical testing - particle size distribution

Phase I - variability testing
- ash content

Phase II testing -
Percent framboidal pyrite
Percent sulfur
Trace metal content

Physical Testing--

Particle size distribution will be determined for the field collected samples by sieve analysis using ASTM method D410 (see Appendix E).

Phase I Variability Testing--

Ash content of coal serves as a gross indicator as to overall chemical variability. Ash content will be determined using ASTM method D3174, refer to Appendix F.

Phase II Variability Testing--

Three types of analyses will be required for overall chemical characterization of the coal pile: framboidal pyrite analysis, percent total sulfur, and trace metal chemistry. The pyrite and sulfur data will be used for evaluation of the acid forming potential and the trace metal data will be used for the water quality modeling program.

Framboidal Pyrite Analysis -- The framboidal pyrite analysis will be performed by the method described by F.T. Caruccio in his paper "Estimating the Acid Potential of Coal Mine Refuse" in The Ecology of Resource Degradation and Renewal, Chadwick and Goodman, (Editors) 1973. Samples will be crushed and formed into pellets. A point count will then be performed to determine the percent framboidal pyrite.

Sulfur Analysis -- Total sulfur determination will be made using the procedures outlined in ASTM D3177, Method A or Method B (refer to Appendix G).

Trace Metal Chemistry -- Trace metals content of the coal samples will be determined using the extraction procedures developed by the TVA in the study of coal and ash samples at the Colbert Steam Plant (in press). Extraction procedures and analytical procedures for specific metals are shown in Table 42.

TABLE 42. COAL EXTRACTION AND ANALYTICAL PROCEDURES

Metal	Extraction	Analytical Procedure
Be, Ca, Cr Cu, Fe, Mg, Mn, Mo Ni, Pb, V, Zn	Ashed, Digested with HF $HClO_4$	Atomic Adsorption
Al, Cd, Co	Digested with Lithium Metaborate	Atomic Adsorption
As	Digested with HNO_3 and H_2SO_4	Atomic Adsorption
Se, Sb	Digested with Eschka Mixture	Atomic Adsorption
F	Digested with NaOH	Specific Ion Electrode
Hg	Digested with Aqua Regia	Atomic Adsorption

Acquisition of Background Data Not Attainable at Site

In addition to the recorded background data obtained during the initial on-site visit, additional background data will be obtained from outside sources. These data are listed below along with their source.

a) Soils survey maps - Soils Conservation Service (SCS)

b) Surficial geology maps - United States Geological Survey (USGS)

c) Bedrock geology maps - United States Geological Survey (USGS)

d) Historical meteorological data - National Oceanic and Atmospheric Administration (NOAA) - National Weather Service

The soils survey and geology maps will provide information on the types of material beneath the coal piles. In addition, approximate ground water elevations will be determined.

The historical meteorological data will be obtained for the three month period prior to the start of the field survey at each of the 12 sites. These data will be used for several purposes:

a) as input to the runoff model;

b) to show trends in meteorological conditions at the site; and

c) to determine when the last storm event occurred at the site so that pre-runoff event water content can be estimated.

DESIGN OF FIELD PROGRAM

The field measurements program will be designed to attain the objectives of the overall program with minimal cost. The individual tasks to be addressed in the development of the field program are discussed in detail in the following sections.

Parameters to be Analyzed

Table 43 is the list of pollutant parameters to be monitored in the runoff and base flow streams during the field program.

Continuous monitoring of pH, temperature, and conductivity will be conducted throughout the field program. pH is important in the determination of the degree of acidity or alkalinity of the stormwater runoff and base flow streams. It will also be an important parameter for correlation analysis and phase distribution analysis of the various pollutant parameters. Water temperature may serve as an indication of the degree of pyrite oxidation occurring within the pile. Conductivity can be correlated with total dissolved solids (TDS) and through initial testing may preclude the need for extensive TDS analysis.

From previous studies[9, 10, 11, 12] related to coal pile runoff, it has been shown that TDS, total suspended solids (TSS), sulfate, iron, manganese, and aluminum are characteristic pollutants. TSS will be analyzed for correlation purposes and for pile washoff coefficient determinations.

Antimony, arsenic, beryllium, cadmium, calcium, total chromium, copper, lead, magnesium, mercury, nickel, selenium, and zinc were identified in past literature reviews[13, 14] as being present in coal pile runoff. In addition, cobalt, fluoride, molybdenum, and vanadium

TABLE 43. POLLUTANT PARAMETERS TO BE ANALYZED DURING FIELD PROGRAM AND MINIMUM VOLUME REQUIRED

Sample Volume Requirements	Minimum Volume Required, ml(1)
pH	-
Temperature	-
Conductivity	-
Acidity/Alkalinity	100
Total Suspended Solids (TSS)	100
Total Dissolved Solids (TDS)	100
Total Organic Carbon (TOC)	25
Sulfate (SO_4)	50
Total Hardness	100
Metals (Total and Dissolved Forms):	300(2)
Aluminum (Al)	
Antimony (Sb)	
Arsenic (As)	
Beryllium (Be)	
Cadmium (Cd)	
Calcium (Ca)	
Chromium, Total (CrT)	
Cobalt (Co)	
Copper (Cu)	
Iron (Fe)	
Lead (Pb)	
Magnesium (Mg)	
Manganese (Mn)	
Molybdenum (Mo)	
Nickel (Ni)	
Selenium (Se)	
Vanadium (V)	
Zinc (Zn)	
Fluoride (F)	300
Mercury (Hg)	200(3)

(1) Minimum volumes obtained from Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020.

(2) 100 ml for total form
200 ml for dissolved form

(3) 100 ml for total form; 100 ml for dissolved form.

were found to be present in coal pile leachate studies[15]. Included in this list of metals are the metals identified in the list of 129 Priority Pollutants, with the exception of silver and thallium.

Due to the large variety of metals which may be present in the runoff and base flow streams, coal samples at the two "test" sites will be screened using laboratory procedures as discussed previously in "Laboratory Testing of Coal Samples". This may allow elimination of certain metals from extensive analysis during the field program. The metals earmarked for analysis will be analyzed in both their total and dissolved forms to determine speciation or phase distribution according to the acidity or alkalinity of the stormwater runoff and base flow.

Total organic carbon (TOC) will be analyzed as an indication of the presence of organic pollutants.

The sample volume requirements will be dictated by the pollutant parameters to be analyzed in the field. The minimum sample volume required for each of the parameters identified in Section 4 is shown in Table 43. These requirements indicate that the volume of each runoff or base flow sample should be at least 1275 milliliters (ml) to ensure sufficient sample for analysis of all of the pollutant parameters.

Number of Samples and Sampling Frequency

The number of samples and the frequency of sampling during a storm or dry day sampling event will be based on:

a) The specific needs of the runoff model, in terms of dry day or base flow input; and

b) The data requirements for establishing a base of coal pile runoff data.

Data on base flow or dry day conditions will be necessary as input to the runoff model. This data will be necessary to calibrate the runoff model, both qualitatively and quantitatively, for the particular coal pile being modeled. As such, the number of samples and sampling frequency should be compatible with the input needs of the model. This will necessitate daily sampling of base flow during dry day conditions. Assuming an average of two base flow streams per coal pile, daily grab samples from each stream will be composited to produce one sample for chemical analysis. It is estimated that, due to climatic patterns in different parts of the country, 36 total base flow samples will be analyzed per site for all pollutant parameters.

The number of samples and sampling frequency for storm events should be selected to:

a) be compatible with the runoff model outputs for ease in comparison; and

b) be sufficient to establish a data base on coal pile runoff for different climatic regions, coal types, and coal pile configurations.

In order to satisfy the above requirements, one of each of the following four types of storm events should be sampled at each site:

a) 24-hour storm
b) 12-hour storm
c) 4-hour storm
d) 2-hour storm

Samples will be obtained half-hourly during the first four hours of the 24 and 12-hour storm events. Hourly samples will then be taken for the remainder of these events. Half-hourly samples will be collected throughout the two and four-hour storm events. This schedule will result in a total of approximately 112 total runoff samples per utility test site. This number assumes an average of two runoff streams per coal pile, each of which will be monitored separately, and one coal pile per utility site. This can be broken down further as 56 total samples for each runoff stream.

Sampling, Flow Monitoring, and Meteorological Equipment

The various types of equipment to be employed for runoff sampling, runoff and base flow monitoring, water quality measurements, and meteorological data collection are discussed below.

Runoff samples will be collected automatically during storm events using pumping type sequential samplers. These samplers will collect samples automatically at a pre-set volume and frequency. Sampling is triggered by a flow actuator switch which senses a change in water level. The initial work plan calls for sample collection on a time proportional basis. However, depending on the characteristics of the runoff quantity, it may be desirable to switch to flow proportional sampling. For example, if the runoff exhibits a "first flush" effect, it will be desirable to sample more frequently then half-hourly during the first flush period to adequately characterize the runoff quality. The automatic sampling equipment minimizes manpower requirements and allows workers to perform other tasks during sample collection.

Coal pile runoff and base flow will be monitored continuously during the field program. As previously mentioned, it is assumed that there will be two streams emanating from a coal pile. Each of these streams

will be monitored separately. A primary measuring device, such as a weir or flume, will be installed across each of these streams. These devices indicate the level of the liquid passing through or over the device. Flumes are specially shaped open channel flow sections which provide a restriction in area. These devices are compatible with coal pile runoff flow measurement because they are self-cleaning. The high velocity through the flume prevents deposition of solids and sediment. Also, the accuracy of the flume is less affected by varying approach velocities than the weir. However, the flume is more expensive than a weir in terms of initial cost and is generally less accurate.

Weirs are basically obstructions built across the stream over which the liquid flows, often through a specially shaped opening. The weir is less expensive than the flume in terms of initial costs. However, maintenance costs are higher due to the deposition of material behind the weir. In addition, time delays behind the weir can cause problems with the correlation of highly variable flow conditions to water quality. Assuming that the weir is well maintained, greater accuracy in flow measurement is obtained than with the flume.

The situation at the site will determine which of these primary devices to use. In areas of relatively flat or average slope, flumes will be employed to prevent deposition of material. Weirs will be installed in areas of steeper slope.

Existing weirs or flumes at the sites will be utilized as much as possible.

The weirs and/or flumes will be used in conjunction with a secondary measuring device, or flow meter, to measure the flow rate of the stream. The flow meter measures the water level in the primary device with a submerged plastic tube which continuously emits bubbles upstream of the weir or flume. As the water level changes, back-pressure changes in the tubing are measured with a sensitive electronic transducer. The transducer then converts this pressure into a digital electronic signal proportional to water level. The water level is then converted to flow rate by an electronic module specially designed for each type of primary measuring device. Flow rate is recorded on a built-in strip chart recorder and total flow is displayed on a six-digit totalizer.

The pH, conductivity and temperature of the runoff and base flow will be monitored continuously during the field program using automatic equipment. Each piece of equipment will be connected to one multichannel chart recorder which will facilitate comparison of these parameters and indicate significant trends. Special probes will be used for each parameter which are resistant to highly acidic water. In addition, these probes will be subject to a regular maintenance program.

As previously discussed in the section "Selection of Locations for Installation of Meteorological Station and Field Laboratory Trailer", the following meteorological data will be continuously recorded at each site:

a) Precipitation
b) Air temperature/relative humidity
c) Solar radiation
d) Evaporation

Precipitation will be measured with a heated tipping bucket gage. This standard National Weather Service gage is operable in temperatures down to -20°F, at a maximum snowfall rate of 3 inches per hour. The gage will be used in conjunction with an event-type recorder and counter. The recorder indicates every 0.01 inch of precipitation while the digital counter shows cumulative rainfall.

Air temperature and relative humidity will be measured with a hygrothermograph. This instrument, made to National Weather Service specifications records both temperature and relative humidity on the same time coordinate of a curvilinear chart. The temperature sensitive element is a highly polished chrome plated bourdon tube. The humidity element consists of multiple strands of specially treated human hair. Through a system of linkages these elements operate individual recording pens.

Solar radiation will be measured with a bimetallic recording pyranometer (actinometer). The measuring element consists of two bimetallic strips mounted to show pen motion corresponding to a differential temperature with ambient temperature compensation. A blackened strip is exposed to both the sun's radiant energy and the ambient temperature, while a chrome plated strip is exposed only to ambient temperature.

Evaporation will be measured with an evaporation pan equipped with a chart recorder. The instrument is designed to record the amount of water evaporated from a surface area of 250 square centimeters.

Plan for Shipping Samples for Laboratory Analysis

In order to maintain the integrity of the samples prior to laboratory analysis, the samples will be shipped to the laboratory as quickly as possible after a sampling event. This will necessitate establishing a plan for shipping the samples. A courier service will be chosen for each utility site. The courier service should provide direct pickup and delivery to the laboratory within a 24 hour period. The courier will be notified as soon as the field personnel have an idea when the samples will be ready for shipping in order to expedite sample delivery.

Laboratory Analyses

Analytical procedures described in EPA's Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020, March 1979) will be used

for the analysis of the pollutant parameters listed in Table 43. In addition, a rigorous quality control program will be maintained for all samples analyzed in the laboratory. This program is based on the general guidelines given in EPA's Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/4-79-019, March 1979). This program suggests guidelines for:

a) Laboratory services
b) Instrument selection
c) Glassware
d) Reagents
e) Analytical performance
f) Data handling and reporting
g) Laboratory safety

In addition, the program will include the following:

a) Triplicate analyses are performed for each parameter on 10% of the samples.

b) Monthly analysis of secondary quality control samples are prepared by the laboratory manager or his designate.

c) 10% of the samples are spiked by the laboratory manager with known amounts of the parameters of interest and re-analyzed to determine the percent recovery. A Shewhart control chart is used for the percent recovery control. (EPA, Handbook of Analytical Quality Control in Water and Wastewater Laboratories, 1979.)

d) Standard curves are determined for each analysis using the appropriate standard. Least squares linear regressions calculations are used in determining the "best fit" to the data. Correlation coefficients are also calculated and are required to be 1.000 over the linear range of the method.

e) Quarterly EPA Quality Control samples are obtained from the Environmental Monitoring and Support Laboratory in Cincinnati. The analytical results are required to be within a 95% confidence level of the true value.

f) One sample per month will be sent to an outside laboratory as part of an intra-laboratory quality control program.

The quality control program thus provides for the analysis of blanks, blind spiked samples, and EPA quality control samples on a routine basis. In addition, control limits are established for the operation and maintenance of all laboratory equipment to provide strict limits on the acceptability of the data.

DATA REDUCTION AND PRESENTATION

During the field measurements program, a large amount of raw field data as well as historical data in various forms will be collected at each site. The various data forms include:

a) Field data sheets
b) Plant operating records
c) Plant coal analysis records
d) Soils survey and geology maps
e) NOAA historical meteorological data magnetic tapes
f) NOAA local climatological data sheets
g) Plant meteorological records
h) Strip charts for continuous monitoring data
i) Laboratory analysis results sheets

These data will be both alphanumeric and numeric. The numeric data, including the continuous monitoring data in the field as well as the results of the laboratory analyses, will be digitized and made easily accessible through magnetic tape. In addition, both this data and the alphanumeric data from each site will be manipulated using computer programs for final presentation.

The first task in the manipulation of the field data will be the reduction or digitization of the strip chart data. This will be accomplished using a Gerber Scientific Instrument Company Digitizer. The following procedure will be followed for processing field data:

a) Scan strip charts for validity
b) Digitize data
c) Input data into a data file
d) Produce preliminary listings of data
e) Edit the data
    1) Manually input correct data
    2) Delete questionable data
    3) Correct data for field calibrations
f) Produce finalized data listings

The types of field data which will be digitized and the frequency of the Gerber readings are shown in Table 44.

TABLE 44. TYPES OF FIELD DATA FOR DIGITIZATION AND FREQUENCY OF GERBER READINGS*

Type of Field Data	Frequency of Readings, Minutes**
Flow	15
Runoff pH	30
Runoff Conductivity	30
Runoff Temperature	30
Precipitation	15
Air Temperature/Relative Humidity	60
Solar Radiation	60
Evaporation	30

* Readings refer to data points taken from the strip charts using the Gerber Scientific Instrument Digitizer.

**All readings are actual values from the strip charts.

The field and historical data from each site will appear in tabular form and/or as plots showing parameter variations with time. In addition, a statistical analysis of the field data will be included. Computer programs will be used to generate the tabular summaries, plots, and statistical analysis. The final format of the data from each site will be broken down by major categories of data as discussed below.

Tabular summaries will be generated for the following categories of data:

a) General plant information
b) Coal data
c) Coal pile data
d) Ground water data

In addition, a summary table will be generated for each site containing the following information:

a) Number of storm events

b) Description of each storm event

   1) Form of precipitation (rain, snow, sleet, hail)
   2) Total precipitation
   3) Duration
   4) Intensity

c) Description of conditions between events

   Number of antecedent dry days

The meteorological data from each test site will appear both in tabular form and as plots. Both forms of this data will be generated by computer programs. The meteorological data will be broken down into historical and actual field data.

Historical meteorological data for the three-month period prior to the beginning of the field survey at each site will be presented in the following forms:

a) A summary table of all data pertaining to:

   1) Total daily precipitation
   2) Average daily air temperature/relative humidity
   3) Average daily solar radiation
   4) Average daily evaporation

b) Plot of this daily data. Figure 19 is an example of such plots for total daily precipitation and average daily air temperature.

As with the historical meteorological data, the continuous field meteorological data will be presented in tabular form as well as plots showing variations with time.

In the same manner as the meteorological data, the runoff and base flow data will appear both in tabular form and as plots. Each form will be generated by computer programs. The runoff data will be broken down into continuous measurement data and routine laboratory analysis data. Included with the laboratory analysis data will be periodic rainfall pH and acidity data.

As previously discussed, the following parameters will be measured continuously at each site during both storm events and dry days:

a) Flow
b) pH
c) Conductivity
d) Water temperature

This data will be presented in the following forms:

a) Summary Tables

   A separate summary table will be generated for each parameter showing all of the data collected in the field. If there is more than one runoff stream from a coal pile, these tables will be presented for each stream.

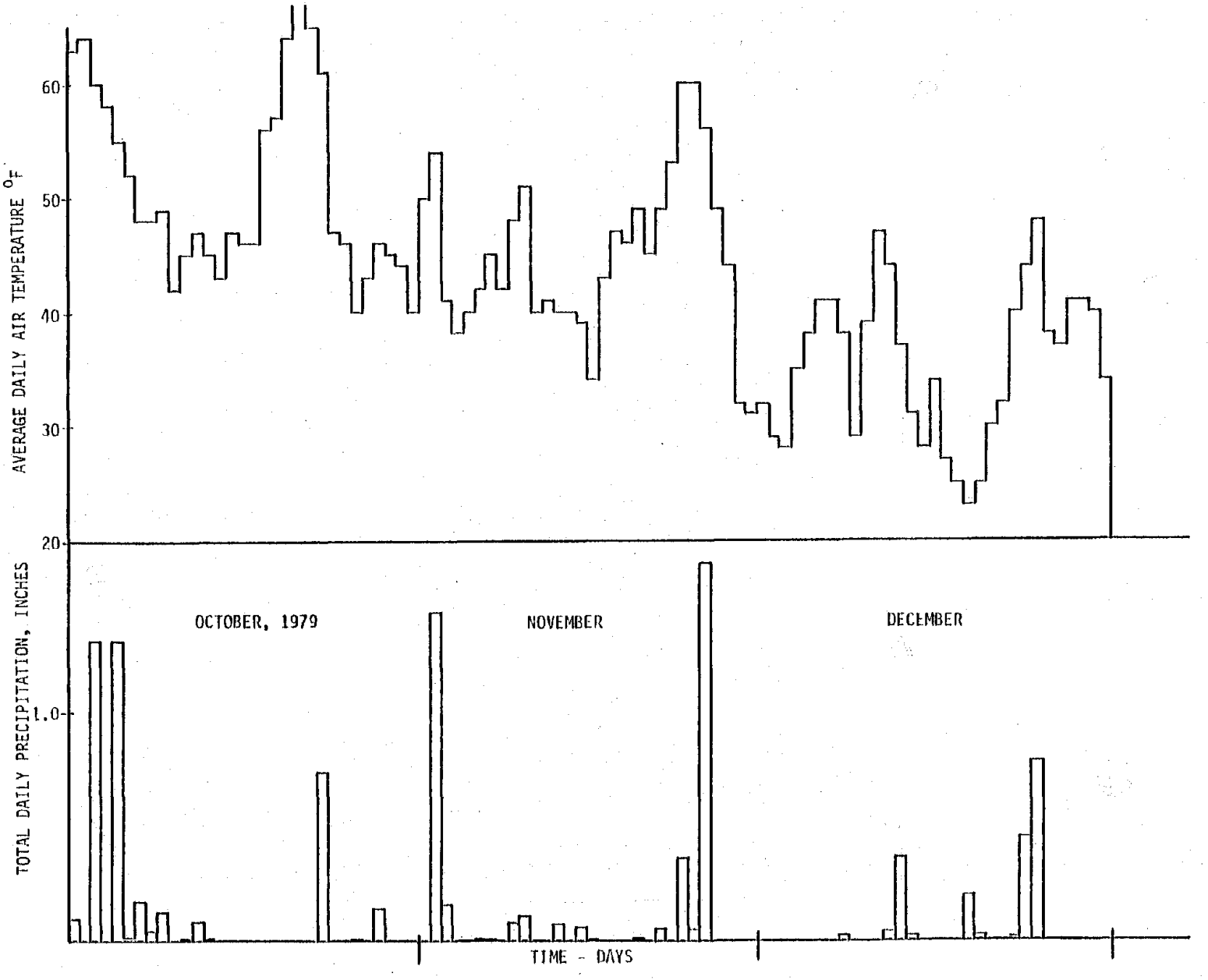


FIGURE 19: EXAMPLE OF HISTORICAL METEOROLOGICAL DATA PLOTS

b) Plots (Storm Events Only)

Two types of plots will be generated for each stormwater runoff stream:

1) Plot of the runoff hydrograph and the rainfall hyetograph for each storm event. Figure 20 is an example of such plots.

2) Plots of the runoff hydrograph and the continuous measurement data (pH, conductivity, and water temperature). Figure 21 is an illustration of these plots.

The routine laboratory analysis data will include the results of the chemical analyses performed on stormwater runoff and dry day base flow samples. Periodic rainfall pH and acidity measurements will also be included in this category. Storm event and dry day (base flow) data will be presented separately.

Storm event data will be presented in both of the following forms:

a) A summary table for each runoff stream for each storm event showing all of the laboratory analysis results and rainfall quality measurements.

b) Plots of the runoff hydrograph and a maximum of three "pollutographs" (pollutant concentration versus time) on the same figure. One or more of the continuous measurement parameters may replace the pollutographs to show interesting trends. For example, a figure showing runoff pH, total arsenic, and dissolved arsenic, along with runoff flow, versus time would be particularly useful in showing the change in arsenic speciation with pH as a function of time.

A summary table will be generated for dry day sampling events showing the average values of the continuous measurement data for the sampling event and the laboratory analysis results.

The results of the statistical analysis of each storm event will be presented in tabular form. The statistical analysis and tabular summary will be performed by computer programs. The summary tables will include:

a) Means, coefficients of variation, and standard deviations for all runoff data collected during a storm event.

b) Statistical summaries indicating worst case conditions monitored during a storm event.

c) Correlation analysis of chemical parameters for each storm event.

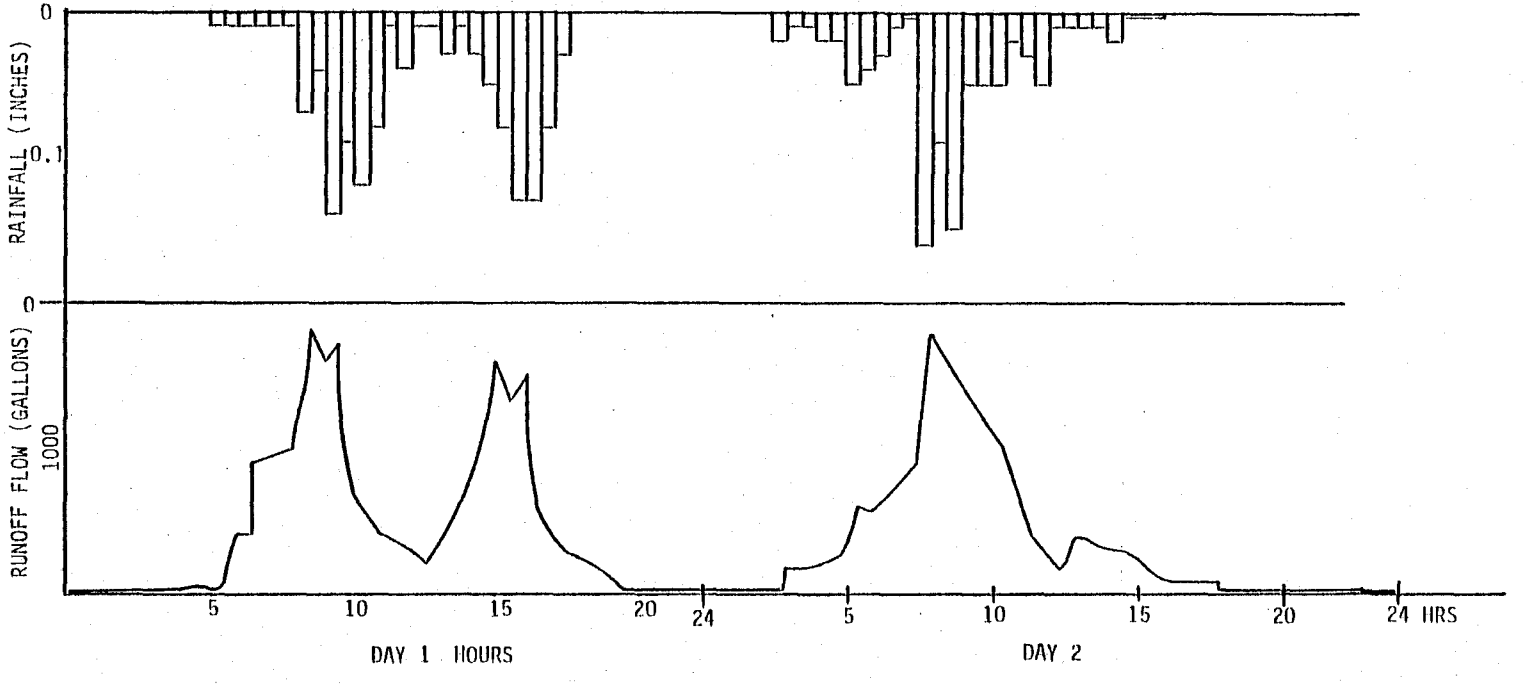


FIGURE 20: EXAMPLE OF RUNOFF HYDROGRAPH AND HYETOGRAPH PLOTS

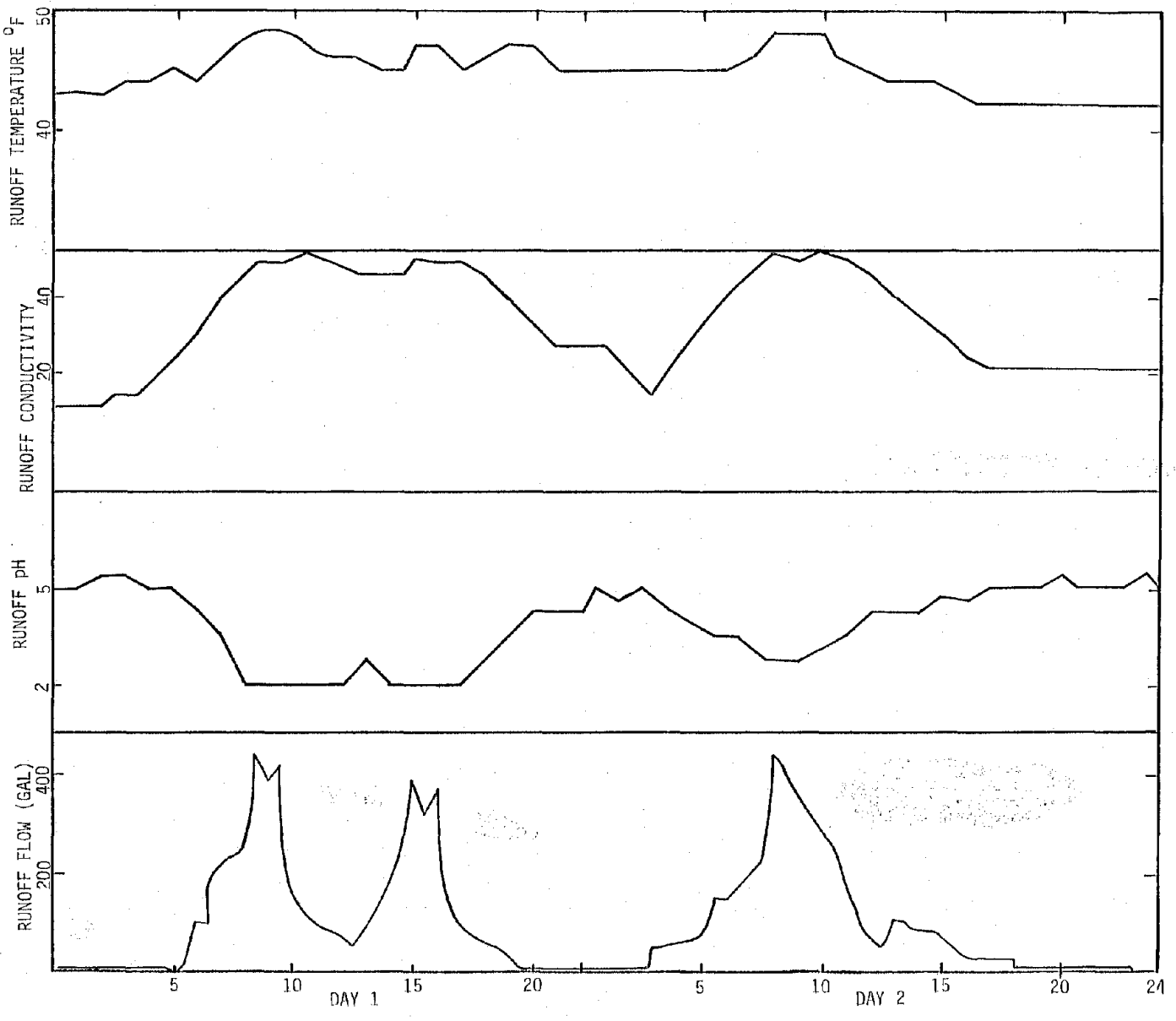


FIGURE 21: EXAMPLE OF RUNOFF HYDROGRAPH AND CONTINUOUS MEASUREMENT DATA PLOTS

The final requirement for data manipulation is the generation of a magnetic tape containing data for direct input into the coal pile runoff model. This will necessitate that the data be in a form which is compatible with the model. A detailed discussion of this data is included in Section 4.

PROGRAM COSTS AND TIME SCHEDULE

The entire runoff program at each utility test site will cover a 22 week period from the initial site visit to the final report preparation. This period includes the 10 week runoff sampling program. The manpower and equipment requirements for the program are discussed in the following sections.

Manpower Requirements

Table 45 shows an estimate of the manpower requirements for the runoff program broken down by major tasks and personnel categories. The Senior Engineer/Scientist will act as project manager and review the results of the program. The Principal Engineer/Scientist will supervise the laboratory analysis of the water and coal samples. The Engineer/Scientist category will include the field supervisor and the computer analyst. The field supervisor will develop the field procedures manual, direct the field effort, analyze the results, and prepare the final report. The computer analyst will be responsible for all computer programs for data reduction and final presentation. The Laboratory Technician will be responsible for performing all laboratory analyses, except those performed in the field. The Technician will handle the field preparation and conduct the field work, as well as perform the field laboratory analyses.

Other Direct Costs

Table 46 shows estimated rental and purchase costs for various equipment as well as other direct costs excluding travel and subsistence for the runoff program. These estimates include the cost for a drilling crew and an outside laboratory to perform special analyses, such as determination of the percent framboidal pyrite in the coal samples. Also included is the estimated cost for computer usage, i.e., the computer time required for data reduction and presentation. These costs are broken down by major task.

TABLE 45. ESTIMATED MANPOWER REQUIREMENTS FOR RUNOFF PROGRAM (PER TEST SITE)

Task	Senior Engineer/ Scientist (Hours)	Principal Engineer/ Scientist (Hours)	Engineer/ Scientist (Hours)	Laboratory Technician (Hours)	Technician (Hours)
1. Project Management	100	-	-	-	-
2. Initial Site Visit	-	-	32	-	-
3. Preliminary Site Work	-	-	40	-	112
4. Laboratory Coal Testing	-	20	-	166	12
5. Acquisition of Additional Background Data	-	-	8	-	-
6. Field Procedures Manual	-	-	16	-	16
7. Field Survey	-	-	240	-	728
8. Laboratory Analysis	-	56	-	900	-
9. Data Reduction and Presentation	-	-	254	-	100
TOTAL HOURS	100	76	590	1065	968
GRAND TOTAL					2800

TABLE 46. ESTIMATED EQUIPMENT RENTAL AND PURCHASE COSTS AND OTHER DIRECT COSTS FOR RUNOFF PROGRAM (Spring, 1980 Dollars)

Task 3 - Preliminary Site Work	
Drilling Crew	$2,200
PVC Screen and Sand	1,000
Resistivity Survey	1,500
Miscellaneous	200
Task 4- Laboratory Coal Testing	
Laboratory Expendables (Chemicals; Glassware)	$ 500
Analysis for Framboidal Pyrite	2,200
Miscellaneous	50
Task 5 - Acquisition of Additional Background Data	
NOAA Tapes; Geological Maps	$ 200
Task 7 - Field Survey	
Equipment Rental and Purchase Costs	
Sequential Samplers (3) 10 wks @$50/wk (Rental)	$1,500
Flow Meters (3) 10 wks @$75/wk (Rental)	2,250
pH Meters (3) 10 wks @$50/wk (Rental)	1,500
Conductivity/Temperature Meters (2) (Purchase)	800
Three channel recorders (2) (Purchase)	4,400
Recording Rain Gage (1) (Purchase)	950
Hygrothermograph (1) (Purchase)	400
Pyranometer (1) (Purchase)	500
Evaporation Recorder (1) (Purchase)	500
Flume or weir (2) (Purchase)	1,000
Mobile Lab (1) 10 wks @ $150/week (Rental) 1,500	
Shelter (1) (Purchase)	300
Shipping	1,000
Vehicle Rental	$1,000
Expendables	$1,000
Chart paper; pH probes; buffer/solution, conductivity cells; ice	
Task 8 - Laboratory Analysis	
Laboratory Expendables (Chemicals; Glassware)	$ 300
Task 9 - Data Reduction and Presentation	
Computer Usage	$ 900

Time Schedule

Figure 22 indicates the estimated time schedule for conducting the entire program at each utility site. The entire effort, including 10 weeks of field monitoring, will be approximately 22 weeks.

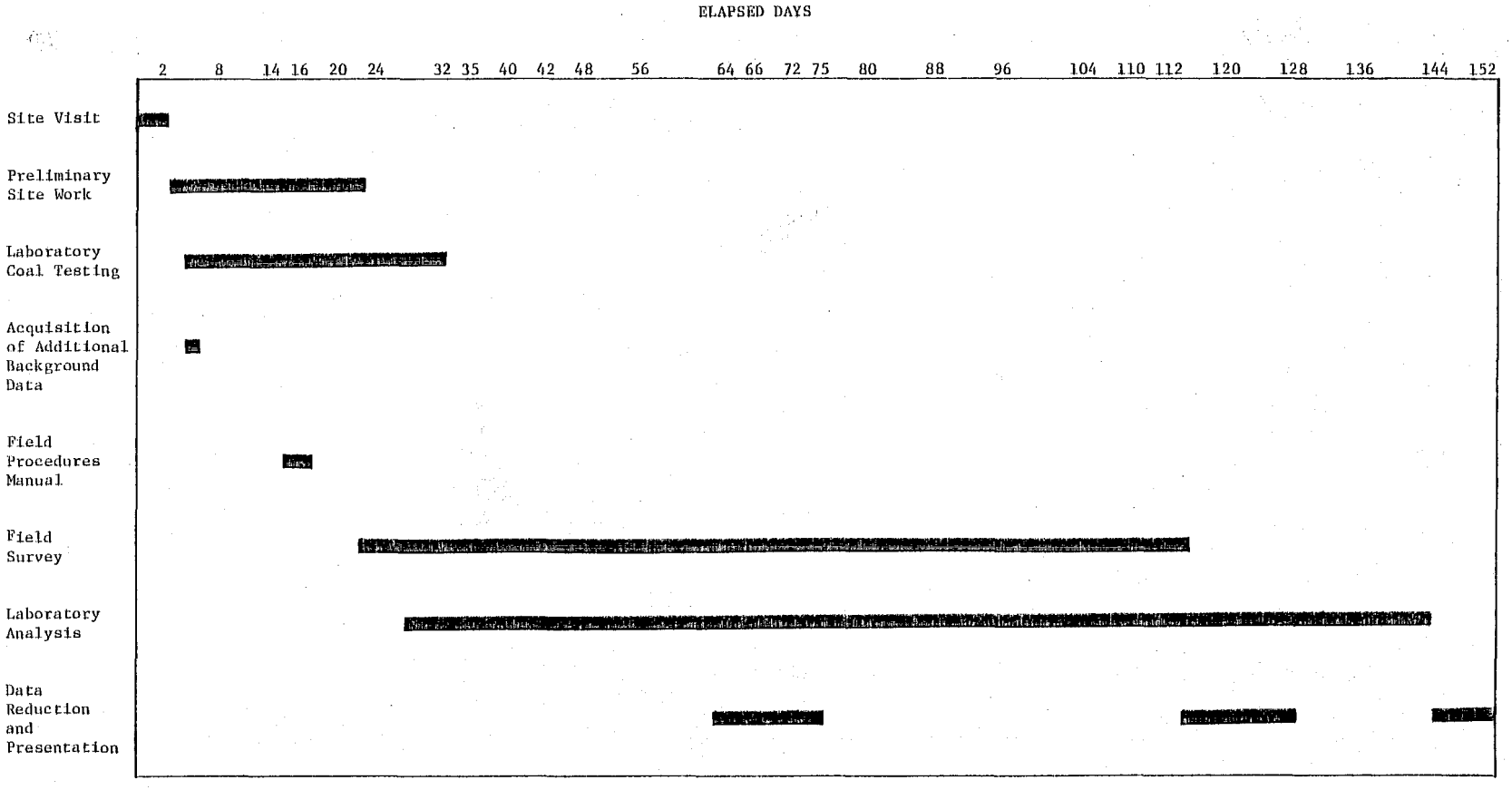


FIGURE 22: ESTIMATED TIME SCHEDULE FOR RUNOFF PROGRAM (PER UTILITY SITE)

SECTION 6

FIELD PROCEDURES MANUAL

INTRODUCTION

An important step in the design of the field program for monitoring coal pile runoff is the development of a field procedures manual. This manual will serve as a guideline or working document for conducting the field monitoring program. Among the areas discussed in detail in the manual are procedures for the training of field technicians, operation and installation of meteorological, flow monitoring, and water quality monitoring equipment, dry day sampling, and storm event sampling. In addition, the manual covers procedures for coal sampling, coal pile and ground water testing, sample identification and custody, sample preservation, on-site chemical analyses, and sample shipping.

The field procedures manual is presented in a general manner which is applicable to any utility site. However, once a specific utility site is chosen, it will be necessary to modify the manual to reflect the specific site conditions. In addition, the manual presented herein is written in draft form. The procedures described herein will be field tested at the two "test" sites, after which time the procedures will be finalized. Procedures will also be added or deleted as necessary.

GENERAL RULES OF CONDUCT

During the field monitoring program there are a number of general rules of conduct which must be followed to ensure the safety of the field crew. All field personnel will be briefed on site safety regulations at the beginning of the program. Each person will also receive a copy of these regulations. It is expected that the field crew will adhere to these without exception. Should any questions arise, these should be promptly resolved with the utility contacts. Rules for all field personnel are summarized as follows:

a) Sign in and out at the guardhouse. This must be done without exception.

b) Check each day with the utility contacts. This should be done immediately after gaining access to the site if they are available. Any questions regarding the site should be directed to them.

c) Use extreme caution when walking or driving around the site. Be aware that all trucks and trains have the right of way and will take it. Vehicles should be driven in low gear. Observe speed limits, flashing lights, and stop signs.

d) Make sure that all equipment is as inconspicuous as possible at all times to prevent vandalism. The equipment shed and mobile laboratory trailer should be kept locked when unattended.

Other responsibilities required to expedite to the program include the following:

a) Call the nearest National Weather Bureau daily regardless of the weather.

b) Establish a laboratory vendor for purchasing expendables such as chemical preservatives, glassware, and filters. In terms of preservatives, nitric acid (HNO_3) and sulfuric acid (H_2SO_4) will be required.

c) Make sure that everything is ready for the next sampling event. This entails:

1) Filling the sequential sampler canisters with clean bottles.

2) Cleaning up the mobile field laboratory.

3) Making sure that there is enough distilled water, dry ice, chemical preservatives, and clean sample shipping bottles.

4) Contacting the courier to forewarn him that samples will be shipped in the near future.

TRAINING OF FIELD TECHNICIANS

Due to the nature of stormwater runoff sampling, the designated field technicians will be trained both in the laboratory and in simulated field conditions before any sampling takes place. The two job functions for conducting this type of field work are separated between:

1. Technical Specialist - permanent on-site technician
2. Technical Assistant - assistant during runoff event sampling.

The Technical Specialist will be responsible for carrying out all aspects of the monitoring program from initial equipment installation to the sample analyses to be performed in the mobile laboratory. He will be the prime on-site representative of the monitoring effort and will remain on-site for the duration of the field program. Responsibilities assigned to the Technical Specialist include the following:

1. Equipment operation, maintenance and calibration
2. Dry period sampling
3. Wet period sampling
4. On-site safety procedures
5. Field analyses of collected samples
6. Field preparation of samples to be shipped to the laboratory
7. Documentation of field activities

The Technical Assistant has the responsibility to be ready, on a moment's notice, to travel to the site when a runoff event occurs and assist in the collection, analyses and preparation of samples. This person should not only be familar with the seven duties of the Technical Specialist shown above but also be most familiar with the procedures of sample collection, preparation, analyses and documentation. His prime duty is to lend a hand to the on-site technician at the time of a runoff event. Once the sampling, sample preparation, analyses and documentation has been completed, the Technical Assistant will return to his former duties.

Depending on the number of runoff streams to be monitored, there may be more than one Technical Assistant designated for field duty. In either case, all field personnel will be trained together in all aspects of the field program.

Training begins with a classroom type series of discussions on each component of the field effort. The topics with a listing of components is shown below:

	Topic		Components
1.	Field Objectives	-	program objectives, monitoring objectives, site objectives, types of runoff events, discrimination of data
2.	Site Descriptions	-	climatic conditions, features of the coal pile, general layout of monitoring stations

3. Equipment Installation - meteorological equipment, runoff flow measurement system, automatic sampling devices, and continuous water quality recorders, field laboratory, plant facilities

4. Equipment Operation and Calibration - review of the operator's manual and calibration procedures for each piece of equipment including:

    1. heated tipping bucket rain gage
    2. hygrothermograph
    3. solar pyranometer
    4. evaporation recorder
    5. pH meter
    6. conductivity and temperature meter
    7. multichannel recorder
    8. sequential sampler
    9. recording flow meter
    10. Parshall flume
    11. sample actuator
    12. pH, temperature and conductivity probes

5. Equipment Maintenance - daily maintenance, checks for field calibrations, probe cleaning and replacing, chart paper replacement, backup devices, procedures to follow when equipment breaks down.

The training program should be divided between classroom discussion and hands on experience.

The second set of topics can be instructed within the chemistry lab. It is assumed that most technical staff have had some chemical experience in their background but for consistency in procedures, the field analysis of samples and preparation of samples for other analyses should be given consideration in the training of technicians. The following items should be reviewed:

6. Total Suspended Solids and Total Dissolved Solids Analysis - equipment, amount of sample, calculations, expendables, recording data, procedures

7. Alkalinity/Acidity Analysis - equipment, amount of sample, calculations, expendables, recording data, procedures

8. Filtering Metals for Dissolved Analysis - equipment, procedures, calculations, recording data

9. Preservation and Refrigeration of Samples for Shipment - EPA procedures, logging samples in, labeling instructions

10. pH of Rain - equipment, procedures, recording data

Once the first 10 topics have been discussed, the training should be centered on the actual site specific applications for all the procedures. With the field equipment on hand, the instructor should give a series of instructions to each technician, in the form of a site set-up sequence, and allow him to physically set-up and start the equipment into operation. Different set-ups can be chosen for practice and the following minimal situations should be simulated:

11. Dry Weather Base Flow Monitoring - composite sampling; calibration checks on the flow meter, pH, conductivity and temperature probes; sample frequency; contingency procedures

12. Pre-storm Event Procedures - pre-storm monitoring of precipitation, adjusting all equipment for runoff monitoring, alerting Technical Assistant, preparing all sample containers and mobile field laboratory

13. Wet Weather Sampling - estimating the length of the storm; selecting sample frequencies; final equipment status check; collection, preparation and analyses of samples

14. Reporting and Documentation - daily site log, sample preparation log, analytical log, equipment maintenance log, sample labeling, sample custody procedures

15. Post Storm Event Procedures - reprogram sequential sampler and flow meter, calibration checks on continuous monitors, verification of meteorological data, shipment of samples to analytical laboratory

The training session should conclude by allowing the technicians to ask questions. Emphasis should be placed on the historical problems in dealing with stormwater runoff programs. The idiosyncrasies of each piece of equipment should be spelled out and the detection of a faulty piece of data or faulty equipment operation should be of prime concern. Keeping the training rigorous from the start with as much hands-on practice as possible can only help simplify the procedures when the first storm event occurs. Finally, the training in this type of study

should be considered an ongoing process. Having the trained technicians install the equipment and even practice a trial run using simulated runoff, e.g., a fire hose discharging through the flume, can only help prevent mistakes during the crucial storm event monitoring.

ACQUISITION OF REPRESENTATIVE COAL SAMPLES

As part of the preliminary site work (prior to the actual runoff sampling program), representative coal samples will be obtained from the coal pile for laboratory testing. This will be conducted in two phases. Phase I will involve determining the overall coal variability. Phase II will entail collecting a sufficient number of samples to provide representative coal quality data.

Phase I - Sampling for Determination of Coal Variability

The traverse method will be employed to determine coal variability. This is a systematic method for obtaining coal samples along a traverse of the entire coal pile. This method is described in Appendix A of ASTM procedure D-2234. Appendix B of this report includes this method.

Phase II - Collection of Coal Samples for Precision

The methodology as described in Section 7 of ASTM procedure D-2234 will be used to obtain representative coal samples for laboratory analysis. Phase I will determine the number of coal samples required. This method is described in detail in Appendix B.

Each coal sample should be placed in a ziplock bag for preservation. The samples should be shipped to the chemical laboratory as soon as possible after collection.

COAL PILE HYDROLOGY AND GROUND WATER TESTS

Coal pile hydrology tests to be conducted as part of the preliminary site work include determination of the average percent moisture content of the coal pile and water infiltration through the pile. The ground water tests to be performed include determination of average percent moisture content and infiltration rates, as well as the depth to ground water, or ground water elevations.

Coal Pile Hydrology Tests

Standard drilling techniques will be used to determine the moisture content in the pile on a preliminary basis. Five to ten hollow stem auger borings, depending on the size of the coal piles, and split spoon samples obtained every five feet will be used to collect discrete samples for determination of the variation of moisture content with depth in the unsaturated zone. Piezometers consisting of slotted PVC pipe with Ottawa sand packed in the lower part of the annulus and a bentonite seal above the zone of interest will be used both as a monitoring well for determining the depth to the saturated zone and as a sampling point for water quality.

Surface resistivity techniques will also be used to determine the depth of the saturated zone. The results of the resistivity survey and the drilling program will be used to construct a contour map of the top of the saturated zone in the pile.

The drilling program and the resistivity survey will be conducted by an outside drilling crew. The field crew will be on hand at all times to supervise the drilling and obtain the coal samples. The moisture content of the discrete sample will be determined in the field according to ASTM procedure D3302. This procedure is included in Appendix C.

The field crew will also be responsible for obtaining water samples from the coal pile for laboratory analysis. A total of five samples will be obtained at various locations throughout the pile. These should be treated in the same manner as the runoff and base flow samples (see Section 6, "Sample Preservation Procedures and Field Laboratory Analyses").

Infiltration rates into the coal pile will be determined using ASTM procedure D3385. This procedure is described in Appendix D. A double ring permeameter or infiltrometer will be used to measure these rates.

Ground Water Tests

In the same manner as the coal pile hydrology tests, the average percent soil moisture content and soil infiltration rates will be determined in the vicinity of the coal pile. In addition, ground water elevations will be determined periodically. A total of five ground water samples will also be obtained for laboratory analysis. (See Section 6, "Sample Preservation Procedures and Field Laboratory Analyses".

SITE SETUP (EQUIPMENT INSTALLATION)

The installation of equipment and facilities will be accomplished only through the assistance of the specific utility at which the program

is located. There are four categories of equipment which have to be installed in order to monitor the coal pile for runoff. Each of these is detailed below. But prior to any equipment setups, the project manager will meet with the utility plant manager and the foreman in charge of the coal pile to arrange for certain facilities. A description of these follows.

Utility Assistance

Once a coal pile has been selected for runoff monitoring, the following information should be collected and studied prior to meeting with the utility personnel.

1. Aerial photographs of the coal pile
2. Topographic map of the coal pile area
3. Location of the natural drainage patterns of the runoff water
4. Determination of whether the pile is lined, diked or ditched on the perimeter

At the meeting, the focus of discussion should center on constructing permanent facilities for monitoring flow. It would be to the advantage of the utility to install a flume or weir, depending on the topographic configuration of the area around the pile, not only for this program but to be used as a permanent primary device for monitoring runoff flow. A flume would be recommended for coal piles built in flat terrains. It should be permanently mounted in a concrete slab with the coal pile dike or ditch butting on the converging section of the flume. For coal piles built on hills or slopes the use of a weir with quiescent chamber is recommended. In either case each device should be mounted in a permanent, concrete structure.

Other items to be considered by the utility are the pouring of three concrete slabs adjacent to the flow device as depicted in Figure 23. These will be used for monitoring an instrument shelter and meteorological station. Assistance will also be required for bringing in AC power to both the runoff monitoring station and a mobile field laboratory. Fresh water will have to be connected to the field laboratory but this can easily be done by garden hose hookups.

A summary of utility assistance items necessary prior to installation of a runoff monitoring station are listed in Table 47.

Once an agreement has been made between the project team and the utility for the following items, the installation of equipment can begin.

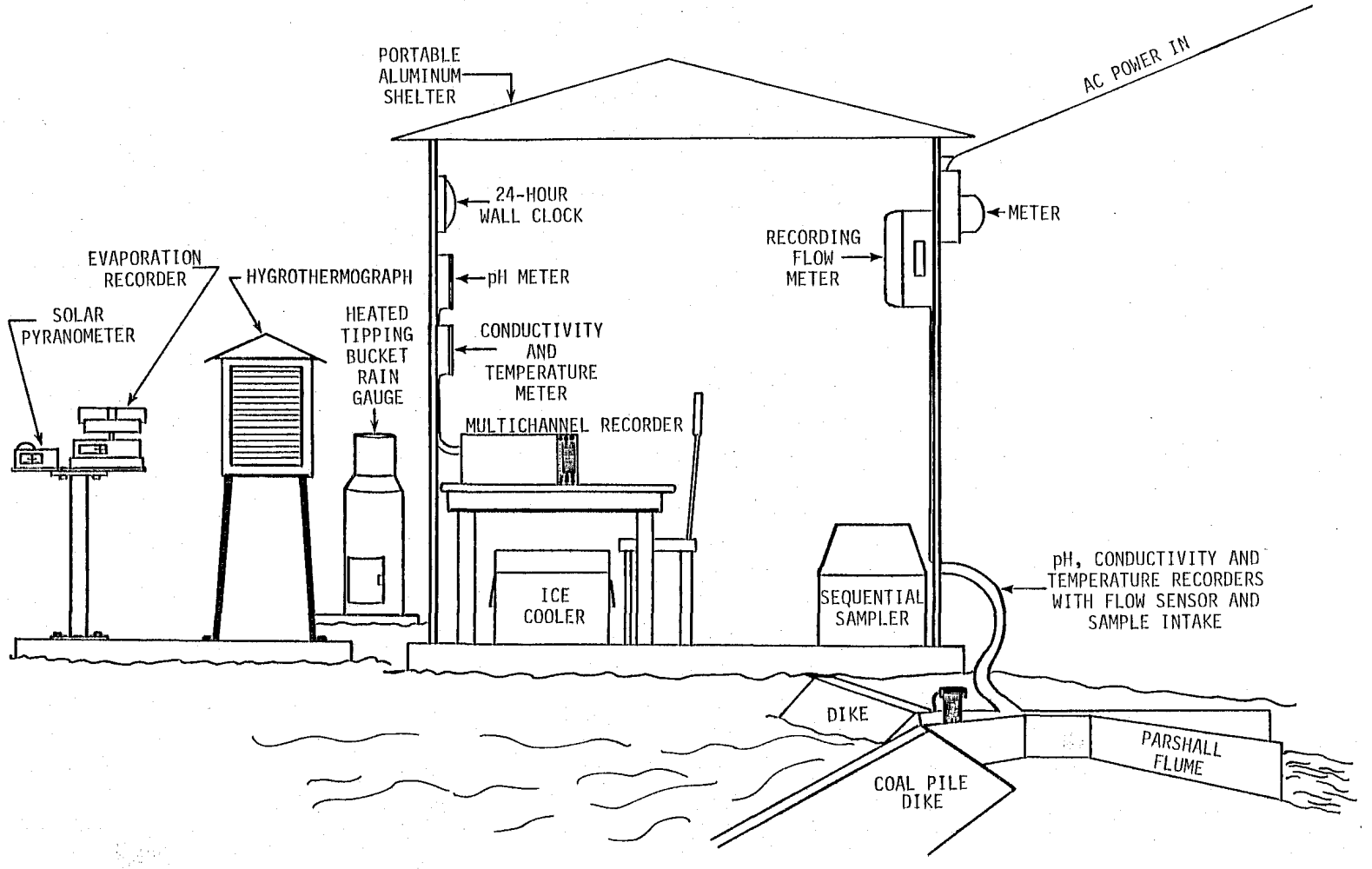


FIGURE 23: SCHEMATIC OF COAL PILE RUNOFF MONITORING STATION

TABLE 47. SUMMARY OF UTILITY ASSISTANCE ITEMS IN ORDER TO MONITOR COAL PILE RUNOFF

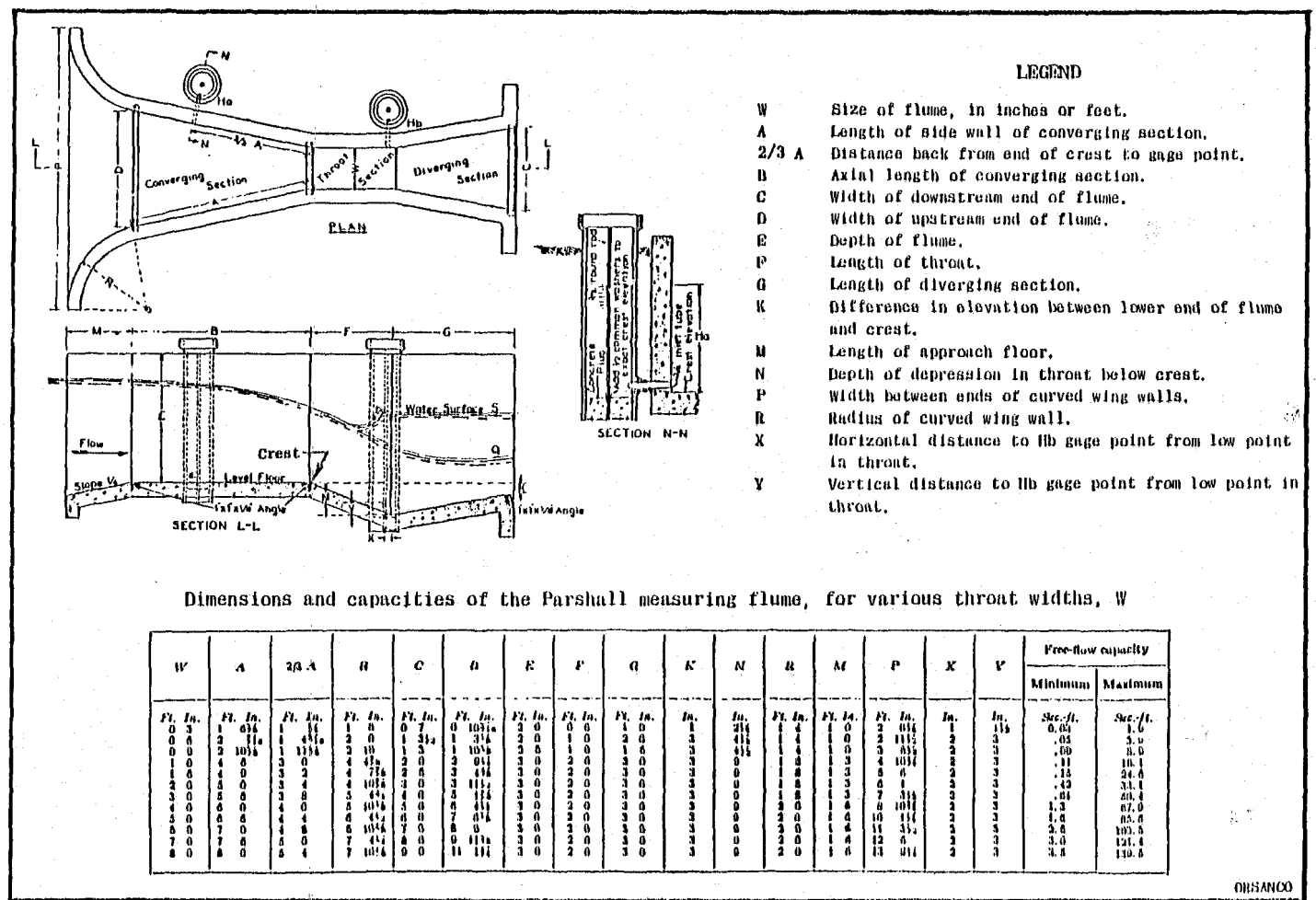
1. Dike or ditch the coal pile area.
2. Locate and construct a permanent flow monitoring station (flume or weir).
3. Pour concrete slabs for locating an instrument shelter and meteorological station.
4. Provide power to the instrument station and mobile field laboratory.
5. Provide water to the field laboratory.

Flow Monitoring Station

The coal pile runoff project team will assist the utility in the design of a primary flow device. Important design criteria for the flow device would include:

1. Design flow - calculated from the Rational Formula using 1.5 times the flow from the 10 year 24 hour storm
2. Materials - the highly acidic character of the runoff stream warrants the use of non-corrosive materials such as fiberglass, acid resistant concrete, and high grade stainless steel such as Carpenter 20.

For the runoff monitoring program accurate measurements of flow are mandatory. Because both wet weather flows and dry weather base flows are to be measured, a combination of measuring devices is recommended. A large flume or weir built for a design storm would be installed permanently to measure the wet weather conditions. For dry weather base flows a small "V" notch 90° weir built downstream of the flume would be installed because of its inherent accuracy at low flows. This weir would be a temporary device to be used only for the monitoring program. A detachable weir plate would be made so that this weir would not restrict flows in times of wet weather. Also the flow meter to be used on the flume, through a quick change of sensors and internal programming cards, can be used for both flow devices. An illustration of the flume with various dimensions and capacities is shown in Figure 24. An illustration of the combined flume and "V" notch weir is shown in Figure 25.



Dimensions and capacities of the Parshall measuring flume, for various throat widths, W

W	A	2/3 A	B	C	D	E	F	G	K	N	R	M	P	X	Y	Free-flow capacity	
																Minimum	Maximum
Ft. In.	Ft. In.	Ft. In.	Ft. In.	Ft. In.	Ft. In.	Ft. In.	Ft. In.	Ft. In.	In.	In.	Ft. In.	Ft. In.	Ft. In.	In.	In.	Sec.-ft.	Sec.-ft.
0 3	1 6	1	1	0 7	0 10	1 0	0 6	1 0	1		1 4	1 0	2	1		0.03	1.6
0 6	2	1	1	1	1	1	0	1	3		1	1	3	2		.05	3.9
0 9	2 6	1 8	1 6	1 5	1 5	1 0	1 0	1 5	3		1 4	1 0	3	2		.09	8.9
1 0	3 0	2 0	2 0	1 10	1 10	1 0	1 0	2 0	3		1 8	1 3	4	2		.11	11.1
1 6	4 0	2 8	2 6	2 3	2 3	1 0	1 0	3 0	3		1 8	1 3	5	2		.15	16.6
2 0	5 0	3 4	3 0	2 8	2 8	1 0	1 0	4 0	3		1 8	1 3	6	2		.21	22.2
3 0	7 6	5 0	4 6	4 0	4 0	1 6	1 6	6 0	3		1 8	1 3	8	3		.31	33.1
4 0	10 0	6 8	6 0	5 4	5 4	2 0	2 0	8 0	3		2 0	1 4	10	3		.42	44.2
5 0	12 6	8 4	7 6	6 8	6 8	2 6	2 6	10 0	3		2 0	1 4	12	3		.54	55.3
6 0	15 0	10 0	9 0	8 0	8 0	3 0	3 0	12 0	3		2 0	1 4	14	3		.65	66.4
7 0	17 6	11 8	10 6	9 4	9 4	3 6	3 6	14 0	3		2 0	1 4	16	3		.77	77.5
8 0	20 0	13 4	12 0	10 8	10 8	4 0	4 0	16 0	3		2 0	1 4	18	3		.89	88.6
10 0	25 0	16 8	15 0	13 4	13 4	5 0	5 0	20 0	4		2 8	1 6	22	4		1.14	111.0
12 0	30 0	20 0	18 0	16 0	16 0	6 0	6 0	24 0	4		2 8	1 6	26	4		1.40	133.5
15 0	37 6	25 0	22 6	20 0	20 0	7 6	7 6	30 0	4		2 8	1 6	32	4		1.80	166.9
20 0	50 0	33 4	30 0	26 8	26 8	10 0	10 0	40 0	4		2 8	1 6	42	4		2.60	233.8
25 0	62 6	41 8	37 6	33 4	33 4	12 6	12 6	50 0	4		2 8	1 6	52	4		3.40	300.7
30 0	75 0	50 0	45 0	40 0	40 0	15 0	15 0	60 0	4		2 8	1 6	62	4		4.20	367.6
40 0	100 0	66 8	60 0	53 4	53 4	20 0	20 0	80 0	4		2 8	1 6	82	4		5.80	501.4
50 0	125 0	83 4	75 0	66 8	66 8	25 0	25 0	100 0	4		2 8	1 6	102	4		7.40	635.2

ORSANCO

FIGURE 24: GENERAL SCHEMATIC OF A PARSHALL FLUME[16]
(Reprinted, with permission, from "Planning and Making Industrial Waste Surveys," page 15. Copyright, Ohio River Valley Water Sanitation Commission, 414 Walnut Street, Suite 900, Cincinnati, OH 45202 -- 1952.)

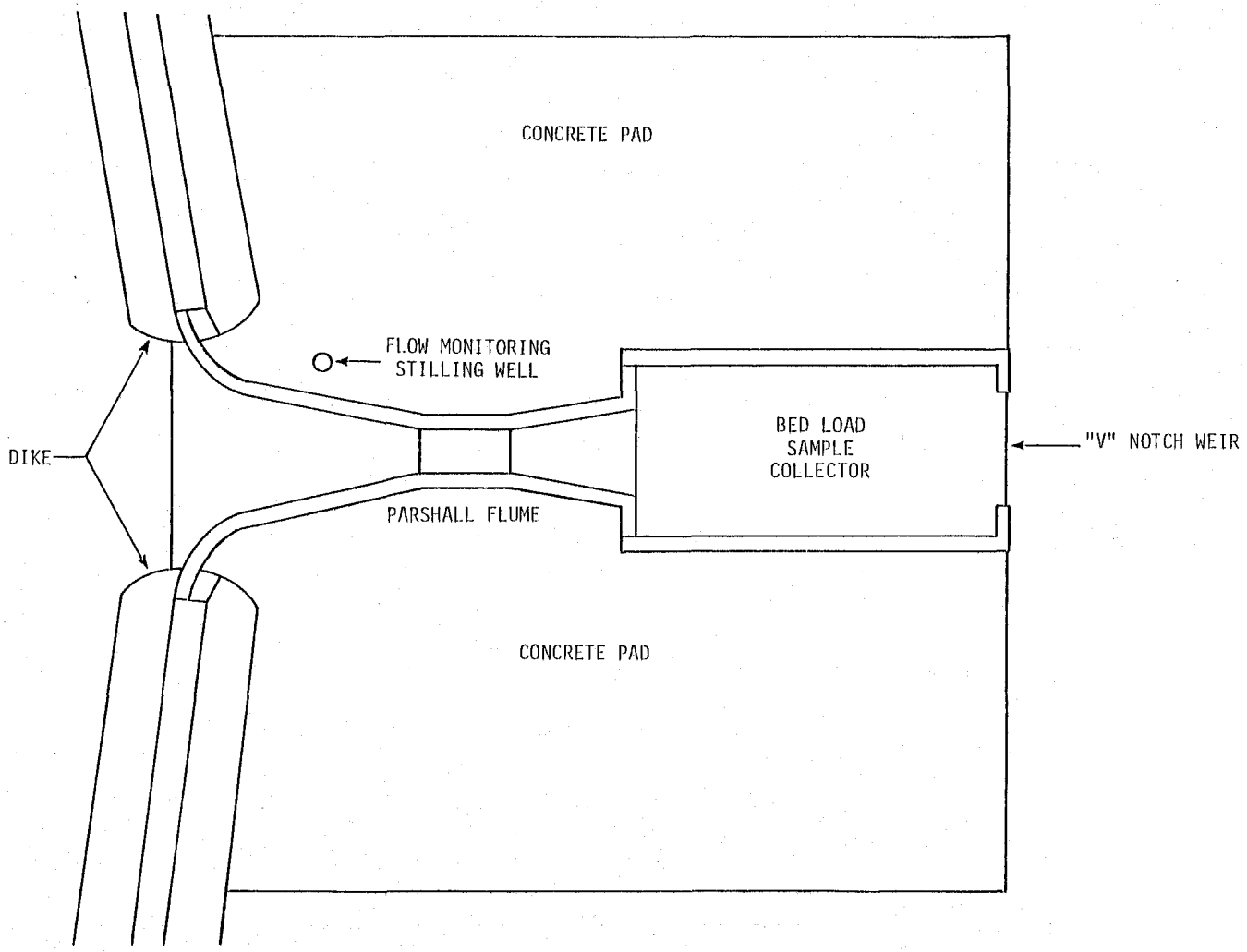


FIGURE 25: SCHEMATIC OF FLUME AND "V" NOTCH WEIR FLOW STATION

Installation of the flow recording device would be performed after the primary devices have been installed. A bubbler type flow meter would be used because of simplicity of installation and maintenance. The meter itself is housed in the instrument shelter and powered by AC. Two bubble tubes would be used: one on the flume and one mounted 4 times the "V" notch height behind the weir plate. Confirmatory flow readings will be taken from staff gages mounted on the wall of the converging section of the flume and on the approach to the weir plate. These staff gages can also be used to calibrate the flow meter by referring to stage versus discharge tables for each device. All bubbler tubing will be routed by electric conduct between the instrument shelter and primary devices. The bubble flow meter can be wall mounted and should have a spare parts box located nearby. The spares box will contain desiccant replacement, spare flow programming cards, spare recorder paper and spare ink cartridges. The installation of the flow meter system can be checked by having a water line, i.e. fire hose, discharge through the flume and by calibrating the meter accordingly.

Runoff Monitoring Station

Besides the flow system, the runoff monitoring station will include continuous recorders for pH, conductivity and temperature together with the automatic sequential wastewater sampler. The wastewater sampler is also housed within the instrument shelter. A pump inlet velocity of at least 1400 milliliters per minute is specified in order that most of the susupended solids are captured. The automatic sampler is programmable and all dial settings will be set depending on the type of samples to be collected. The most critical parameter in the installation of the sampler is the position of the intake tubing. The inlet tubing should be mounted upstream of the neck of the flume and in a position not to be susceptible to runoff bedload effects. This may conflict with the ability to sample the initial flows of a runoff stream during a storm event. An adjustable arm for holding the inlet tubing should therefore be installed so that the sampler may collect samples from any vertical position upstream of the flume. Spare parts to be kept with the sampler include spare pump tubing, desiccant, small hose clamps and extra sample collection bottles.

Conductivity, pH and temperature sensors will be mounted on the flume or within a stilling well on the flume for the continuous recording of all parameters. Conductivity and temperature will be recorded from the same probe and meter but the separate analog signals will be recorded individually. pH will have its own meter and channel on the recorder. Sensor cables from each of these probes will be routed though electrical conduit between the instrument shelter and the flow channel. Analog meters will be used to have a visual indication of the data coming from each probe. The pH, conductivity and temperature meters can be either wall mounted or installed in a rack also holding the multichannel recorder. The wall mounted setup is shown in Figure 23. Care must be taken when submerging the probes in the runoff stream due to the

high acidity of the water. Experience has shown that iron oxide can quickly foul the pH and conductivity probes. Therefore daily observations of probe fouling and deterioration must be made by the Technical Specialist. Also all probes should be either plastic or teflon coated to avoid corrosion. Spare items to be kept with the continuous metering devices will include:

1. Spare probes for all sensors
2. Desiccant
3. Calibration buffers for pH and solutions for conductivity
4. ASTM thermometer
5. Spare chart paper, ink and pens for the multi channel recorder

Meteorological Station

The meteorological station will consist of four instruments, three of which are battery powered and one which is AC powered. The station consists of:

1. Solar pyranometer
2. Evaporation recorder
3. Hygrothermograph
4. Heated typing bucket rain gage (AC powered)

All four instruments are to be mounted on level surfaces and their individual locations should be sited according to the following criteria.

1. Solar pyranometer - in an open space away from any obstructions that may block the sun's rays. Also it should be kept away from heavy traffic areas due to the sensitivity of the recorder pen to ground vibrations.

2. Evaporation recorder - in a non-shaded area away from heavy traffic areas.

3. Hygrothermograph - on a level surface inside a louvered shelter which will allow free passage of air. Ground vibrations can also affect its recorder sensitivity.

4. Heated Tipping Bucket-Rain Gage - absolutely away from all ground vibrations, on a level platform and close to a power source. This instrument will record snowfall when the heater has been turned on and for all other cases will record precipi-

Heating Tipping Bucket (Cont) - tation. This gage should be at least four times the height of the closest obstruction away from that obstruction so that wind effects are minimal.

Figure 23 depicts the meteorological station adjacent to the runoff monitoring site but as is the case around most utility coal piles, this is in an area of intense heavy traffic movement. Therefore the meteorological station should be positioned out-of-the way in a field or vacant lot within the confines of the power plant. Roof installations should be avoided so that the field technician will not have to walk too far in order to check on the equipment's operation. Spares for the meteorological equipment can be kept in the instrument shelter and consist of charts, pens, batteries, ink, desiccant, etc.

Mobile Field Laboratory

Prior to sampling any runoff, a field laboratory must be sited and set-up in order to handle the samples once they have been collected at the monitoring station. A 20 x 8 foot mobile laboratory is of sufficient size to handle the storage, sample preparation and analysis of 4 to 5 parameters. Assistance from the utility will be necessary in the form of a power source and freshwater inlet. If at all possible, the sink drain should be directed into a holding tank.

The general layout for the laboratory is shown in Figure 26. It includes specified bench areas for the following tasks:

1. Storage and logging of incoming samples
2. Sample labeling and preservation with acids
3. Filtering for metals and solids
4. Titration for acidity and alkalinity
5. Cleaning and washing area
6. Ice making and refrigerated sample storage

A checklist of items to be included in the field laboratory is found in Table 48. Many of the items are expendable so provisions should be made to have plenty on hand. Once the laboratory has been moved to its field location, it should be set-up on blocks and leveled. The scale will have its own leveling screws but the trailer should be as level as possible before use. A heater/air conditioner along with plenty of storage cabinets is recommended for comfort and ease of equipment manipulation.

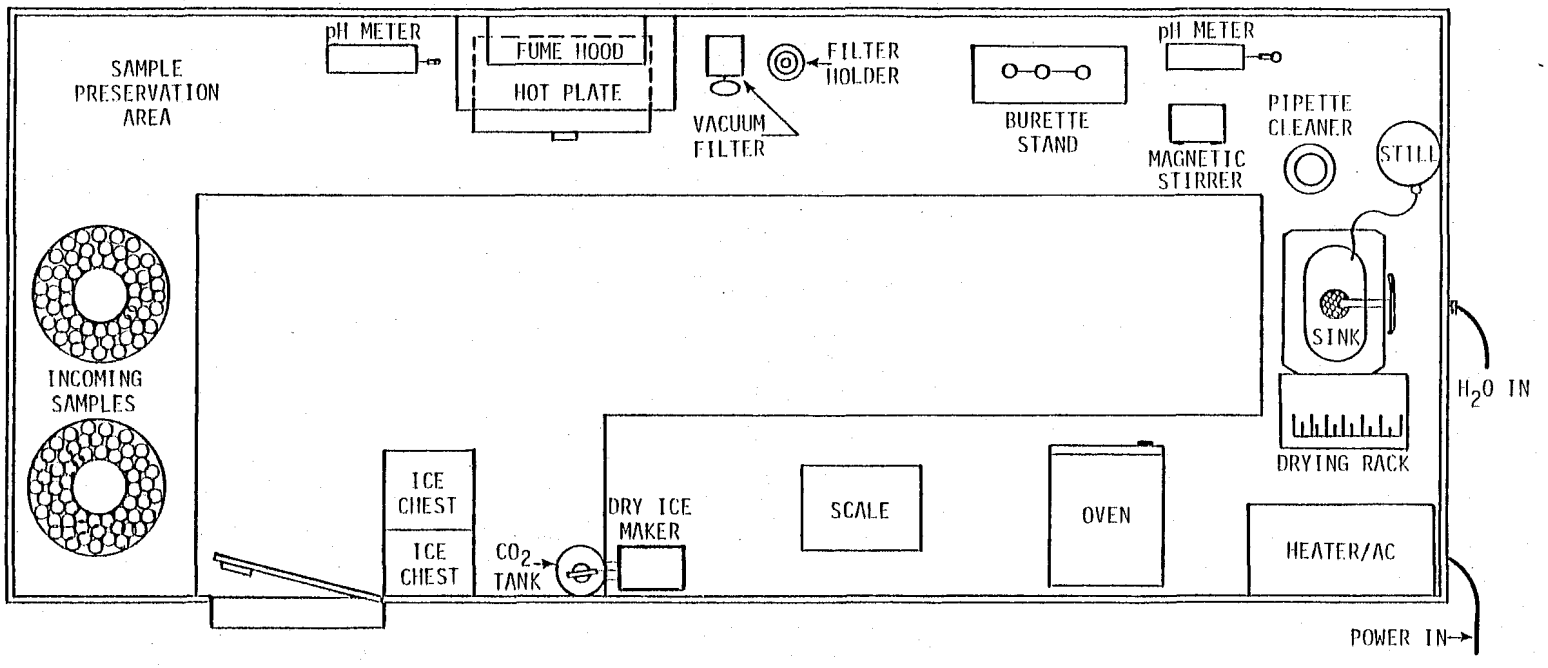


FIGURE 26: SCHEMATIC FOR A 20' x 8' MOBILE FIELD LABORATORY FOR ANALYSIS OF COAL PILE RUNOFF WATER

TABLE 48. CHECKLIST FOR MOBILE FIELD LABORATORY

1. Heater/AC 3.	16. 200 and 500 ml beakers
2. Fume hood/hotplate	17. Assorted flasks
3 Vacuum Filter	18. Squeeze bottles
4. Millipore filter holder	19. Pipettes, squeeze bulb, tweezers
5. Filters	
6. Burette stand and burettes	20. Concentrated nitric and sulfuric acids
7. Two pH meters (portable)	
8. Magnetic Stirrer	21. Paper towel, kemwipes
9. Drying oven	22. pH paper, eyedroppers, rubber gloves
10. Scale	
11. Pipette cleaner	23. Lab markers, labels
12. Drying rack for glassware	24. Insulated shipping containers
13. Water still or demineralizer (Type II Reagent Grade Water)	25. 250 ml polyethylene sample bottles
14. CO_2 Tank/Dry Ice Maker	26. Log book, analytical reference manuals
15. Graduated cylinder	

DAILY EQUIPMENT STATUS CHECKS

Calibrations

Calibration of the equipment used at the runoff monitoring station is essential to a field program where reliable data is to be generated. By either adjusting an instrument to a calibration curve or adjusting the raw data from the instrument to a calibration curve, the reduced field data will be more accurate. Procedures for calibrating a particular instrument are normally found in the instruction manual accompanying the instrument. The frequency of calibrations is usually not stated in the instrument manual and is established according to the frequency of use of the equipment. Table 49 presents a frequency of calibration and field calibration checks for each piece of equipment illustrated in Figure 23.

Field calibration checks are a system of procedures to quickly check the zero, span and overall operation of each piece of equipment while it is being used in the field. These checks will let the Technical Specialist know when a certain instrument has drifted out of calibration or if maintenance is required. For the solar pyranometer, evaporation recorder and tipping bucket rain gage quick checks can be made for zero and span. The hygrothermograph can be calibrated in the field against an ASTM thermometer and wet/dry bulb sling psychrometer. Daily checks should be made by the Technical Specialist on the recording flow meter and pH meter because these instrument sensing units may be grossly affected by the low pH and iron deposits in the runoff stream.

Conductivity and temperature meters can be checked on a weekly basis. All calibration and field check data is to be kept in a separate log book which is maintained by the Technical Specialist onsite.

TABLE 49. FREQUENCY OF CALIBRATIONS AND FIELD CALIBRATION CHECKS

	Equipment	Full Calibration	Field Check
1.	Solar Pyranometer	once every three months	once every two weeks
2.	Evaporation Recorder	"	"
3.	Hygrothermograph	"	"
4.	Heated Typing Bucket	"	"
5.	Sequential Sampler	"	once per month
6.	Recording Flow Meter	pre and post field monitoring	daily
7.	pH Recorder	pre and post field monitoring	daily
8.	Conductivity/Temperature Recorder	once per month	weekly

Status Checks and Equipment Maintenance

The purpose of equipment status checks is to insure that all monitoring equipment, especially the automated devices, is in a state of readiness so that once a storm event happens, the samples are collected and continuous data is recorded without interruption. Figure 27 provides a sample check list to be used by the onsite Technical Specialist on his daily rounds to each runoff station. All checklists will be kept on site in a separate log book. From this log the history of each piece of equipment while in the field can be traced.

The only time that a daily site check is omitted is during a runoff sampling event. In this case other monitoring site documentation will be used. Once a storm event has ceased and all the equipment is put back into the dry weather mode, the Technical Specialist will continue with his status checks.

DRY WEATHER SAMPLING

Coal pile runoff samples will be collected once per day at each runoff point and composited to form one sample. The use of automatic sampling equipment is not necessary during dry weather sampling. Grab sampling techniques will be used on dry days.

Figure 27: Daily Equipment Status Check List

Project No. _____ Runoff Station ____
Site _____ Date _____

Instruments	S/N	Record OK	Chart Replaced	Ink Replaced	Battery Replaced	Pen Replaced	Instr. Replaced	Probes Cleaned	Probes Replaced	Zero Check	Span Check	Calibration Check	Instr. In Error	Required Maintenance
Solar Pyronometer														
Evap. Recorder														
Hygrothermograph														
Tiping Bucket														
pH Meter														
Cond./Temp. Meter														
Multi-Channel Recorder														

Flow System S/N ___

1. Record OK ___
2. "V" Notch In Place ___
3. Bubbler Rate ___
4. Calibration Adjustment ___
5. Expendables Replaced ___
6. Clock Update ___
7. Maintenance Reg'd ___
8. Bubble Line Cleaned Out ___

Automatic Sampler S/N ___

1. Actuator Operational ___
2. System on Standby ___
3. Expendables Replaced ___
4. Intake Cleaned ___
5. Clock Update ___
6. First Bottle Position ___

Flows of dry weather runoff resulting from seepage from the base of the pile will be continuously measured by using the "V" notch weir plate and the recording flow meter. Samples are to be collected from the same point and at the same time of day for each dry weather day. The discharge from the weir plate is the recommended grab sampling point. Sampling should occur once the equipment status checks have been completed.

The routine procedures to be followed on a dry weather day are itemized as follows:

1. Install "V" notch weir plate, reposition flow bubble line at weir and readjust the flow meter for size and capacity of weir. (This should be completed immediately after each storm when the flow approaches base flow.)

2. Perform daily equipment status checks (refer to the previous Section).

3. Using clean one liter glass bottles, collect a sample from the discharge from each runoff station weir remembering to keep the time of sampling consistent.

4. Place an event mark on the flow record and the pH, conductivity and temperature records indicating that a dry weather sample was taken.

5. Return the samples to the field laboratory and pour each 1 liter sample into a large beaker capable of containing all runoff samples.

6. Stir the collected sample and split the composite into individual sample containers.

7. Perform the field analyses and preservation on the composite samples.

8. Log in the dry weather sample and record the results of the field analysis.

9. Clean the 1 liter collection bottles, compositing beaker and associated glassware used in the analyses and preservations.

10. Check on the weather forecast and make the necessary preparations if a precipitation event is likely.

WET WEATHER SAMPLING

Anticipation of a Storm Event

The Technical Specialist will have the responsibility to monitor weather forecasts in the area of the site so that basic preparations necessary before sampling can be accomplished. There are various ways to keep an eye on the weather, e.g. TV reports, radio, etc., but one of the best methods is to arrange a contact with the nearest National Weather Service that maintains a weather radar. The closest National Weather Service station is most likely at the closest national airport. Weather radar may or may not be used but it is the best method to have confirmatory information on the approach of precipitation. Once a storm event has been predicted, the Technical Specialist must perform the following at each runoff station:

1. Make a final check on the meteorological instruments to insure there is enough chart paper to last at least through the precipitation period. Changing charts in the middle of a storm can lead to wet charts and blurred data.

2. Remove the "V" notch weir plate and clean out the bed load sediment trap between the flume and weir.

3. Check on the pH, conductivity and temperature probes to see that they are responding and operational.

4. Check the sequential sampler's intake strainer for debris.

5. Check that the sample activator switch is in its correct position. This device will only be used when a storm event occurs at night or at times when the site technician is not available to turn on the sampler.

6. Check that the flow meter bubble line is in its proper position in the flume.

7. Check the multichannel recorder to see that there is enough chart paper.

8. Turn all runoff monitoring instruments to "ON".

9. Call the Technical Assistant and alert him of the approaching storm. The assistant should make plans to travel to the site once the runoff event has been confirmed.

10. Make ready a second set of sample collection bottles in case the event lasts long enough to fill the first set of bottles.

The Technical Specialist should be ready for the storm event no matter what time of the day it starts. If the storm is to break during

the night, the technician should put the equipment on automatic so that sampling for the initial storm runoff can be accomplished. If the storm breaks during the day, the technicians should manually start the sampler once the flow has risen above base flow.

Event Sampling

The Technical Specialist must be able to differentiate a runoff event from a non-runoff storm event. There will be many cases when the precipitation will be of such light intensity that either no runoff will be observed or there will be a lag in the timing of the runoff. For the purposes of this program only those storms causing the runoff to increase above base flow will be considered storms. Table 50 lists the types of storms desired during the program and the frequency of sampling for each runoff stream. There may be cases when sampling may have commenced and after the samples have been collected it is determined that the runoff event duplicated the conditions of a previous event. If this situation arises the Technical Specialist should confer with the project manager to determine whether the samples should be prepared for analyses or thrown out. The final decision can be made only by the project manager.

TABLE 50. STORM TYPES TO BE SAMPLED WITH CORRESPONDING SAMPLE FREQUENCY

Storm	Frequency
24 hour*	1/2 hour for the first four hours, 1 hour after 4 hours
12 hour	1/2 hour for the first four hours, 1 hour after 4 hours
4 hour	1/2 hour for runoff duration
2 hour	1/2 hour for runoff duration

*24 hour refers to the length of time the runoff hydrograph is above base flow.

Procedures to be followed by the Technical Specialist during a runoff event are listed below:

1. Monitor the runoff stream and record visual observations of the character of the runoff, e.g. color, intensity, flow peak, etc.

2. Monitor all the automatic recorders and equipment to see that they are recording properly.

3. When the initial samples (first four hours) have been collected, switch the automatic sampler to a 1 per hour rate.

4. Call the Technical Assistant if the amount of laboratory work anticipated requires his presence. For most storms where sampling occurs through the night, the assistant will be required.

5. Place event marks on all records and charts indicating the start of sampling, storm number, data and time.

6. Once the runoff has dropped back to base flow make an estimate of the volume of bed load sediment trapped between the flume and weir and collect a representative sample for moisture content determinations.

7. After the runoff has ceased, make the necessary adjustments for dry weather monitoring.

8. Once a set of samples has been collected, carry them to the field laboratory and perform the preservations and analysis.

9. Collect a sample from the rain gage for pH and acidity analysis. Clean out the rain collector once the sample has been collected.

10. Clean off all probes immersed in the runoff stream, all sample intakes and bubblers from the flow meter. Desiccant charges, on instruments having them, should be checked also.

SAMPLE IDENTIFICATION AND CUSTODY PROCEDURES

In order to avoid confusion in the handling of water samples, a system for numbering the sampling stations and identifying samples is crucial. In addition, custody procedures are necessary to track the samples from the time of collection through laboratory analysis. Sample shipping procedures are also an important part of the field program. Each of these areas will be discussed in this section.

Number of Sampling Stations and Sample Identification

All sampling stations will be identified with three-digit numerical codes. For example, if there are two sampling stations for a particular program, these would be identified as site #001 and #002 respectively.

Each sample shipping bottle will be pre-labeled with sticky back waterproof labels. The following example shows the format of the labels and the information to be pre-printed on each:

Project No.	1234-A12
Date	_____
Sample No.	_____
Analysis	TOC
Preservation	Cool, $4^{o}C$; H_2SO_4 to pH 2
Sample Type	_____

In the field, the only information that has to be entered on the labels is the date, sample number, and sample type (i.e., surface water, ground water, precipitation, or coal pile water).

Sampling Custody Procedures

Sample custody procedures will be established to ensure that the samples are not lost prior to chemical analysis. These procedures will consist of a number of data sheets which will be used to track the samples. In the field, each sample will be entered into a sample log sheet prior to being packed for shipping to the chemical laboratory. Figure 28 is an example of the log sheet showing the information to be entered. A copy of this sheet will be included with the samples being shipped.

When the samples arrive at the chemical laboratory, a laboratory check-in sheet will be completed prior to storing the samples in the cold storage room. This sheet will be kept outside of the storage room. Figure 29 is an example of this sheet. At the same time, a Request for Water Analysis sheet, as shown in Figure 30, will be completed and submitted to the laboratory director.

When samples are to be taken from the cold storage room for analysis, a laboratory checkout sheet will be completed. As with the check-in sheet, this sheet will be kept outside of the cold storage room. Figure 31 is an example of this sheet.

After the chemical analyses have been performed, a report of water analysis sheet, as shown in Figure 32, will be filled out and sent to the project manager. In addition, all chemical analyses performed in the field will be entered on one of these sheets with a copy mailed to the project manager.

The project manager and laboratory director will each retain a copy of all data sheets and will be responsible for setting up individual data files.

Sample Shipping Procedures

A courier will be chosen to deliver the sample shipping boxes to the chemical laboratory. The location of the utility site will determine

Figure 28: Sample Log Sheet

Sample No.	Sampling Site	Date of Collection	Time of Collection	By	Time Preserved	Preservation Method	Reserved for Analysis of	Volume of Sample	Date Shipped to Chem. Lab	Shipping Box No.

Location _____
Project. No. _____
Supervisor _____

FIGURE 29: INITIAL SIGN IN SHEET

All samples must be logged in when they are initially stored in the cold room.

PROJECT NO.	SAMPLE ID	DATE SAMPLE COLLECTED	DATE/TIME ENTERED COLD ROOM	INITIALS

FIGURE 30: REQUEST FOR WATER ANALYSIS

CLIENT:_____ LABORATORY NO:_____

CONTRACT NO.:_____ NUMBER OF SAMPLES:_____

SENT BY:_____ DATE RECEIVED:_____

REPORT TO:_____BY_____ DATE COMPLETED:_____

ESTIMATED TIME FOR ANALYSIS:_____

SURFACE_____WELLWATER_____CITY WATER_____WASTE WATER_____SEA WATER_____

	1	2	3	4	5	6	7	8	9
Demand Analysis:									
BOD_5									
TOC									
COD									
Residue Analysis:									
Total Solids									
Volatile Solids									
Fixed Solids									
Total Suspended Solids									
Total Dissolved Solids									
Nitrogen Constituents:									
Total Kjeldahl Nitrogen									
Ammonia									
Organic Nitrogen									
Nitrate									
Nitrite									
Phosphates:									
Total									
ortho									
Condensed									
Physical Analysis:									
Color									
Odor									
Turbidity									
pH									
Oil and Grease									
Sulfate									
Cyanide									
Phenol									
Fluoride									
Chloride									
Alkalinity									
Acidity									
Hardness									
Detergent (MBAS)									
Specific Conductance									
Metals (Specify)									
Others (Specify)									

Notes: Split Sample Yes_____No_____

Form CL-003

FIGURE 31: SIGN OUT SHEET

All samples taken from the cold room must be signed out and also signed in when they are returned.

PROJECT NO.	SAMPLE ID	REASON FOR REMOVAL	DATE REMOVED	INITIALS	DATE RETURNED	INITIALS

FIGURE 32: REPORT OF WATER ANALYSIS

Client: ____________________ Laboratory No: ____________________

Contract No: ____________________ Date Received: ____________________

Reviewed by: ____________________ Date Reported: ____________________

Reported to: ____________________ Type(s) of Sample(s): ____________________

	mg/L as	1	2	3	4	5	6	7	8

Notes:	ANALYZED:
	CHECKED:

Form CL-004

whether air or land transportation is necessary. The main objective is to transport the samples to the chemical laboratory in as short a time as possible. This will ensure that the integrity of the samples is maintained prior to analyses.

The courier should be contacted as soon as the field crew has an idea when the samples will be preserved and packed for shipping. A location at the utility site will be designated as the pickup point. At the time of pickup, the field crew will give the courier a packing list indicating the number of shipping boxes and the contents of each. The courier will then specify the waybill number (i.e., shipping number) and all other pertinent shipping information, including the name of the airlines or trucking company, the flight number, and the delivery date and time. The field crew will convey this information to the person at the laboratory receiving the shipment.

The shipping boxes should be made of metal to avoid serious damage during shipping. In addition, the boxes should be insulated with some type of foam or other packing material as added protection and also to keep the samples cool. Dry ice will be added to the boxes to preserve the integrity of the samples. The shipping address should be clearly visible on the boxes.

SAMPLE PRESERVATION PROCEDURES AND FIELD LABORATORY ANALYSES

This section discusses the procedures to follow when compositing, filtering and preserving samples. In addition, the laboratory analyses to be performed in the field are also discussed.

Sample Preservation

Table 51 indicates the parameters to be analyzed in the stormwater runoff and base flow samples, the volume required for each analysis, the proper preservation method, and the maximum holding time for each. A minimum of 1275 ml of sample will be required for each sample. This will necessitate that three sequential sampler bottles be filled during the storm event (each bottle holds approximately 460 ml of sample) which will be composited to produce one sample for analysis. During dry day events the individual grab samples from each base flow stream will be composited to again produce one sample.

Compositing Samples

Equipment Required: 1500 ml beakers
Magnetic stirrer
Stirring bars

TABLE 51. SAMPLE VOLUMES, PARAMETERS TO BE ANALYZED, PRESERVATION METHODS, AND MAXIMUM HOLDING TIMES[1]

Parameter	Sample Volume, ml	Preservation Method	Maximum Holding Time[2]
Acidity/Alkalinity	100	Cool, 4°C	24 hrs.
Total Suspended Solids (TSS)	100	Cool, 4°C	7 days
Total Dissolved Solids (TDS)	100	Cool, 4°C	7 days
Total Organic Carbon (TOC)	25	Cool, 4°C, H_2SO_4 to pH [2]	24 hrs.
Sulfate (SO_4)	50	Cool, 4°C	7 days
Total Hardness	100	Cool, 4°C, HNO_3 to pH [2]	6 mos.
Metals:	300[3]	Total: HNO_3 to pH [2]	6 mos.
		Dissolved: Filter on site; HNO_3 to pH [2]	6 mos.
Aluminum (Al)			
Antimony (Sb)			
Arsenic (As)			
Beryllium (Be)			
Cadmium (Cd)			
Calcium (Ca)			
Chromium, Total (CrT)			
Cobalt (Co)			
Copper (Cu)			
Iron (Fe)			
Lead (Pb)			
Magnesium (Mg)			
Manganese (Mn)			
Molyldenum (Mo)			
Nickel (Ni)			
Selenum (Se)			
Vanadium (V)			
Zinc (Zn)			
Fluoride (F)	300	None Req'd	7 days
Mercury (Hg)	200[4]	Total: HNO_3 to pH [2]	13 days
		Dissolved: Filter on site; HNO_3 to pH [2]	13 days
	Total = 1275 ml		

[1] Sample volumes, preservation methods, and holding times taken from Methods for Chemical Anlysis of Water and Wastes, EPA-600/4-79-020, March 1979.

[2] EPA has proposed extending these holding times (Federal Register, Tuesday, December 18, 1979, Vol. 44, No. 244, pp. 75050-52).

[3] 200 ml for dissolved metals; 100 ml for total metals.

[4] 100 ml for dissolved mercury; 100 ml for total mercury.

Procedure: Pour the contents of the three automatic bottles (storm events) or the grab sample bottles (dry weather events) corresponding to one sample into the beaker. Place the beaker on the magnetic stirrer. Stir rapidly about one minute to obtain a homogeneous mixture.

Filtration Method For Dissolved Metals and Dissolved Mercury

The samples for dissolved metals and dissolved mercury must be filtered on site prior to shipping. Mercury is distinguished from the other metals because of the difference in maximum holding times. The samples should be filtered as soon as possible after collection.

Equipment Required:
2 Suction Pumps
2-47 mm Millipore Filtration Funnels
2 Filtration Flasks
0.45 Millipore Filters
500 ml Beakers
100 ml Graduated Cylinders
Magnetic Stirrer
Nitric Acid (HNO_3)
Stirring Bars
pH Meter and Probe
Buffer Solutions

Procedure:

1. Filter a small portion of the composite sample to rinse the filter flask and discard.

2. Filter 300 ml of the composite sample. The filter may become clogged if a large amount of particulate is present. If filtration slows, change the filter and continue.

3. Pour the filtrate into the 500 ml beaker and place on the magnetic stirrer. Immerse the pH probe. While stirring, add nitric acid (HNO_3) until the pH is less than 2.

4. Place the sample in a 500 ml sample bottle.

Preserving Samples

The preservation procedures for the various parameters are discussed below. Before pouring off each split, insure the suspension of all particulate matter by stirring the composite sample on the magnetic stirrer. Table 52 shows which parameters can be combined to reduce the

number of sample bottles. Note that although the preservation procedures for SO_4 and fluoride are different (Cool, 4°C and none required respectively), they will be combined in one bottle because the shipping box will be cooled with dry ice.

TABLE 52. SAMPLE STORAGE BOTTLES AND CORRESPONDING PARAMETERS

Parameters	Sample Bottle Required(*)
Acidity/Alkalinity; TSS; TDS	500 ml
TOC	25 ml
F, SO_4	500 ml
Total Hardness; Total Metals, Total Hg	500 ml
Dissolved Metals; Dissolved Hg	500 ml

(*) All bottles are plastic.

Equipment Required:
- 500 ml Beakers
- 100 ml Beakers
- Magnetic Stirrer
- Stirring Bars
- Pipets
- Graduated Cylinder
- pH Meter and Probe
- Buffer Solutions
- 25 ml Plastic Bottles
- 500 ml Plastic Bottles
- Nitric Acid (HNO_3)
- Sulfuric Acid (H_2SO_4)

Procedure:

Acidity/Alkalinity TSS, and TDS: Pour 300 ml of the composite sample into a 500 ml sample bottle and keep the bottle cool.

Total Organic Carbon: Pour 25 ml of the composite sample into the 100 ml beaker and place on the magnetic stirrer. Immerse the pH probe. While stirring, add sulfuric acid (H_2SO_4) until a pH of less than 2 is obtained. Pour the sample into a 25 ml sample bottle and keep cool.

Sulfate and Fluoride: Pour 350 ml of the composite sample directly into a 500 ml sample bottle and keep cool. No preservative is needed.

Total Hardness, Total Metals, Total Mercury	Pour 300 ml of the composite sample into the 500 ml beaker and place on the magnetic stirrer. Immerse the pH probe. While stirring, add nitric acid (HNO_3) until the pH is lowered to less than 2. Place the preserved sample in a 500 ml sample bottle.

Field Laboratory Analyses

Acidity/alkalinity, total suspended solids (TSS), and total dissolved solids (TDS) will be analyzed in the mobile field laboratory. The analysis methods as specified in Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March, 1979, will be followed. These methods are discussed in detail in Appendix H. Acidity and alkalnity are determined titrimetrically. TSS and TDS analyses are gravimetric methods.

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APPENDIX A

COAL FIRED POWER PLANT QUESTIONNAIRE FOR ASSESSMENT OF COAL STOCKS AND TREATMENT DESIGN OF COAL PILE RUNOFF

COAL FIRED POWER PLANT QUESTIONNAIRE

for

Assessment of Coal Stocks and Treatment Design

of

COAL PILE RUNOFF

A-2

TRC Project: 1119-B80-00
Client: EPA-IERL-RTP and Edison Electric Institute

General Information for Each Plant

Plant Name_____ Utility_____ Person Responding to Questionnaire_____

City,State_____ Title_____

Type of Plant, Check One: Base Load_____ Peaking_____ Phone No._____

Total Plant Capacity _____MW % Usage Coal_____

Coal Pile Information

Is there segregation between live storage and reserve storage piles?_____ If not, explain_____

_____. Is the coal rotated within the pile?_____. What type of surface are the piles constructed on?_____(Clay, Compacted Coal, Compacted Ash, etc.)

For Reserve or Non Segregated Piles

PILE CHARACTERISTICS

Pile Id. No.	Volume (tons)	Average Pile Height (ft.)	Average Pile Width (ft.)	Average Pile Length (ft.)	Side Slope (ft.vert./ft.horiz.)	Storage Time at Plant (months)	Is the Pile Compacted
1							
2							
3							
4							
5							

COAL CHARACTERISTICS PER PILE

Pile Id. No.	Coal Rank	Coal Source Seam	Coal Source County, State	Avg. % Sulfur	Avg. BTU Content	Avg. % Moisture	% Pyritic Sulfur	Avg. % Ash	Is the Coal Cleaned
1									
2									
3									
4									
5									

Are there seasonal changes with reserve or nonsegregated pile sizes? Please explain

Additional Comments:

For Live Storage Piles

PILE CHARACTERISTICS

Pile Id. No.	Volume (tons)	Height (tons)	Width (ft.)	Length (ft.)	Side Slope (ft.vert./ft.horiz.)	Storage Time at Plant (months)	Is the Pile Compacted
1							
2							
3							
4							
5							

COAL CHARACTERISTICS PER PILE

Pile Id. No.	Coal Rank	Coal Source Seam	Coal Source County,State	Avg. % Sulfur	Avg. BTU Content	Avg. % Moisture	% Pyritic Sulfur	Avg. % Ash	Is the Coal Cleaned
1									
2									
3									
4									
5									

Are there seasonal changes with live pile sizes? Please explain_____

_____

_____

_____

Additional Comments:_____

_____

Coal Pile Runoff Treatment Design

1. Has a treatment or control system been designed for coal pile runoff at this plant?_____ If yes, please provide the following.
2. Is the runoff from all piles treated?_____ If not, which piles are treated?_____

   _____
3. At what time (month & year) did the treatment system go on line?_____
4. Provide the name and address of the person responsible for the treatment design:_____

   _____

5. List the design criteria used at this plant:

Design Storm Size_____ Source of This Design Storm Data_____
Tech. Paper 40, Local Data, Other

Design Runoff Flow From:_____
Rational Formula, On-Site Data, Other

If the rational formula was used, what runoff coefficient was selected?_____

Do you collect runoff from areas not specifically coal storage?_____ If yes, what type of areas?_____

_____

6. List the types of treatment or control used at this plant:

Are the coal piles diked?_____, How_____

Is the runoff directed through a flow measuring device?_____ What type device_____

Is the runoff treated for:

Flow Equalization_____ How_____

TSS Removal _____ How_____

pH Neutralization _____ How_____

Metals Removal _____ How_____

Other_____

_____

7. List the types of data normally collected on site for control, design or treatment of coal pile runoff.

Coal pile runoff water quality_____ Which parameters_____

8. Continuous or Periodic Monitoring of

Runoff pH _____ Precipitation _____

Runoff Temperature _____ Ground Water Quality _____

Runoff Conductivity _____ Ground Water Level _____

Runoff Flow _____

Runoff Other _____

APPENDIX B

STANDARD METHODS FOR COLLECTION OF A GROSS SAMPLE OF COAL

AMERICAN NATIONAL STANDARD ANSI/ASTM D 2234 – 76

Standard Methods for COLLECTION OF A GROSS SAMPLE OF COAL[1]

This Standard is issued under the fixed designation D 2234; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

INTRODUCTION

Data obtained from coal samples are used in establishing price, controlling mine and cleaning plant operations, allocating production costs, and determining plant or component efficiency. The task of obtaining a sample of reasonable weight to represent an entire lot presents a number of problems and emphasizes the necessity for using standard sampling procedures.

Coal is one of the most difficult of materials to sample, varying in composition from noncombustible particles to those which can be burned completely, with all gradations in between. The task is further complicated by the use to be made of the analytical results, the sampling equipment available, the quantity to be represented by the sample and the degree of precision required.

These standard methods give the over-all requirements for the collection of coal samples. The wide varieties of coal handling facilities preclude the publication of detailed procedures for every sampling situation. The proper collection of the sample involves an understanding and consideration of the physical character of the coal, the number and weight of increments, and the over-all precision required.

1. Scope

1.1 These methods cover procedures for the collection of a gross sample under various conditions of sampling. The gross sample is to be crushed and further prepared for analysis in accordance with Method D 2013.[2] However, the procedures for dividing large gross samples before any crushing are given in this standard.

1.2 These methods describe general and special purpose sampling procedures for coals (*1*) by size and condition of preparation (for example mechanically cleaned coal or raw coal) and (*2*) by sampling characteristics.

2. Applicable Documents

2.1 ASTM Standards:

D 431 Designating the Size of Coal from Its Sieve Analysis[2]

D 2013 Preparing Coal Samples for Analysis[2]

E 177 Recommended Practice for Use of the Terms, Precision, and Accuracy as Applied to Measurement of a Property of Material[3]

3. Summary of Methods

3.1 General-purpose sampling procedures are intended to provide a precision of $\pm 1/10$ of the ash content of the coal sampled in 95 out of 100 cases. To obtain a stated precision in terms of other constituents, the special purpose sampling procedures should be used.

3.2 Special purpose sampling procedures apply to the sampling of coal when other precision limits are required, or when other con-

[1] These methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.23 on Sampling.

Current edition approved Oct. 29, 1976. Published December 1976. Originally published as D 2234 – 63 T. Last previous edition D 2234 – 72.

[2] *Annual Book of ASTM Standards*, Part 26.

[3] *Annual Book of ASTM Standards*, Parts 13 and 14.

stituents are used to specify precision, or for performance tests.

3.3 For coals of known size and condition of preparation, tables are given for the determination of the number and weight of increments required for a gross sample for both general and special purpose sampling. For coals having known sampling characteristics, as determined by the use of appropriate test and statistical procedures given in these methods, the number and weight of the increments required for either general purpose or special purpose precision can be determined.

3.4 The procedures appear in the following order:

4. Definitions

4.1 *accuracy:*

4.1.1 generally, a term used to indicate the reliability of a sample, a measurement, or an observation.

4.1.2 specifically, a measure of closeness of agreement between an experimental result and the true value. Example: the observed and true sulfur content of a coal consignment. This measure is affected by chance errors as well as by bias.

4.2 *air drying*—a process of partial drying of coal to bring it near to equilibrium with the atmosphere in the room in which further reduction and division of the sample is to take place.

4.3 *analysis sample*—final subsample prepared from the original gross sample but reduced to 100 percent through No. 60 (250-μm) sieve and divided to not less than 50 g.

4.4 *bias (systematic error)*—an error that is consistently negative or consistently positive. The mean of errors resulting from a series of observations which does not tend towards zero.

4.5 *chance error*—error that has equal probability of being positive or negative. The mean of the chance errors resulting from a series of observations tends toward zero as the number of observations approaches infinity.

4.6 *consignment*—a discrete amount of coal, such as a shipment, a carload, a unit train, or a day's production. A consignment may include more than one lot of coal and may correspond to a specified period of time such as a sampling period or billing period.

4.7 *error*—difference of an observation or a group of observations from the best obtainable estimate of the true value.

4.8 *free impurity*—the impurities in a coal that exist as individual discrete particles that are not a structural part of the coal and that can be separated from it by coal preparation methods.

4.9 *gross sample*—a sample representing one lot of coal and composed of a number of increments on which neither reduction nor division has been performed.

4.10 *increment*—a small portion of the lot collected by one operation of a sampling device and normally combined with other increments from the lot to make a gross sample.

4.11 *inherent ash*—the residue remaining from the inherent impurities after ignition under conditions specified for the ash determination.

4.12 *inherent impurity*—the inorganic material in coal that is structurally part of the coal and cannot be separated from it by coal preparation methods.

4.13 *lot*—a quantity of coal to be represented by a gross sample.

4.14 *precision*—a term used to indicate the capability of a person, an instrument, or a method to obtain reproducible results; specifically, a measure of the chance error as expressed by the variance, the standard error, or a multiple of the standard error (see Recommended Practice E 177).

4.15 *representative sample*—a sample collected in such a manner that every particle in the lot to be sampled is equally represented in the gross sample.

4.16 *sample*—a quantity of material taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity.

4.17 *sample division*—the process whereby a sample is reduced in weight without change in particle size.

4.18 *sample preparation*—the process that may include air drying, crushing, division and mixing of a gross sample for the purpose of obtaining an unbiased analysis sample.

4.19 *significant loss*—any loss that introduces a bias in final results that is of appreciable economic importance to the concerned parties.

4.20 *size consist*—the particle size distribution of a coal.

4.21 *standard deviation*—the square root of the variance.

4.22 *subsample*—a sample taken from another sample.

4.23 *systematic error* (see 4.4 *bias*)

4.24 *top size*—the opening of the smallest screen in the series upon which is retained less than 5 percent of the sample (see Method D 431).

4.25 *unbiased sample* (*representative sample*)—a sample free of bias.

4.26 *variance*—the mean square of deviations (or errors) of a set of observations; the sum of squared deviations (or errors) of individual observations with respect to their arithmetic mean divided by the number of observations less one (degrees of freedom); the square of the standard deviation (or standard error).

4.26.1 *random variance of increment collection* (*unit variance*), s_r^2—the theoretical variance calculated for a uniformly mixed lot and extrapolated to 1 lb (0.5 kg) increment size. For a method of estimating this variance see Appendix A1.

4.26.2 *segregation variance of increment collection*, s_s^2—the variance caused by nonrandom distribution of ash content or other constituent in the lot. For a method of estimating this variance see Appendix A1.

4.26.3 *total variance*, s_o^2—the over-all variance resulting from collecting single increments, and including division and analysis of the single increments. For a method of estimating this variance see Appendix A2.

5. Increment Collection Classification

5.1 The type of selection, the conditions under which individual increments are collected, and the method of spacing of increments from the coal consignment or lot are classified according to the following descriptions and Table 2. These designations are to be used for sampling specifications and for descriptions of sampling programs, and sampling equipment.

5.2 *Types of Increments*—The types of selection of increments are based on whether or not there is human discretion in the selection of the pieces of coal or portions of the coal stream.

5.2.1 *Type I*, in which specific pieces or portions are not subject to selection on a discretionary basis. This includes that in which the increment is collected in precise accord with previously assigned rules on timing or location that are free of any bias. Type I selection increments generally yield more accurate results.

5.2.2 *Type II*, in which some measure of human discretion is exercised in the selection of specific pieces of coal or of specific portions of the stream, pile, or shipment.

5.3 *Conditions of Increment Collection*—The conditions under which individual increments are collected are the conditions of the main body of coal relative to the portion withdrawn. Four conditions are recognized.

5.3.1 *Condition A* (*Stopped-Belt Cut*), in which a loaded conveyor belt is stopped and a full cross-section cut with parallel sides is removed from the coal stream. The distance between the parallel faces shall not be less than three times the diameter of the largest piece.

5.3.2 *Condition B* (*Full-Stream Cut*), in which a full cross-section cut is removed from a moving stream of coal.

5.3.3 *Condition C (Part-Stream Cut)*, in which a portion, not a full cross section, is removed from a moving stream of coal.

5.3.4 *Condition D (Stationary Coal Sampling)*, in which a portion of coal is collected from a pile, a rail car, a barge, or a shiphold.

5.4 *Spacing of Increments*—The spacing of increments pertains to the kind of intervals between increments. Two spacing methods are recognized: systematic and random. Systematic spacing is usually preferable.

5.4.1 *Systematic Spacing 1*, in which the movements of individual increment collection are spaced evenly in time or in position over the lot.

5.4.2 *Random Spacing 2*, in which the increments are spaced at random in time or in position over the lot.

6. Organization and Planning of Sampling Operations

6.1 *Precaution*—It is imperative that every gross sample be collected carefully and conscientiously and in strict accordance with the procedures prescribed in these methods; for if the sampling is done improperly, the sample will be in error and it may be impossible or impracticable to take another sample. However, if the analysis is in error, another analysis can easily be made of the original sample, except for moisture.

6.2 *Selection of Appropriate Sampling Procedure*—Variations in coal-handling facilities make it impossible to publish rigid rules covering every sampling situation in complete and exact details. Proper sampling involves an understanding and proper consideration of the minimum number and weight of increments, the size consist of the coal, the condition of preparation of the coal, the variability of the constituent sought, and the degree of precision required.

6.2.1 *Number and Weight of Increments*—The number and weight of increments required for a given degree of precision depends upon the variability of the coal. This variability increases with an increase in free impurity. A coal high in inherent impurity and with comparatively little free impurity may exhibit much less variability than a coal with a low inherent impurity and a relatively high proportion of free impurity. For most practical purposes, an increase in the ash content of a given coal usually indicates an increase in variability. It is imperative that not less than the minimum specified number of increments of not less than the minimum specified weight be collected from the lot. For Condition D the increments shall be of equal weight.

6.2.2 *Increment Collection Method to Be Used*—In order to obtain complete representation of all sizes, it is most desirable that the sample increments be withdrawn from the full cross section of the stream. The best possible increment is a full cross-section cut removed from a stopped belt; Classification I-A-1 in Table 1. The best possible increment from a flowing stream of coal is one obtained by moving a cutter device entirely across the stream at a uniform speed, the same for each increment, into one side of the stream and out of the other, without allowing the receptacle to overflow (Classification I-B-1 in Table 1). Classification methods given in Table 1 are listed in order of decreasing reliability. The highest possible classification method, wherever feasible, should be used. Details of sampling procedures should be agreed upon in advance by all parties concerned. Whenever circumstances dictate utilization of increment collection classifications "Condition C" or "Condition D" or "Type II", details of sampling procedure shall be agreed upon in advance by all parties concerned.

6.3 *Distribution of Increments*—It is essential that the increments be distributed throughout the lot to be sampled. This distribution is related to the entire volume of the lot, not merely its surface or any linear direction through it, or over it. If circumstances prevent the sampler from applying this principle, the lot is sampled only in part, and the gross sample is representative only of this part. The spacing of the increments shall be varied if the possibility exists that increment collection may get "in phase" with the sequence of coal variability. Example: routine sampling of commercial coal from a continuous stream (conveyor belt), where increment collection is automatic and its sequence coincides with the "highs" or "lows" in the content of fines.

6.4 *Dimensions of Sampling Device*—The

opening of the sampling device shall be at least 2 1/2 to 3 times the top-size of the coal. However, for practical reasons it is recommended that the opening of any sampling device be not less than 1 1/4 in. (31.8 mm), regardless of the top size of the coal. In sampling from a moving stream, the effective width is the width projected perpendicular to the relative direction of the stream (Note 1). The sampling device shall be of sufficient capacity to completely retain or entirely pass the increment without loss or spillage.

NOTE 1: *Geometry of Cutting Samples from Moving Streams*—Consider the geometric plane that is common to the directions of the cutter movement and of the stream being sampled as shown in the vector diagram in Fig. 1. In this plane, determine α, the angle between these two directions. Draw the vectors V_c and V_s so that their lengths are proportional to the velocities of the cutter and of the stream, in the common plane. See Fig. 1(*a*). Join the origins of these vectors to give V_r, the vector of stream movement relative to the cutter and to get angle β. Apply this angle to a scaled cross section of the sample cutter with W_a being the actual width in the direction of travel. See Fig. 1 (*b*). Measure W_e, the effective cutter width as the width in the relative direction of the stream. When angle β differs appreciably from 90 deg the height of the cutter blades must be sufficient to prevent contamination of sample by particles bounced off the leading face of the cutter.

6.5 *Movement of Sampling Device*—In sampling from moving streams of coal the sampling device shall be designed to minimize disturbance of the coal, thereby avoiding separation of various coal densities and sizes or both. To prevent segregation and rejection due to disturbance of the coal stream, practical evidence indicates that the velocity with which the sampling instrument travels through the stream shall not exceed 18 in./s (457 mm/s).

6.6 *Preservation of Moisture*—The increments obtained during the sampling period shall be protected from changes in composition due to exposure to rain, snow, wind, sun, contact with absorbent materials, and extremes of temperature. The circulation of air through equipment must be reduced to a minimum to prevent both loss of fines and moisture. Samples in which moisture content is important shall be protected from excessive air flow and then shall be stored in moisture-tight containers. Metal cans with air-tight lids, or heavy vapor-impervious bags, properly sealed, are satisfactory for this purpose.

6.7 *Contamination*—The sampling arrangement shall be planned so that contamination of the increments with foreign material or unrelated coal is avoided.

6.8 *Mechanical System Features*—With mechanized systems, it is essential that the system as a whole including the sample cutter, chutes, conveyors, crushers and other devices be self-cleaning and nonclogging and be designed in a manner that will minimize the need for maintenance.

6.9 *Personnel*—Because of the many variations in the conditions under which coal must be sampled, and in the nature of the material being sampled, it is essential that the samples be collected under the direct supervision of a person qualified by training and experience for this responsibility. Where human labor is employed to collect the increments, it is essential that samples be collected by a trained and experienced sampler or under the direct personal observation of such a person. This includes sampling for the purpose of determining sampling characteristics of a coal or characteristics of a particular sampling apparatus.

6.10 *Criteria of Satisfactory Performance*—A satisfactory sampling arrangement is one that takes an unbiased sample at the desired degree of precision of the constituent for which the sample is to be analyzed. One fundamental characteristic of such an arrangement is that the size consist of the sample will adequately represent the true size consist of the coal. Sampling systems shall be tested initially and at regular intervals to determine whether the sample adequately represents the coal. In addition, sampling systems should be given a rough performance check as a matter of routine. This is done by comparing the weight or volume of collected sample with that of the total flow of coal, to assure a constant sampling ratio.

6.11 *Relative Location of Sampling and Weighing*—It is preferable that coal be weighed and sampled at the same point in time. If there is a lapse in time between these two events, consideration should be given by both purchaser and seller to changes in mois-

ture during this interval and the consequent shift in relationship of moisture to the true quality of the coal at the instant when ownership of the coal transfers from one to the other.

7. Sampling of Coals Based on Size and Condition of Preparation

7.1 *General-Purpose Sampling Procedure:*

7.1.1 The general-purpose sampling procedure is intended for a precision such that if gross samples are taken repeatedly from a lot or consignment and one ash determination is made on the analysis sample from each gross sample, 95 out of 100 of these determinations will fall within $\pm 1/10$ of the average of all the determinations. Under some conditions, as detailed in 6.2.1 and 6.2.2, Condition C and D and Type II, this precision may not be obtained.

7.1.2 *Number and Weight of Increments*—Obtain the number and weight of increments as specified in Table 2 except as provided in 7.1.5.2. Determine the minimum number of increments from the condition of preparation, and determine the minimum weight of each increment from the top size of the coal. Classify the coals to be sampled according to the general purpose procedure into three groups by top size. Further classify each of these groups into two subgroups in accordance with the condition of preparation. These classifications are shown in Table 2.

7.1.3 Variations in construction of the sampling device and flow, structure, or size consist of the coal may make it impracticable to collect increments as small as the minimum weight specified in Table 2. In such cases, collect an increment of greater weight. However, do not reduce the minimum number of increments, regardless of large excesses of individual increment weights. Table 2 lists the absolute minimum number of increments for general purpose sampling which may not be reduced except as specified in 7.1.5.2. Other considerations may make it advisable or necessary to increase this number of increments.

7.1.4 *Number of Gross Samples*—Under the general-purpose sampling procedure, for quantities up to approximately 1000 tons (908 Metric Tons) (908 Mg) it is recommended that one gross sample represent the lot. Take this gross sample in accordance with the requirements prescribed in Table 2.

7.1.5 For quantities over 1000 tons (908 Mg), use any of the following alternatives:

7.1.5.1 Take separate gross samples for each 1000-ton lot of coal or fraction thereof.

7.1.5.2 Use one gross sample to represent the total tonnage provided the number of increments, as stated in Table 2, are increased as follows:

$$N_2 = N_1 \sqrt{\frac{\text{total lot size (tons or Mg)}}{\text{1000 tons or 908 Mg}}} \quad (1)$$

where:

N_1 = number of increments specified in Table 2, and

N_2 = number of increments required.

For example, a 4000-ton (3632-Mg) lot will require twice the number of increments specified in Table 2. Using this technique, theoretically it is possible to collect one gross sample to represent a lot of infinite tonnage. However, practical experience indicates maximum size of a lot of coal to be represented by one gross sample should not exceed 10 000 tons (9080 Mg).

7.2 *Special-Purpose Sampling Procedure:*

7.2.1 This special-purpose sampling procedure shall apply to the sampling of coal when increased precision is required, and the only knowledge of the coal is its top size and conditions of preparation.

7.2.2 *Number and Weight of Increments*—Take the same number and weight of increments per gross sample as specified in Table 2, or as specified in 7.1.5.3.

7.2.3 *Number of Gross Samples*—To obtain increased precision for the laboratory result for a given consignment, increase the number of gross samples collected from that consignment and analyze each gross sample separately, reporting the average of results. To reduce errors to one half, that is, to "double" the precision, take four times as many gross samples. Similarly, to reduce errors to one third, to "triple" the precision, take nine times as many gross samples.

8. Sampling of Coals Based on Known Sampling Characteristics

8.1 *Principles of Sampling by Sampling Characteristics:*

8.1.1 The relationship between sampling characteristics (expressed as variances) and the number of increments which will give a desired precision (expressed as the specified variance of one gross sample) is shown as follows:

$$N_I = (s_s^2 + s_r^2/W)/(s_G^2 - s_{da}^2/P) \quad (2)$$

where:

N_I = the number of increments in one gross sample,

W = the weight in pounds of each increment; this is selected for convenience or by the limitations imposed by the particular mechanical sampling apparatus,

s_r^2 = the random variance of a 1-lb (0.5-kg) increment; this value is obtained from the special sampling program given in Appendix A1 (Note A1),

s_s^2 = the segregation variance, this value is also obtained from the special sampling program given in Appendix A1 (Note A1),

s_{da}^2 = the variance of division and analysis. Procedures for calculating this quantity are given in Appendix A2 of ASTM Method D 2013.

P = the number of analysis samples (prepared independently from the same gross sample), and

s_G^2 = the specified variance of one gross sample. The procedure for determining this variance is given in 8.1.2 and 8.1.3.

NOTE 2—The random variance and the segregation variance, s_r^2 and s_s^2, are each inflated by unknown amounts of variance due to division and analysis. Since this results in an increased numerator in Eq 2, and consequently, a larger calculated number of increments, N_I, it can be considered a "safety factor" for the sampling program. However, if too many large increments are taken for the evaluation of s_r^2 and s_s^2, the "safety factor" may become unreasonably large.

8.1.2 The relationship between the specified variance of one gross sample, s_G^2, and the precision for the result of several gross samples in one test period, expressed as the test period variance, s_T^2, is given as follows:

$$s_T^2 = s_G^2/N_G \quad (3)$$

where:

s_T^2 = the test period variance,

s_G^2 = the specified variance of one gross sample, and

N_G = the number of gross samples in the test period.

8.1.3 Fig. 2 shows the relationship between variance and sampling precision (plus or minus percent of a given constituent, 95 cases out of 100). The variance (Fig. 2) can be either the test period variance, s_T^2, or the specified variance of one gross sample, s_G^2. This choice will depend upon the sampling situation to be evaluated. The sampling precision (Fig. 2) can be based on any coal constituent, provided it is expressed as a percentage of that constituent. The following example is an illustration of the calculations necessary to determine the number of increments for one gross sample:

8.1.3.1 *Accuracy Limits*—Assume a coal of 6.5 percent average ash content. If the desired accuracy is ±1/10 of the ash content, the sampling accuracy can be expressed as ±0.65 percent ash.

8.1.3.2 *Test Period Variance*—The accuracy limits given in 8.1.3.1 correspond to a test period variance, s_T^2, of 0.112, from Fig. 2 (Line 1).

8.1.3.3 *Specified Variance of One Gross Sample*—The specified variance of one gross sample is equal to the test period of variance, s_T^2, multiplied by the number of gross samples in the test period, N_G (Eq 3). Assuming seven gross samples in the period, the specified variance for one gross sample is then equal to 0.112 × 7 or 0.784.

8.1.3.4 *Number of Increments*—Assume the following information was obtained from the special sampling procedures outlined in Appendix A1 of this procedure and Appendix A2 of ASTM Method D 2013: $s_r^2 = 12.5$, $s_s^2 = 10.2$, and $s_{da}^2 = 0.06$ (one analysis sample per gross sample). The specified variance of one gross sample, s_G^2, as found previously, is 0.784. Further, the weight per increment for this sampling device, is found to be 50 lb (23 kg). Then, substituting into Eq 2:

$$N_I = (10.2 + 12.5/50)/(0.784 - 0.06/1)$$
$$= 14.4 \text{ or } 15 \text{ increments}$$

For this coal, 15 increments of 50 lb (23 kg) each would be required for each gross sample, and seven gross samples would constitute the sampling period. The weighted average for the test period will be within ±0.65 percent ash, 95 cases out of 100.

8.1.4 The following variance relationship

can be derived from Eq 2. It combines the random variance, s_r^2, and the segregation variance, s_s^2. This derivation is applicable when the incremental weight is fixed by the characteristics of the sampling equipment:

$$s_T^2 = [(s_o^2 - s_{da}^2)/N_G N_I] + (s_{da}^2/N_{Gp}) \quad (4)$$

where:

s_o^2 = the over-all variance for single increments (including division and analysis), as determined by Appendix A2.

Other terms are as defined in 8.1.1 and 8.1.2. The following example demonstrates the use of the overall variance for increments, s_o^2, in determining the number of increments for one gross sample:

8.1.4.1 *Test Period Variance*—Assume a required accuracy of ±0.5 percent ash. This corresponds to a test period variance, s_T^2, of 0.066 from Fig. 2 (Line 2).

8.1.4.2 *Number of Increments*—Assume the following information was obtained from the special sampling procedures outlined in Appendix A2 of ASTM Method D 2013, and Appendix A2 of this procedure: $s_o^2 = 3.5$ and $s_{da}^2 = 0.08$. If it is desired to take ten gross samples during the test period (N_G), with only one analysis (p) for each gross sample, the number of increments for each gross sample (N_I) can be determined by substitution into Eq 4:

$$0.066 = [(3.5 - 0.08)/10N_I] + [0.08/(10 \times 1)]$$

where:

N_I = 5.9 or 6 increments.

For this coal, six increments would be required for each of the ten gross samples. The weighted average for the test period will be within ±0.5 percent ash, 95 cases out of 100.

8.1.5 For sampling mixed coals, the values of random variance, segregation variance, over-all variance for increments, and the variance of division and analysis for use in Eq 2, 8.1.1 and Eq 4, 8.1.4, are those obtained from special sampling programs using the mixture which is the most difficult to sample.

8.2 *General-Purpose Sampling Procedure:*

8.2.1 *Number and Weight of Increments*—Use this general-purpose sampling procedure for the commercial sampling of coal. Use this procedure to obtain a precision of ±1/10 of the average of all ash determinations in 95 out of 100 cases by taking gross samples repeatedly from one lot, and making one ash determination on the analysis sample from each gross sample. For this degree of precision, determine the number and weight of increments for each gross sample as illustrated in 8.1.3 and 8.1.4.

8.2.2 *Number of Gross Samples*—Select the number of gross samples for coals of known sampling characteristics to suit the parties concerned with results from the sampling, since the number of gross samples is directly related to the establishment of the required number of increments as outlined in 8.1.3 and 8.1.4. The following factors should be remembered in selecting the number of gross samples for the test period:

8.2.2.1 Too few gross samples will result in additional preparation work because of the large number of increments per gross sample which will be required.

8.2.2.2 The preparation of the samples for analysis purposes will be simplified by using samples of minimum weight.

8.2.2.3 Use special precautions if the gross samples are to be used for moisture determinations. A gross sample collected over a long period of time will change in moisture content depending upon the surrounding atmospheric conditions. See 6.6.

8.3 *Special-Purpose Sampling Procedure:*

8.3.1 Apply this special-purpose sampling procedure to the sampling of coal when other precision limits are required, or when other constituents are used to specify precision.

8.3.2 *Number and Weight of Increments and Number of Gross Samples*—For a precision of ±1/20 of the average of all the ash determinations in 95 out of 100 cases when gross samples are repeatedly taken from the same lot, use Fig. 2 to determine the test period variance, s_T^2. In this case, use the new sampling precision limitations of ±1/20 of the average ash in Fig. 2. Then determine the number of increments and number of gross samples as outlined in 8.1.3, 8.1.4, and 8.2.2.

8.3.3 For a precision of ±1/30 of the average ash, use Fig. 2 again to determine the test pe-

riod variance, s_T^2. In this case, use the new sampling precision limitations in Fig. 2.

8.3.4 Other precision limits may be used or other constituents may be used to specify precision when agreed upon by the parties concerned. The principles outlined in this section will apply to all special precision limits.

8.3.5 Greater accuracy cannot be obtained by merely increasing the weight and number of increments if significant bias exists.

9. Division of the Gross Sample Before Crushing

9.1 In the case of very large and unwieldy gross samples, it is permissible to divide the gross sample to reduce its weight, provided the following conditions are fulfilled:

9.1.1 If the entire gross sample is mixed in a suitable blender (double cone or twin shell tumbler) it is permissible to divide the sample using the schedule of Table 2. Test the divided sample for bias.

9.1.2 If each very large increment is reduced in quantity by secondary sampling, take at least six secondary increments from each primary increment. The method of collection of secondary increments must be proved to be free from bias. In no case shall the weight of a secondary increment be less than shown in the schedule of Table 2.

10. Sampling of Coal for Total Moisture Determinations

10.1 *Types of Moisture Samples*—Moisture determinations as specified in the method to be employed, are to be made on the following kinds of samples.

10.1.1 *Entire Gross Sample*—For referee tests, air dry the entire gross sample and measure the weight loss from the entire gross sample during this drying. This procedure can be carried out on the entire gross sample as a single batch or on groups of primary increments, or as separate operations on the individual primary increments; but obtain, by one of these means, the total weight loss from the entire gross sample. After this air drying, the sample can be crushed or divided or both as required by the referee test for moisture.

10.1.2 *Special Moisture Subsample*—For moisture testing, a special subsample can be taken from a gross sample before any operations of air drying or crushing. Take this subsample from the gross sample in accord with the requirements of Section 9.

10.1.3 *Other Subsamples for Moisture Testing*—For moisture testing, a subsample can be used that is collected after the initial crushing and dividing of a gross sample. The procedures for the crushing and dividing, and for this subsequent subsampling for moisture, are given in Method D 2013.

10.2 *Special Precautions*—Collect samples and subsamples for moisture in such a manner that there is no unmeasured loss of moisture of significant amount. Make adequate weighings before and after drying or other operations to measure all significant weight losses.

10.3 *Weight of Increments*—The minimum weight of each increment must be that which is sufficient as to be free of bias. This depends on the top size of the coal in the stream being sampled, the dimensions of the collection device, and other factors of the withdrawal of the increment. Since much of the moisture tends to be distributed uniformly across the surface, moisture bias is present when the size consist of the sample is not the same as the size consist of the lot sampled. In addition, when there is no knowledge of the sampling characteristics for moisture, each increment shall weigh not less than the values in Table 2.

10.4 *Number of Increments*—The number of increments required for a given degree of precision depends on the weight of the increments, the distribution of the moisture, and the total amount of moisture. However, the distribution of moisture is not easily evaluated independent of total moisture; consequently, the combined effects can be measured by determining the sampling characteristics for moisture.

10.4.1 *Moisture Sampling Based on Known Sampling Characteristics*—When the sampling characteristics for moisture are known, calculate the number of increments required for a desired degree of precision. The procedures are those given in Section 8 of this procedure.

10.4.2 *Moisture Sampling Based Only on Size*—When there is no knowledge of the sampling characteristics for moisture, collect at least the number of increments from the lot of coal as those given in Table 2. Also, when a special moisture subsample is taken from the gross sample before any drying or crushing operations, collect the number of increments for the subsample as specified in Section 9.

TABLE 1 Increment Types, Conditions, and Spacing

Condition of Increment Collection from the Main Body of Coal	Types of Increment			
	Type I No Human Discretion Is Employed		Type II Human Discretion Is Employed	
	Spacing of Increments		Spacing of Increments	
	1. Systematic	2. Random	1. Systematic	2. Random
Condition A, stopped belt cut	I-A-1	I-A-2	II-A-1	II-A-2
Condition B, full stream cut	I-B-1	I-B-2	II-B-1	II-B-2
Condition C, part stream cut	I-C-1	I-C-2	II-C-1	II-C-2
Condition D, stationary sampling	I-D-1	I-D-2	II-D-1	II-D-2

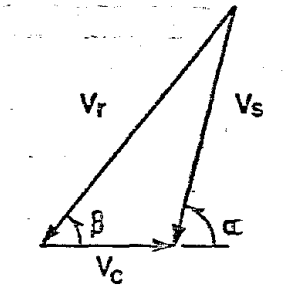
TABLE 2 Number and Weight of Increments for General Purpose Sampling Procedure[a]

Top Size	⅜ in. (16 mm)	2 in. (50 mm)	6 in. (150 mm)
Mechanically Cleaned Coal[c]			
Minimum number of increments	15	15	15
Minimum weight of increments, lb	2	6	15
Minimum weight of increments, kg	1	3	7
Raw (Uncleaned Coal)[c]			
Minimum number of increments	35	35	35
Minimum weight of increments, lb	2	6	15
Minimum weight of increments, kg	1	3	7

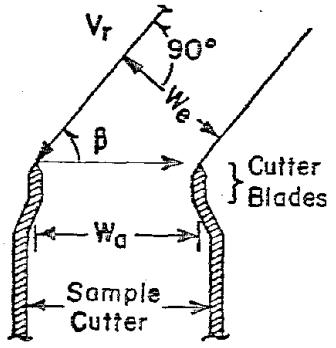
[a] Under conditions C and D see 6.2.1 and 6.2.2.

[b] For coals above 6 in. (150 mm) top size, the sampling procedure should be mutually agreed upon in advance by all parties concerned.

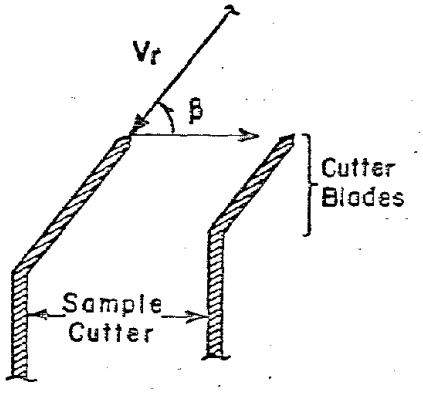
[c] If there is any doubt as to the condition of preparation of the coal (for example, mechanically cleaned coal or raw coal) the number of increments for raw coal shall apply. Similarly, although a coal has been mechanically cleaned, it may still show great variation because of being a blend of two different portions of one seam or a blend of two different seams. In such cases, the number of increments should be as specified for raw (uncleaned) coal.



(*a*) Vector Diagram.



(*b*) Effective Width.



(*c*) Inclined Cutter Blades.

FIG. 1 The Geometry of Cutting Samples from a Moving Stream.

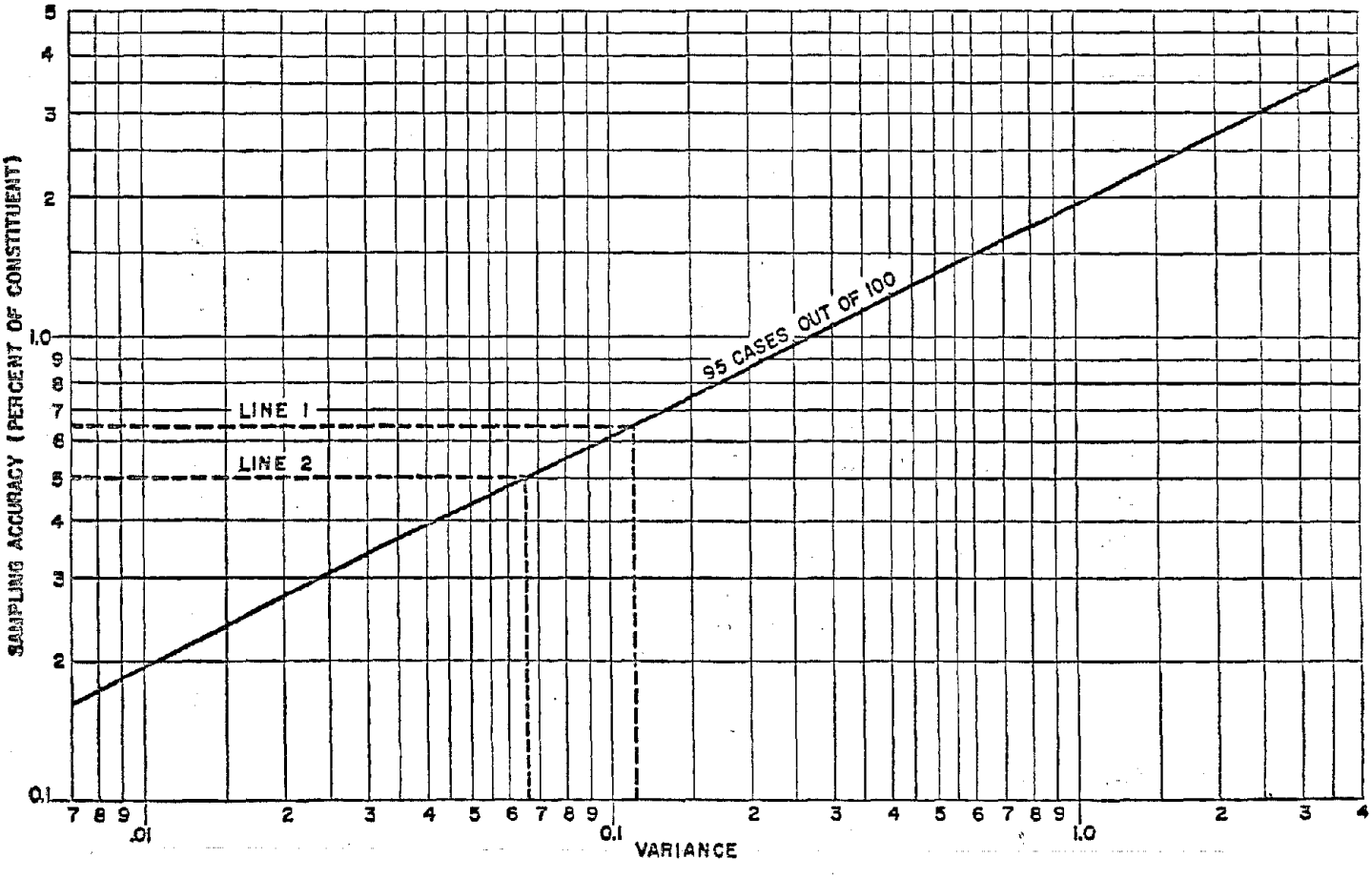


FIG. 2 Conversion of Sampling Accuracy to Variance.

APPENDIXES

A1. METHOD FOR DETERMINING THE VARIANCE COMPONENTS OF A COAL

A1.1 Scope

A1.1.1 This method covers a procedure for determining the following variance components of a coal:

A1.1.1.1 The random variance of a 1-lb (0.5-kg) increment, s_r^2, and

A1.1.1.2 The segregation variance, s_s^2, the variance caused by nonrandom distribution of the ash content in the lot or consignment.

A1.1.2 In this method, each different coal will require a complete experiment, which involves the collection of two sets of 30 samples from a stopped conveyor belt. The first set of samples includes 30 very small samples to furnish data for the random variance; the second set includes 30 large samples to furnish data for the segregation variance. Since one of the important components of variance is that due to segregation, it is essential that the 30 large samples be so distributed with respect to time that coverage of all subtypes of coal are represented.

A1.2 Apparatus

A1.2.1 The following equipment, in addition to that equipment normally provided for routine sampling, will be required:

A1.2.1.1 *Two-Section Belt Divider*—One of the sections should be approximately the width corresponding to three times the top size of the coal, and should trap a sample of between 4 and 20 lb (2 and 9 kg). The other section should be approximately the width corresponding to 20 times the top size of the coal, and should trap a sample of between 80 and 150 lb (36 and 68 kg). The bottom edges of the divider should be shaped to conform to the surface of the conveyor belt.

A1.2.1.2 *Riffle Splitter*, with slots at least 2½ to 3 times as wide as the maximum size of the particles, or, a manual divider and canvas for subdividing the small samples by hand.

A1.3 Procedure

A1.3.1 The following sampling procedure should be used for each of the two required sets of samples:

A1.3.1.1 Stop the loaded belt and insert the belt divider with the division plates perpendicular to the direction of belt movement. Scrape off the coal from each section, and put each section into a separate completely labeled container. The container holding the coal from the small section of the belt divider should be labeled "A." The container holding the coal from the large section of the belt divider should be labeled "B."

A1.3.1.2 Collect a subsample from the "A" section by riffling or by manual subdivision after spreading the sample evenly on a smooth flat surface. Tag the subsample with a label "A", and weigh to the nearest gram. The weight of the subsample should be between 100 to 200 g.

A1.3.1.3 Dry the "A" subsample, grind to minus No. 60 sieve size and determine the ash content to the nearest 0.1 percent, dry basis.

A1.3.1.4 Weigh the entire "B" section, dry, and work down to an analysis sample. Determine the ash content to the nearest 0.1 percent, dry basis.

A1.4 Calculation

A1.4.1 Calculate the variance of the "A" and "B" series (Note A1) as follows:

$$\text{Variance} = (\Sigma x^2 - (\Sigma x)^2/n)/(n - 1) \quad (5)$$

where:

Σx^2 = the sum of the squares of ash results,
$(\Sigma x)^2$ = the square of the sum of ash results, and
n = the number of individual ash results in the series.

A1.4.2 The random variance, s_r^2, is found from:

$$s_r^2 = [W_1 W_2 (s_A^2 - s_B^2)]/(W_2 - W_1) \quad (6)$$

where:

W_1 = average weight of small samples, lb or equivalent kg,
W_2 = average weight of large samples, lb or equivalent kg,
s_A^2 = variance of small, "A" samples, and
s_B^2 = variance of large "B" samples.

A1.4.3 The segregation variance, s_s^2, is found from:

$$s_s^2 = s_B^2 - s_r^2/W_2 \quad (7)$$

NOTE A1—An actual example illustrating the treatment of data from this sampling experiment is given in Tables A1 to A3 and in Fig. A1.

A1.4.3.1 Using log-log paper, plot the point corresponding to an increment weight w = 1 lb (0.5 kg) and variance s_r^2 = 7.6; draw a straight line through this point, downward at 45 deg. This line gives the random component of variance for an increment of any weight. Plot the point corresponding to an increment weight w_2 = 106 lb (48 kg) and variance s_s^2 = 1.2; draw a straight horizontal line through this point. This line gives the segregation component of variance for an increment of any weight.

A1.4.3.2 On Fig. A1, find the algebraic sum of the random component and the segregation component of variance for a number of increment weights: draw a curve through these points. This curve gives the total variance of sampling for increments of any weight, including those used in the "A" and "B" series.

A2. METHOD FOR ESTIMATING THE OVER-ALL VARIANCE FOR INCREMENTS

A2.1 Scope

A2.1.1 This method describes the procedure for estimating the over-all variance for increments of one fixed weight of a given coal. It is applicable to mechanical sampling when there is no need to explore system and random variance components, but there is a need for obtaining the over-all variance for increments (the size of increments is dictated by the sampling equipment).

A2.2 Procedure

A2.2.1 The following procedure should be used to determine the over-all variance for increments:

A2.2.1.1 Collect two series of individual increments at widely spaced intervals; for example, a series of 10 increments, two each day for five days, followed by a second series of 10 collected in similar fashion. Both series must be from the same coal.

A2.2.1.2 Collect each increment by using as much of the equipment and procedure employed in routine sampling operations as possible. Remove the individual increment from the sampling system without mixing with or contaminating by any other increment. Where possible, allow it to pass through any mechanical crusher or subsampler, or both, which is located in the system prior to the point of blending with other increments.

A2.2.1.3 Then weigh the individual increment (if desired for record purposes) and reduce to a laboratory sample by procedures identical as possible to those employed in the routine preparation and reduction of gross samples.

A2.2.1.4 Analyze the sample for the constituents for which the variance calculations are to be made. Usually sampling specifications are based on dry ash; but where total moisture or as-received Btu is of particular concern, the analyses should be made for these.

A2.3 Calculation

A2.3.1 For each series, compute a variance value from the analyses of the ten increments as follows:

$$s^2 = (\Sigma x^2 - (\Sigma x)^2/n)/(n - 1) \quad (8)$$

where:

s^2 = the variance value for the series,
Σx^2 = the sum of squares of ash results,
$(\Sigma x)^2$ = the square of the sum of ash results, and
n = the number of individual ash results in the series.

A2.3.2 For the two series, the ratio of the larger variance to the smaller should not exceed the value given in Table A4, Column 2. If they differ by less than this amount, the variances are combined to give the estimated over-all increment variance for the coal as follows:

$$s_o^2 = F[(s_1^2 + s_2^2)/2] \quad (9)$$

where:

s_o^2 = the probable maximum value of the over-all variance for increments,
F = a factor from Table A5, Column 3, corresponding to the number of increments per set,
s_1^2 = s^2 from first series, and
s_2^2 = s^2 from second series.

A2.3.3 If the ratio of the larger variance to the smaller does give a greater value than the Table A4, Column 2 value, the two series are to be considered in a single set of increments, and another set equal to this enlarged set is to be taken. For example, if originally two sets of 10 increments were taken, these would be combined to give a set of 20. Then an additional set of 20 increments would be collected, giving two sets of 20 increments each. Variance values are computed for the two new series and the test is repeated using the appropriate factors given in Table A5. If these results have a ratio which is less than the appropriate value in Column 2 of Table A5, they are combined by using Eq 10 and used as the new variance for increments.

A2.3.4 *Example*—The example given in Table A5 illustrates the computation of the over-all variance for increments, s_o^2. Two series of 10 increments each are used.

TABLE A1 Schedule I: Sample Weights

Set Number	"A" Series, g	"B" Series, lb
1	89	117.4
2	126	117.5
3	152	123.4
4	109	90.7
5	149	101.7
6	87	89.6
7	110	107.7
8	142	110.8
9	123	123.0
10	111	106.2
11	140	116.4
12	121	96.7
13	112	109.0
14	122	106.9
15	158	99.8
16	160	87.6
17	55	88.6
18	76	92.3
19	105	93.0
20	132	99.8
21	108	106.6
22	86	124.2
23	142	127.8
24	123	111.3
25	133	111.6
26	261	107.2
27	129	106.0
28	150	102.8
29	108	97.7
30	99	107.4
Sum	3732	3180.7
Average	124.4 g, or 0.27 lb = w_1	106.0 lb or 48.1 kg = w_2

TABLE A2 Schedule II: Ash Results "A" Series

Set	Ash, percent	Ash, percent-squared
1	14.2	201.64
2	13.4	179.56
3	13.7	187.69
4	15.8	249.64
5	13.7	187.69
6	14.1	198.81
7	13.6	184.96
8	18.7	349.69
9	16.3	265.69
10	12.4	153.76
11	5.8	33.64
12	12.2	148.84
13	10.9	118.81
14	8.9	79.21
15	34.5	1190.25
16	8.7	75.69
17	7.5	56.25
18	15.7	246.49
19	21.8	475.24
20	11.8	139.24
21	12.2	148.84
22	11.8	139.24
23	7.1	50.41
24	12.8	163.84
25	14.0	196.00
26	6.3	39.69
27	12.3	151.29
28	7.2	51.84
29	13.1	171.61
30	11.3	127.69
Sum	391.8	5963.24

$s_A^2 = [5963.24 - (391.8)^2/30]/29 = 29.2$

TABLE A3 Schedule III: Ash Results "B" Series

Set	Ash, percent	Ash, percent-squared
1	13.6	184.96
2	13.2	174.24
3	14.3	204.49
4	15.3	234.09
5	15.0	225.00
6	14.3	204.49
7	13.6	184.96
8	14.7	216.09
9	14.2	201.64
10	12.5	156.25
11	13.0	169.00
12	14.3	204.49
13	13.7	187.69
14	12.8	163.84
15	13.2	174.24
16	14.0	196.00
17	10.5	110.25
18	13.5	182.25
19	15.4	237.16
20	15.0	225.00
21	14.4	207.36
22	12.8	163.84
23	13.0	169.00
24	13.0	169.00
25	12.3	151.29
26	13.1	171.61
27	14.2	201.64
28	11.6	134.56
29	13.1	171.61
30	11.4	129.96
Sum	405.0	5506.00

$$s_B^2 = \frac{[5506.00 - (405)^2/30]}{29} = 1.3$$

then:

$$s_r^2 = \frac{[0.27 \times 106\ (29.2 - 1.3)]}{106 - 0.27}$$

$$= 7.6$$

and

$$s_s^2 = 1.3 - (7.6/106)$$
$$= 1.2$$

TABLE A4 Variance Ratio Limit Values

1	2	3
Increment per Set	Variance Ratio Limit	"F" Factor
10	3.18	1.92
20	2.17	1.53
30	1.86	1.40
40	1.70	1.33
50	1.61	1.29

TABLE A5 Determination of the Over-All Variance for Increments[a]

Series 1			Series 2		
Increment Number, n	Dry Ash[b] (x)	$(\text{Dry Ash})^{2,b}\ (x)^2$	Increment Number, n	Dry Ash[b] (x)	$(\text{Dry Ash})^{2,b}\ (x)^2$
1	4.17	17.3889	11	3.07	9.4249
2	3.62	13.1044	12	4.88	23.8144
3	1.79	3.2041	13	5.14	26.4196
4	4.37	19.0969	14	3.63	13.1769
5	4.64	21.5296	15	3.17	10.0489
6	7.03	49.4209	16	7.20	51.8400
7	6.27	39.3129	17	3.52	12.3904
8	3.91	15.2881	18	0.87	0.7569
9	6.04	36.4816	19	0.72	0.5184
10	4.18	17.4724	20	4.78	22.8484
Sum	46.02	232.2998	Sum	36.98	171.2388

[a] This example involves increment weights in the approximate range of 100 to 200 lb (45 to 90 kg).
[b] 10 percent ash was subtracted from each of the ash results to simplify the calculations.

$$s^2 = (\Sigma(x)^2 - (\Sigma x)^2/n)/(n-1)$$

Series 1:

$$s_1^2 = (232.2998 - (46.02)^2/10)/9 = 2.2795$$

Series 2:

$$s_2^2 = (171.2388 - (36.98)^2/10)/9 = 3.8319$$

Variance ratio limit from Table A5 = 3.18
Variance ratio for two test series:

$$s_2^2/s_1^2 = 3.8319/2.2795 = 1.68 < 3.18$$

Since the computed value for the ratio is less than 3.18, variances are combined to give an estimate of the over-all variance for increments, s_o^2:

$$s_o^2 = [1.92\ (2.2795 + 3.8319)]/2 = 5.867$$

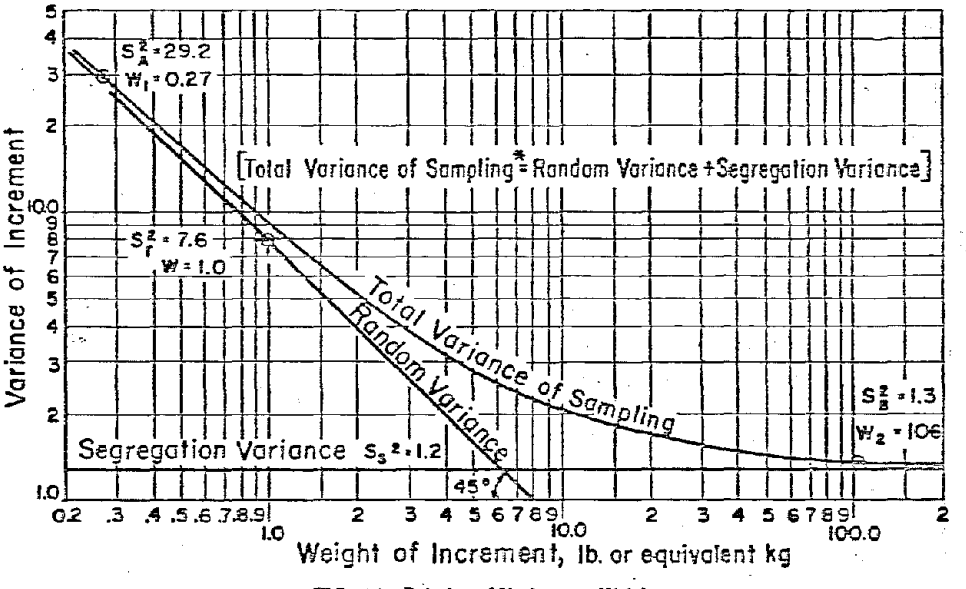


FIG. A1 Relation of Variance to Weight.

APPENDIX C

STANDARD METHOD FOR TOTAL MOISTURE IN COAL

Designation: D 3302 – 74$^{\epsilon}$

Standard Test Method for TOTAL MOISTURE IN COAL[1]

This Standard is issued under the fixed designation D 3302; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

$^{\epsilon}$ NOTE—Editorial addition of Fig. 1 was made in June 1976.

1. Scope

1.1 This method covers the measurement of the total moisture in coal as it exists at the site, at the time, and under the conditions it is sampled. It is applicable to coals as mined, processed, shipped, or utilized in normal commercial pursuits. Exceptions are coal-water slurries, sludges, or pulverized products, or both, under 0.5-mm diameter sieve size. It is applicable to coals of all ranks within the recognized limitations imposed by oxidation and decomposition variations of lower rank coals. Because of its empirical nature, strict adherence to basic principles and permissive procedures are required for valid results (see Appendix). The standard is available to producers, vendors, and consumers as a total moisture method when other procedures or modifications are not mutually agreed on.

1.2 Since coal can vary from extremely wet (water saturated) to completely dry, special emphasis must be placed on the sampling, sample preparation, and the moisture determination itself to ensure total reliability of measurement. Therefore, this standard is recommended entailing collection of the gross sample, sample preparation, and the method of determination.

1.3 While it is recognized that such a standard may be unwieldy for routine usage commercial operations, it can provide a common base for agreement in cases of dispute or arbitration. Also, embodied in the standard are segments that can be extracted and used as is, or, with the agreement of the parties involved, as the basis for contract specifications.

2. Applicable Documents

2.1 *ASTM Standards:*

D 2013 Preparing Coal Samples for Analysis[2]

D 2234 Collection of a Gross Sample of Coal[2]

D 3173 Test for Moisture in the Analysis Sample of Coal and Coke[2]

3. Summary of Method

3.1 This method is based on the loss in weight of a coal sample in an air atmosphere under rigidly controlled conditions of temperature, time, and air flow.

3.2 The gross moisture sample is air-dried to equilibrate it with the atmosphere at each stage of division and reduction. Final moisture determination is made in a heated forced-air circulation oven under rigidly defined conditions.

3.3 The total moisture is calculated from losses (or gains) in air-drying and the residual moisture as shown in Section 11.

4. Significance

4.1 The collection and treatment of the sample as specified herein is intended for the express purpose of determining the total moisture in coal. The standard is available to producers, vendors, and consumers as a method of determination when other techniques or modifications are not mutually agreed on.

4.2 Specific sections of the procedure may be designated as a valid total moisture determination upon agreement between or among the parties involved and designated as such.

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[1] This method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke.

Current edition approved Feb. 27, 1974. Published April 1974.

[2] *Annual Book of ASTM Standards*, Part 26.

5. Definitions

5.1 *air drying*—a process of partial drying of coal to bring to near equilibrium with the atmosphere in the room in which further reduction/division of the sample is to take place.

5.2 *air dry loss*—the loss in weight, expressed in percentage, resulting from the partial drying of coal at each stage of reduction or division.

5.3 *residual moisture*—that moisture remaining in the sample after determining the air-dry loss.

5.4 *total moisture*—the loss in weight in an air atmosphere under the rigidly controlled conditions of temperature, time, and air flow as set in this standard. It is calculated from losses (or gains) in air-drying and the residual moisture as shown in Section 11.

5.5 *gross moisture sample*—a sample representing one lot of coal and composed of a number of increments on which neither reduction nor division has been performed *or* a subsample for moisture testing that is taken in accordance with Section 10 of Method D 2234.

6. Apparatus

6.1 *Drying Floor*—A smooth clean floor area in a room free of contamination by dust or other material and that permits air circulation without excessive heat or air currents. Conditions for an air-drying floor should approach those established for oven drying as much as possible.

6.2 *Air Drying Oven*—A device for passing slightly heated air over the sample. The oven should be capable of maintaining a temperature of 10 to 15°C (18 to 27°F) above room temperature with a maximum oven temperature of 40°C (104°F) unless ambient temperature is above 40°C (104°F) in which case ambient temperature shall be used. In case of easily oxidized coals, the temperature should not be over 10°C (18°F), above room temperature. Air changes should be at the rate of 1 to 4/min. A typical oven is shown in Fig. 1.

6.3 *Drying Pans*—Noncorroding metal pans of sufficient size so that the sample may be spread to a depth of not more than 25 mm (1.0 in.) with sides not more than 38 mm (1.5 in.) high.

6.4 *Scale (Gross Sample)*—A scale of sufficient capacity and sensitive to 23 g (0.05 lb) in 45 kg (100 lb).

6.5 *Laboratory Sample Containers*—Heavy vapor-impervious bags, properly sealed, or noncorroding cans such as those with an airtight, friction top or screw top sealed with a rubber gasket and pressure-sensitive tape for use in storage and transport of the laboratory sample. Glass containers, sealed with rubber gaskets, may be used but care must be taken to avoid breakage in transport.

7. Precautions

7.1 In collecting, handling, reducing, and dividing the gross moisture sample, all operations shall be done rapidly and in as few operations as possible, since moisture loss depends on several factors other than total moisture content, such as time required for crushing, atmospheric temperature and humidity, and type of crushing equipment.

7.2 While awaiting preparation, the uncrushed gross moisture sample shall be protected from moisture change due to exposure to rain, snow, wind, and sun, or contact with absorbant materials.

7.3 The initial weight of the original gross moisture sample and container shall be recorded, and the moisture loss or gain of sample and containers shall be determined before the sample is reduced.

7.4 Whenever a distinct change of humidity occurs during the course of preparation of an air-dried subsample, the subsample should be weighed and equilibrated with the new atmosphere, and the weight loss or gain used in the calculation of moisture content.

7.5 Whenever subsamples are stored or transported, the containers and subsamples shall be weighed, equilibrated to the new atmosphere by air-drying, and the weight loss or gain shall be used in the calculation of moisture content.

7.6 Since most coals oxidize on exposure to air, the air drying procedure should not be prolonged past the time necessary to bring the sample to equilibrium with the air in the room in which further reduction and division are to be made. In no case should the air drying be done at a temperature over 40°C. The sample shall be allowed to attain room temperature before

weighing and further reduction. Air drying of low rank coals should not exceed 18 h because of oxidation.

8. Sampling

8.1 The principles, terms, organization, and collection as set forth in Method D 2234 shall apply to the collection of the total moisture sample. Particular attention is directed to Section 10. The increments as established in Table 2 of Method 2234 for mechanically cleaned coal are deemed adequate for general-purpose sampling for total moisture (see Table 1).

9. Sample Preparation (Air-Drying)

9.1 The principles, terms, organization, and preparation procedures as established in Method D 2013 shall apply to the handling and preparation for total moisture. Specific Reference is made to Sections 1.3.1, Fig. 4, Sections 8.2.1, 8.4.2. One of two procedures must be used in handling the total moisture sample.

9.2 Procedure A provides for using a drying floor to reduce the surface moisture to such a level that the sample can be reduced in size or amount, or both, without moisture loss during the processing.

9.3 Procedure B provides for using an air-drying oven to equilibrate the sample prior to reduction in size or amount, or both.

10. Procedure

10.1 *Procedure A; Drying Floor*—This procedure is particularly applicable if the gross moisture sample is too large in amount to ship reasonably or is too wet to handle or ship without loss of moisture.

10.1.1 Weigh the gross moisture sample and spread on the drying floor to a depth of not more than twice the top size of the coal. Mix or stir the coal from time to time, being careful not to lose any of the coal particles. Continue the air-drying and mixing until the surface of the sample appears dry. Weigh the entire sample and redistribute over the floor for additional drying. Continue the drying, stirring, and weighing until the weight loss of the total sample is not more than 0.1 %/h.

NOTE 1—If the sample surface appears dry, and the time contemplated for reduction and division is well established, air drying can be stopped when the weight loss is less than 0.1 % per twice the contemplated time for processing.

Example—If reduction and division of the sample is estimated to require 20 min, the air-drying procedure can be stopped when the rate of moisture loss is less than 0.1 %/40 min. If this procedure is used, a second air-drying is required to establish the 0.1 %/h rate before the final preparation of the laboratory sample.

10.1.2 Avoid excess drying. Record the weight of the gross moisture sample after drying time. Proceed with sample reduction and division in accordance with Method D 2013.

10.2 *Procedure B; Air Dry Oven*—Distribute the gross moisture sample over the required number of tared pans. Weigh each pan with sample as it is filled from the gross sample. Place in the moisture oven that has been adjusted to the proper temperature and air flow. The oven temperature should be held at a temperature of 10 to 15°C (18 to 27°F) above room temperature (not to exceed 40°C). Air circulation through the oven should be maintained at a rate of at least one air exchange per minute but in no case should it be sufficiently high to blow fine particles from the pans. Gently stir the sample from time to time to ensure uniform drying throughout the sample. Continue drying with intermittent stirring until the coal surfaces appear to be dry. Remove from oven, weigh, and record the weight. Return the pans with sample to the oven and continue the operation. Calculate the percentage weight loss. Repeat the drying and weighing process at 1 to 2-h intervals until the weight loss is less than 0.1 %/h (Note 1). Allow the sample to reach equilibrium with room temperature and humidity before the final air dry weight is recorded.

11. Residual Moisture on Prepared Sample

11.1 The procedure for determining the residual moisture on the prepared sample is dependent upon the top size to which the prepared sample is crushed. Procedure A is used on samples prepared to a top size of No. 8 (2.36-mm) sieve U.S.A. Standard. Procedure B is used on samples prepared to a top size of No. 60 (250-μm) sieve U.S.A. Standard.

11.2 *Procedure A, Top Size—No. 8 (2.36-mm) sieve U.S.A. Standard:*

11.2.1 That portion of the sample used for

the residual moisture determination shall have a minimum weight of 500 g.

11.2.2 Determine and record the weight of a drying pan preconditioned at 107°C. Place the sample and sample container in the drying pan and determine the total weight (Note 2). Distribute the sample uniformly over the pan, being sure not to exceed a depth of 1 in. (25 mm).

NOTE 2—If the moisture determination is to be made in the immediate vicinity of sample preparation, the sample need not be placed in a container but can be weighed directly in a tared drying pan.

11.2.3 Place both the pan containing the sample and the empty sample container in the oven at a temperature of 107 ± 3°C. Dry for 1½ h, remove from the oven, and weigh immediately.

11.2.4 Return to the oven for an additional ½ h, remove, cool, and weigh. Repeat the drying at ½-h intervals until the weight loss is not more than 0.05 % for the ½-h period. For the final weighing, place the dried sample container on the pan of dried coal and record the total weight.

11.2.5 Remove any residual coal from the dried sample container and weigh the empty container (Note 2).

11.3 *Procedure B—Top Size—No. 60 (250-μm) sieve U.S.A. Standard:* (This procedure is the same as that described in Method D 3173.)

11.3.1 Heat an empty capsule under the conditions, at which the sample is to be dried, place the cover on the capsule, cool over a desiccant for 15 to 30 min, and weigh. Dip out with a spoon or spatula from the sample bottle approximately 1 g of the sample. Put this quickly into the capsule, close, and weigh at once. Place the capsules in a preheated oven (107 ± 3°C) through which passes a current of air that has been dried by H_2SO_4 (sp gr 1.84) or other suitable desiccant such as Drierite or magnesium perchlorate. Close the oven at once and heat for 1 h. Open the oven, cover the capsules quickly, cool in a desiccator over desiccant, and weigh as soon as cold.

12. Calculations

12.1 Calculate the percent total moisture, M, as follows:

$$M = [R\ (100\text{-}A)/100] + A$$

where:
M = total moisture, %,
A = air-dry loss, %, and
R = residual moisture, %

12.2 Calculate percent air-dry loss, A, as follows:

$$A = (L/G)100$$

where:
A = air-dry loss, %
L = loss in weight in air-drying, and
G = weight of gross sample.

12.3 Calculate percent residual moisture, R, as follows:

$$R = [(W\text{-}H)/W] \times 100$$

where:
W = weight of sample used, and
H = weight of sample after heating.

13. Precision

13.1 *Repeatability and Reproducibility*—Since the entire sample is subjected to the air-dry process, there is no opportunity to compare data for repeatability in the air-drying process. However, repeatability data between duplicate subsamples at each reduction and division stage can be obtained by analyzing duplicate cuts. Reproducibility data for total moisture is contingent with the taking of two or more samples simultaneously. Within the above limitations the repeatability and reproducibility are listed below:

13.1.1 *Repeatability*—Results of duplicate moisture determinations carried out on the same sample in the same laboratory by the same operator using the same equipment should not differ by more than 0.30 %. (These limits may not be applicable to low rank coals; additional data are required to establish such limits.)

13.1.2 *Reproducibility*—The mean of results of duplicate moisture determinations carried out by different laboratories on different portions of the sample should not differ by more than 0.50 %.

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TABLE 1 Number and Weight of Increments for General-Purpose Sampling Procedure

Top Size	⅜ in. (16 mm)	2 in. (50 mm)	6 in. (150 mm)
Mechanically Cleaned Coal			
Minimum number of increments	15	15	15
Minimum weight of increments, lb	2	6	15
Minimum weight of increments, kg	1	3	7
Raw (Uncleaned Coal)			
Minimum number of increments	35	35	35
Minimum weight of increments, lb	2	6	15
Minimum weight of increments, kg	1	3	7

APPENDIX

X1. ACCURACY DETERMINATION OF MOISTURE IN COAL

X1.1 The accurate determination of moisture in coal of various ranks has long been a subject of discussion and investigation. As has been pointed out in the referenced investigations, one of the major difficulties in assigning absolute merit to a particular procedure is the multiplicity of conditions under which water exists in coals and the difficulties involved in obtaining sharp separation and distinction among these conditions.

X1.2 As stated by Gauger, "Water recoverable from coal is obtained from the following sources: (*1*) decomposition of organic molecules (sometimes called combined water), (*2*) surface adsorbed water, (*3*) capillary condensed water, (*4*) dissolved water, and (*5*) water of hydration of inorganic constituents of the coal."

X1.3 Brown further refers to (*1*) "Free" or "adherent" Moisture (essentially surface adsorbed) possessing the physical properties of ordinary water, (*2*) physically bound or "inherent" moisture of vapor pressure lowered by the small diameter of the pores of the coal structure in which it is absorbed, and (*3*) chemically bound water of hydration or "combined" water.

X1.4 It becomes apparent, then, that "total moisture" in principle represents a measurement of all of the water not chemically combined.

X1.5 Traditionally, thermal treatment has provided the most commonly used basis for attempting to separate the nonchemically bound water from coal, and the measurement is normally made by weight loss. The "absolute" separation of adsorbed moisture without loss of a portion of chemically bound water is most difficult, if not impossible. The separation is particularly difficult in geologically younger coals of lower rank. Investigators have shown that the amount of water extracted is a function of both temperature and time. The problem is further compounded by the susceptibility of certain coals to oxidation.

X1.6 Because of such problems, investigators have proposed a number of schemes to satisfy their particular objectives in the measurement of water associated with coal. These include:

X1.6.1 Heating in air at varying temperatures and for varying time intervals.

X1.6.2 Heating in inert atmospheres (nitrogen, helium, argon, etc.).

X1.6.3 Separation of water by distillation with benzene, toluene, xylene, kerosine, etc.

X1.6.4 Measurement of water by such chemical methods as Karl Fischer titrations.

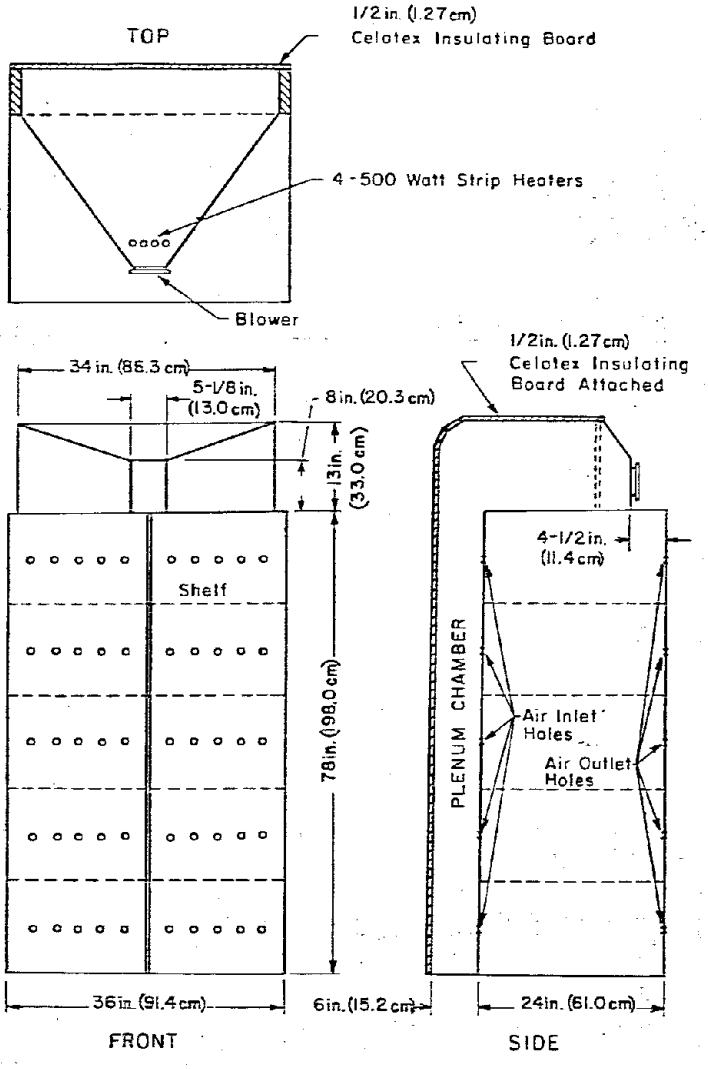


FIG. 1 Air Drying Oven

APPENDIX D

STANDARD TEST METHOD FOR INFILTRATION RATE OF SOILS IN FIELD USING DOUBLE RING-INFILTROMETERS

Designation: D 3385 – 75

Standard Test Method for INFILTRATION RATE OF SOILS IN FIELD USING DOUBLE-RING INFILTROMETERS[1]

This Standard is issued under the fixed designation D 3385; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

INTRODUCTION

This method describes a procedure for field measurement of the infiltration rate of soils. Infiltration rate is defined as a soil characteristic determining or describing the maximum rate at which water can enter the soil under specified conditions, including the presence of an excess of water. Infiltration rates have application to such problems as erosion rates, leaching and drainage efficiencies, irrigation, water spreading, rainfall runoff, and evaluation of potential septic-tank disposal fields, among other applications.

Rates determined by ponding of large areas are considered the most reliable method of determining infiltration rate but the high cost makes the infiltrometer-ring method more feasible economically. The infiltration rate is controlled by the least-permeable zone in the subsurface soils. The double-ring infiltrometer is used to help prevent divergent flow in layered soils by providing an outer water barrier to encourage only vertical flow from the inner ring. Many other factors[2] affect the infiltration rate in addition to the soil structure, for example, the condition of the soil surface, the moisture content of the soil, the chemical and physical nature of the soil and of the applied water, the head of applied water, and the temperature of the water. Thus, tests made at the same site are not likely to give identical results and the rate measured by the procedure described in this method is primarily for comparative use. Some aspects of the test, such as the length of time the tests should be conducted and the head of water to be applied, must depend upon the experience of the user, the purpose for testing, and the kind of information that is sought.

1. Scope

1.1 This method describes a procedure for field measurement of the rate of infiltration of water into soils using double-ring infiltrometers. This method is difficult to use and the resultant data may be unreliable in very coarse or heavy clay soils, or in frozen or highly fractured ground. This test may be conducted at the surface or at given depths in pits, and on bare soil or with vegetation in place, depending upon the conditions for which infiltration rates are desired.

2. Summary of Method

2.1 Infiltration rates can be measured in the field using double-ring infiltrometers. Two open cylinders, one inside the other, are driven into the ground and partially filled with water which is then maintained at a constant level. The volume of water added to maintain the water level constant is the measure of the volume of water that infiltrates the soil. The volume infiltrated during timed intervals is converted to an infiltration velocity, usually expressed in inches per hour (or centimetres per hour). The maximum infiltration velocity is

[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock for Engineering Purposes.

Current edition approved Feb. 28, 1975. Published April 1975.

[2] Johnson, A. I., *A Field Method for Measurement of Infiltration*, U.S. Geological Survey Water-Supply Paper 1544-F, 1963, pp. 4–9.

equivalent to the infiltration rate.

3. Apparatus

3.1 *Infiltrometer Rings*—Cylinders approximately 20 in. (50 cm) high and with different diameters of approximately 12 and 24 in. (30 and 60 cm). Cylinders can be made of ⅛-in. (3-mm), hard-alloy, aluminum sheet or other material sufficiently strong to withstand hard driving, with the bottom edge beveled from the outside to the inside edge (see Fig. 1). The beveled edges should be kept sharp. Larger rings can be calibrated in accordance with Table 1 and used, if economically feasible.

3.2 *Driving Caps*—Disks, of ½-in. (13-mm) aluminum alloy with centering pins around the edge. The diameters of the disks should be slightly larger than those of the infiltrometer rings.

3.3 *Driving Equipment*—A 12-lb (5.5-kg) mall or sledge and a 2 or 3-ft (60 or 90-cm) length of wood approximately 2 by 4 in. (5 by 10 cm) or 4 by 4 in. (10 by 10 cm).

3.4 *Depth Gage*—A hook gage, steel tape or rule, or length of heavy wire pointed on one end, for use in measuring and controlling the depth of water (head) in the infiltrometer ring.

3.5 *Splash Guard*—Several pieces of rubber sheet or burlap 6 in. (15 cm) square.

3.6 *Rule or Tape*—A 6-ft (2-m) steel tape or 1-ft (30-cm) steel rule.

3.7 *Metal Tamp*—Pipe, 14 in. (35 cm) long, with 6-in. (15-cm) length of 1 in. (25 mm) wide, ¼ in. (6 mm) thick steel strap welded to the end.

3.8 *Shovels*—One long-handled shovel and one trenching spade.

3.9 *Hand Auger*—Orchard-type auger with 3 in. (75 mm) in diameter, 9-in. (225-mm) long barrel and a rubber-headed tire hammer for knocking sample out of the auger.

3.10 *Water Containers*—One 50-gal (190-litre) barrel for the main water supply; one 1000-ml graduated cylinder or a graduated Mariotté tube for measurement of water during the test; a 12-qt (12.7-litre) pail for initial filling of the infiltrometer; and a length of rubber hose to siphon water from the barrel (see Fig. 2).

3.11 *Water Supply*—Preferably, water of the same quality and temperature as that involved in the problem being examined.

3.12 *Stop Watch.*

3.13 *Level*—A carpenter's level or bull's-eye (round) level.

3.14 *Recording Materials*—Record books and graph paper, 20 by 20 lines/in. (8 by 8 lines/cm), or special forms with graph section (see Fig. 3).

4. Test Site

4.1 The test requires an area of approximately 10 by 10 ft (3 by 3 m), accessible by a truck.

4.2 The test site should be as nearly level as possible, or a level surface should be prepared.

4.3 The test may be set up in a pit if infiltration rates are desired at depth rather than at the surface.

4.4 Avoid sites where interference with test equipment is possible, such as sites near children or in pastures with livestock.

5. Procedure

5.1 *Driving Infiltration Rings with a Sledge:*

NOTE 1—Driving rings with a jack is the preferred method (see 5.2).

5.1.1 Place driving cap on the outer ring and center it thereon. Place the wood block (see 3.3) on the driving cap.

5.1.2 Drive the outer ring 6 in. (15 cm) into the soil with blows of a heavy sledge on the wood block. Use blows of medium force to prevent fracturing of the soil surface. Move the wood block around the edge of the driving cap every one or two blows so that the ring will penetrate the soil uniformly.

5.1.3 Center the smaller ring inside the larger ring and drive to a depth of 2 in. (5 cm), using same technique as in 5.1.2.

5.1.4 Check rings with the level, correcting the attitude of rings to be vertical, as needed.

5.2 *Driving Infiltration Rings with Jacks:*

5.2.1 Use a heavy jack and a truck to drive the rings as an alternative to the sledge method (see 5.1).

5.2.2 Center the wood block across the driving cap of the ring. Center a jack on the wood block. Place the top of the jack and the assembled items vertically under the previously positioned end of a truck body and apply force to the ring by means of the jack and truck reaction.

5.2.3 In heavy-textured soils, add additional

weight to the truck if needed to develop sufficient force to drive the ring.

5.2.4 Check rings with the level, correcting attitude of rings to be vertical, as needed.

5.3 *Tamping Disturbed Soil:*

5.3.1 If the soil is visibly disturbed more than ⅛ in. (3 mm) from the wall of the ring, reset the ring with less disturbance of the sample.

5.3.2 If the soil is visibly disturbed less than ⅛ in. (3 mm), tamp the disturbed soil adjacent to the inside wall of the ring with a metal tamp until the soil is as firm as it was prior to the disturbance.

5.4 *Maintaining Water Level:*

5.4.1 Install a depth gage or a Mariotté tube for each infiltrometer ring to assist the investigator visually in maintaining a constant head. For a depth gage, use a steel tape or rule, if the soils have high permeability. Use a hook gage, simple-point gage, or Mariotté tube if the soils have low permeability.

5.4.2 Install the gages to be used near the center of the center ring and also in the annular space midway between the two rings.

5.4.3 Cover the soil surface within the center ring and between the two rings with splash guards (6-in. (15-cm) square pieces of burlap or rubber sheet) to prevent erosion of the soil when the initial water supply is poured into the rings.

5.4.4 Use a pail to fill both rings with water to the same desired depth in each ring. Do not record this initial volume of water. Remove the splash guards.

5.4.5 Choose a head of at least 1 in. (2.5 cm) and not greater than 6 in. (15 cm) and maintain the water level at the selected head as near as possible throughout the test. The head is selected on the basis of the permeability of the soil, the higher heads being required for lower permeability soils.

5.5 *Measurements:*

5.5.1 Record all volumes of water that are added to maintain a constant head during a timing interval.

5.5.2 For average soils, record the volume of water used at intervals of 15 min for the first hour, 30 min for the second hour, and 60 min during the remainder of a period of at least 6 h, or until a maximum rate is obtained.

5.5.3 For high-permeability materials, take early readings more frequently. The exact schedule of readings can be determined only through experience.

5.5.4 For low-permeability materials, use a longer test interval, possibly 24 h, or more. Again, the exact schedule is determined through experience.

5.5.5 Place the driving cap or some other covering over the rings during the intervals between water measurements to minimize evaporation.

5.5.6 Upon completion of the test, remove the rings from the soil assisted by light hammering on the sides with a rubber hammer.

6. Calculations

6.1 Convert the volume of water used during each measured time interval into the depth of water per unit of time in inches (or centimetres) per hour. Table 1 can be used to convert volume measurements into equivalent depths of water by multiplying the volume of water used during the time interval by the area factor shown in the table. The infiltration velocity is obtained by dividing the depth of water by the time interval, in hours.

6.2 Make these calculations for the inner ring, the annular space, and both rings combined.

7. Report

7.1 Record the infiltration velocities in a record book or on a report form (see Fig. 3). Record the depth to the water table, if known, and a description of the soils found between the rings and the water table, or at least to a 20-ft (6-m) depth. Plot the data on the cross-sectioned part of the report form (see Fig. 3).

7.1.1 Record the temperature and the pH of the water used in the test. If available, a full analysis of the water should be recorded also.

7.2 The rate for the inner ring should be the value used if the rates for both rings and annular space differ. The difference in rates is due to divergent flow.

NOTE 2—Although not considered a required part of the test, the determination of the moisture pattern in the moistened soil beneath the infiltration rings commonly provides information useful in interpreting the movement of water through any particular soil profile. For example, water movement horizontally may be caused by lower-permeability layers and will result in a lateral spreading of the wetted zone. Thus, the exploration of the soils below an infiltration test in an unfamiliar area can determine the subsurface

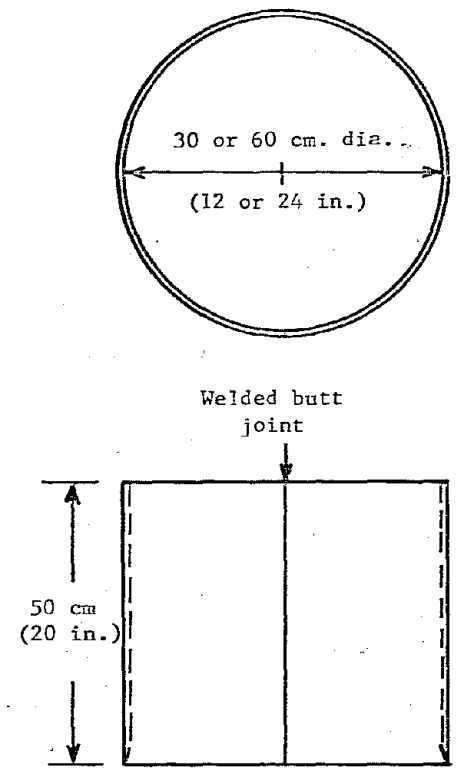
conditions that may affect the test and later applications of the data.

If the investigator chooses to make such a study, dig a trench so that one wall of the trench passes along the center line of the former position of the rings. Orient the trench so that the other wall is illuminated by the sun, if the day is sunny. If feasible, dig the trench large enough to include all of the newly moistened area. Collect samples from the shaded wall of the trench for determination of moisture content. If preferred, an auger, such as the orchard barrel type, may be used to determine the approximate outline of the moistened area below the rings and to collect samples for moisture content.

Plot the visibly-moistened area on graph paper or on the cross-section part of the report form (see Fig. 3). If samples were collected and moisture contents were determined, the contour of moisture content also can be plotted on the graph.

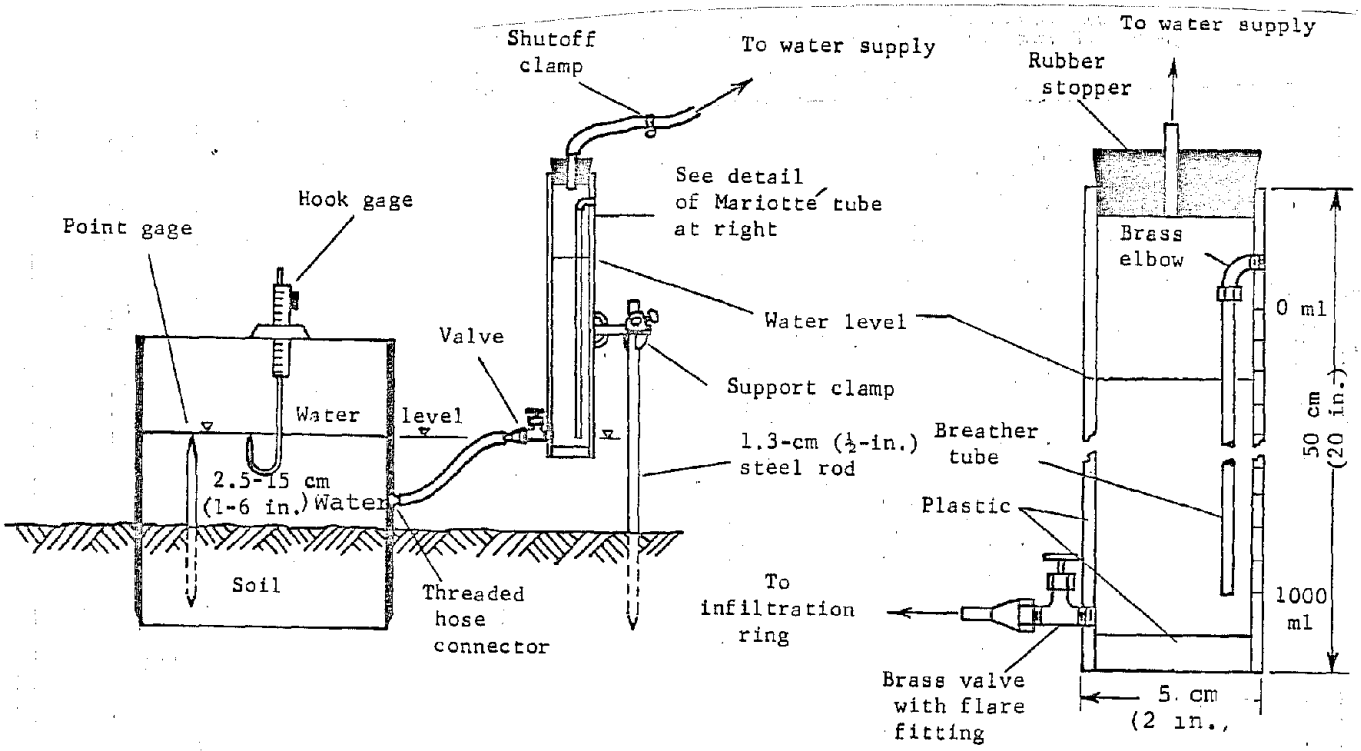
TABLE 1 Data for Double-Ring Infiltrometers

Diameter of Ring(s), in. (mm)	Area of Annular Space, in^2 (cm^2)	Area of Ring, in^2 (cm^2)	Volume of Water Providing a 1-in. (2.5-cm) Depth, ml	Multiplication Factors to Convert Volume of Water Used in ml to Depth of Water in in. (cm)
12 and 24 (30 and 60)	339.3 (2189.2)	...	5561	1.80×10^{-4} (4.57×10^{-4})
12 (30)	...	113.1 (729.7)	1854	5.39×10^{-4} (13.70×10^{-4})
24 (60)	...	452.4 (2918.9)	7415	1.35×10^{-4} (3.43×10^{-4})



NOTE—The material shall be a ⅛-in. (3-mm) aluminum-alloy sheet or material of similar strength.

FIG. 1 Infiltrometer Construction.



NOTE—Center ring has been eliminated for simplification of the illustration.

FIG. 2 Ring Installation and Mariotté Tube Details.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103, which will schedule a further hearing regarding your comments. Failing satisfaction there, you may appeal to the ASTM Board of Directors.

Joe Smith — Data for inner ring

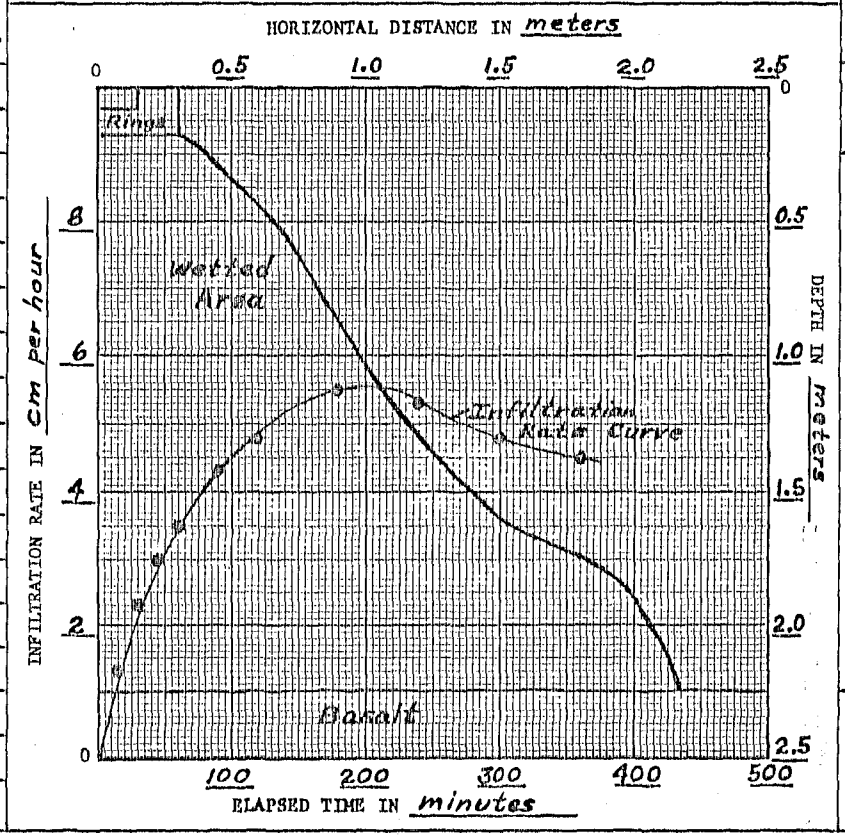
Date 10-14-55 Technician _____ Method 30 & 60 cm Double-ring Infiltrometer

Water Level 5.2 m Site Lost River alluvium pH: 8.0 PROJECT NRTS

WATER HEAD: 4cm TEMP: 62°F / INFILTRATION RATE LOCATION IDAHO

Elapsed time, min	Quantity of water, ml	Infiltration rate, cm/hr
15	240	1.3
30	420	2.3
45	550	3.0
60	640	3.5
90	1560	4.3
120	1750	4.8
180	4000	5.5
240	3870	5.3
300	3500	4.8
360	3280	4.5
	for Ring	
DATA		
INNER	(30 cm)	

CROSS-SECTION OF MOIST AREA AND GRAPH OF INFILTRATION RATE



Soil profile description
Soil, sandy, thin grass stand
Sand, v.f. to f., dry, loose
Basalt, dense surface

FIG. 3 Report Form for Infiltration Test With Sample Data.

APPENDIX E

STANDARD METHOD OF SIEVE ANALYSIS OF COAL

Designation: D 410 – 38 (Reapproved 1976)

American National Standard K20.8-1970
American National Standards Institute

Standard Method of SIEVE ANALYSIS OF COAL[1]

This Standard is issued under the fixed designation D 410; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

NOTE—An editorial change in 6.3 was made in June 1947, and the title was changed in July 1969.

1. Scope

1.1 This method for sieve analysis is applicable to all coal except anthracite, powdered coal as used in boiler plants, and crushed coal as charged into coke ovens.

NOTE—The values stated in U.S. customary units are to be regarded as the standard.

2. Apparatus

2.1 Sieves of the following series shall be used conforming to ASTM Specification E 11, for Wire-Cloth Sieves for Testing Purposes[2], or ASTM Specification E 323, for Perforated-Plate Sieves for Testing Purposes[2].

Round-Hole Screens:

8-in.	(...)	1½-in.	(37.5-mm)
6-in.	(...)	1¼-in.	(31.5-mm)
5-in.	(125-mm)	1-in.	(25.0-mm)
4-in.	(100-mm)	¾-in.	(19.0-mm)
3-in.	(75-mm)	½-in.	(12.5-mm)
2½-in.	(63-mm)	⅜-in.	(9.5-mm)
2-in.	(50-mm)		

Wire-Cloth Sieves with Square Openings:

4.75-mm	(No. 4)
2.36-mm	(No. 8)
1.18-mm	(No. 16)
600-μm	(No. 30)
300-μm	(No. 50)
150-μm	(No. 100)
75-μm	(No. 200)

3. Time of Sampling

3.1 Sample coal when it is being loaded into or unloaded from railroad cars, ships, barges, or wagons, or when discharged from supply bins, or from industrial railway cars, or grab buckets, or from any coal-conveying equipment, as the case may be. It is not feasible to collect representative samples for screen analysis from the surface of coal in piles or from loaded cars or bins.

4. Collection of Gross Sample

4.1 Collect increments regularly and systematically, so that the entire quantity of coal sampled will be represented proportionately in the gross sample, and with such frequency that a gross sample of the required amount shall be collected. The number of increments collected shall be not less than 20. When the coal is passing over a conveyor or down a chute, increments the full width and thickness of the stream of coal shall be taken either by stopping the conveyor and removing all coal from a transverse section of it, or by momentarily inserting a suitable container into the stream. If it is impracticable to collect increments the full width and thickness of the coal stream, increments shall be systematically collected from all portions of the stream.

5. Weight of Gross Sample

5.1 The weight of the gross sample collected shall conform to the following:

Run-of-mine coal	not less than 4000 lb (1800 kg)
Screened coal with upper limit larger than 4-in. (100-mm) round	not less than 4000 lb (1800 kg)
Coal smaller than 4-in. (100-mm) round	not less than 2000 lb (900 kg)
Coal smaller than 2-in.	not less than 1000 lb

[1] This method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke.

Current edition effective Sept. 1, 1938. Originally issued 1935. Replaces D 410 – 35 T.

[2] *Annual Book of ASTM Standards*, Part 26.

(50-mm) round	(450 kg)
Coal smaller than 1-in. (25-mm) round	not less than 500 lb (215 kg)
Coal smaller than ½-in. (12.5-mm) round	not less than 100 lb (45 kg)

6. Reduction of Gross Sample

6.1 *Coal Larger than 1-in. (25-mm) Round*—Screen without mixing or dividing.

6.2 *Coal Smaller than 1-in. Round*—Divide in amount to not less than 125 lb (56.5 kg) by riffling or by arranging it in a long, flat pile and successively halving it or quartering it by the alternate-shovel method as follows: Take successive shovelfuls in passing around the pile (advancing a distance equal to the width of the shovel for each shovelful), and retain alternate shovelfuls or every fourth shovelful for the sample.

6.3 *Coal Smaller than ½-in. (12.5-mm) Round*—Divide to not less than 25 lb (11.4 kg) by passing it through a riffle or equally accurate dividing device, or by hand-quartering as described in ASTM Method D 346, Sampling Coke for Analysis.[2]

6.4 *Coal Smaller than No. 4 Sieve*—Divide to not less than 2 lb (1000 g) by riffling or hand-quartering.

6.5 *Coal Smaller than No. 8 Sieve*—Divide to not less than 1 lb (500 g) by riffling or hand-quartering.

7. Drying Sample

7.1 In case the coal is wet, the sample may be tested on sieves 1 in. (25 mm) round and larger, without drying, but dry the sample of coal smaller than 1-in. round (divided in amount to 125 lb (56.5 kg), as described in Section 6.2), sufficiently to remove surface moisture which causes small particles to cling to the larger pieces. In cases of lignite, subbituminous, and high volatile *C* bituminous coals, take care not to over-dry and cause weathering of the coal.

8. Sieve Analysis

8.1 Accurately weigh the sample before screening. Starting with the largest sieve, screen the sample in such amounts as will allow the pieces to be in direct contact with the openings at the completion of the screening of each amount. Determine the smallest sieve through which all of the sample passes by actual test, in accordance with 8.2, 8.3, and 8.4.

8.2 *Coal Larger than 2½-in. (63-mm) Round*—Try by hand pieces of coal not passing readily through sieves 2½-in. round and larger to see if they will pass through the openings in any position. Do not shake sieves 2½-in. round and larger except for whatever jiggling may be necessary to clear the sieves of fine coal.

8.3 *Coal Smaller than 2½-in. Round*—Test coal passing the 2½-in. round sieve with sieves down to and including 1-in. (25-mm) round as follows: Move the sieve horizontally a distance of about 8 in. at just sufficient rate to cause the pieces of coal to tumble or roll on the sieve. Stop the motion of the sieve without impact. After ten such shakes (five in each direction), screening of the increment shall be considered complete.

8.4 *Coal Smaller than 1-in. Round*—Coal passing the 1-in. round and smaller sieves may be weighed and then divided in amount as provided in Section 6 and then dried as provided in Section 7. Shake sieves smaller than 1-in. round gently with a reciprocating horizontal motion until practically no more coal will pass through the openings. When both 150-μm (No. 100) and 75-μm (No. 200) sieves are used, use the latter first in order to facilitate screening.

9. Report

9.1 Report the sieve analysis results to the nearest 0.1 percent as follows:

	Percent
Retained on .... in. round	0
Retained on .... in. round, passing .... in. round	.....
.....	.....
.....	.....
.....	.....
Retained on No ...., passing .... in. round	.....
Retained on No ...., passing No	.....
Passing No.	.....
Total	.....

9.2 If the sum of the weights shows a loss of over 2 percent, reject the analysis and make another test.

APPENDIX F

STANDARD METHOD FOR ASH IN THE ANALYSIS SAMPLE OF COAL AND COKE

Designation: D 3174 – 73

Standard Test Method for ASH IN THE ANALYSIS SAMPLE OF COAL AND COKE[1]

This Standard is issued under the fixed designation D 3174; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers determination of the ash content in the analysis sample of coal or coke as prepared in accordance with ASTM Method D 2013 or Method D 346. The results obtained can be applied as the ash in the proximate analysis, Method D 3172, and in the ultimate analysis, Method D 3176. For the determination of the constituents in ash, reference is made to Method D 2795.

2. Applicable Documents

2.1 *ASTM Standards:*

D 346 Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 388 Specification for Classification of Coals by Rank[2]

D 2013 Preparing Coal Samples for Analysis[2]

D 2795 Test for Analysis of Coal and Coke Ash[2]

D 3172 Proximate Analysis of Coal and Coke[2]

D 3173 Test for Moisture in the Analysis Sample of Coal and Coke[2]

D 3176 Ultimate Analysis of Coal and Coke[2]

3. Summary of Method

3.1 Ash is determined by weighing the residue remaining after burning the coal under rigidly controlled conditions of sample weight, temperature, time, and atmosphere.

4. Apparatus

4.1 *Gas or Electric Muffle Furnace for Coal*—For determination of ash of coal, the furnace shall have an adequate air circulation and be capable of having its temperature regulated between 700 and 750°C.

4.2 *Gas or Electric Muffle Furnace or Meker Burner for Coke*—For determination of ash of coke, the furnace shall have an adequate air circulation and be capable of having its temperature regulated at the prescribed temperature(s) of 750 and 950°C.

4.3 *Porcelain Capsules*, about ⅞ in. (22 mm) in depth, and 1¾ in. (44 mm) in diameter, or similar shallow dishes or platinum crucibles.

5. Procedure for Coal

5.1 Transfer approximately 1 g of the sample to a weighed porcelain capsule with a spoon or spatula and quickly establish the weight to the nearest 0.1 mg. An alternative way is to use the dried coal from the moisture determination Method D 3173. Place the porcelain capsule containing the sample in a cold muffle furnace, or on a hearth at a low temperature, and gradually heat to redness at such a rate as to avoid mechanical loss from too rapid expulsion of volatile matter. Finish the ignition to constant weight (±0.001 g) at a temperature between 700 and 750°C. Cool in a desiccator over desiccant, and weigh as soon as cold.

NOTE 1—Ash results obtained by this method differ in composition from the inorganic constituents present in the original coal. Incineration causes an expulsion of water from the clays and calcium sulfate, of carbon dioxide from carbonates, and the conversion of iron pyrites into ferric oxide. Each of these reactions involves a loss in weight from the original inorganic material. Formulas and references for correcting ash results to a mineral matter basis are

[1] This method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke.

Current edition approved April 27, 1973. Published July 1973.

[2] *Annual Book of ASTM Standards*, Part 26.

listed in Method D 388, Section 7. Also, see the report on Fixed Carbon and Ash.[3]

NOTE 2—Difficulty may be experienced in securing satisfactory check determinations of ash in the same or different laboratories for coals unusually high in calcite and pyrite. This is caused by varying amounts of sulfate sulfur being retained in the ash. When such difficulty is encountered, or when coals of relatively high ash content whose mineral matter composition is unknown are encountered, the ash should be determined by the following modified procedures:

(*a*) Place the capsules containing the dried coal from the moisture determination in a cold muffle furnace and heat gradually so that the temperature reaches 500°C in 1 h, and 750°C in 2 h. Heat to constant weight at 750°C. By this means, pyritic sulfur will be oxidized and expelled before the calcite is decomposed. An ample supply of air in the muffle must be assured at all times to ensure complete oxidation of the pyritic sulfur and to remove the SO_2 formed.

(*b*) The modified procedure described in Paragraph (*a*) should be adequate for determining ash in troublesome commercial samples. However, samples may be encountered in certain special studies whose ash values are quite high and whose mineral matter contains much greater than normal amounts of calcite and pyrite. In such cases sulfate sulfur should be determined on the ash obtained by the modified cold muffle method and the value properly corrected, or the Parr[4] sulfated ash method as modified by Rees[5] should be used.

6. Procedure for Coke

6.1 Weigh the sample as described in 5.1.

6.2 Place the capsules containing the coke sample in a muffle furnace or over a burner, and heat to redness at such a rate as to avoid mechanical loss. Finish the ignition to constant weight (±0.001 g) at a temperature not exceeding 950°C. Cool in a desiccator and weigh.

6.3 For those samples containing an exceptionally low ash, a 5-g sample weight is permissible.

7. Calculations

7.1 Calculate the ash percent in the analysis sample as follows:

$$\text{Ash in analysis sample, \%} = [(A - B)/C] \times 100$$

where:

A = weight of capsule and ash residue, g,
B = weight of empty capsule, g, and
C = weight of analysis sample used, g.

8. Precision

8.1 The following criteria should be used for judging the acceptability of results:

8.1.1 *Repeatability*—Duplicate results by the same laboratory should not be considered suspect unless they differ by more than the following percentages:

No carbonates present	0.2
Carbonates present	0.3
Coals with more than 12 % ash, containing carbonates and pyrites	0.5

8.2.1 *Reproducibility*—Results submitted by two or more laboratories should not be considered suspect unless they differ by more than the following percentages:

No carbonates present	0.3
Carbonates present	0.5
Coals with more than 12 % ash, containing carbonates and pyrites	1.0

[3] Report on Fixed Carbon and Ash, Proceedings, Am. Soc. Testing Mats., Vol XIV, Part I (Committee Reports), 1914, p. 426.

[4] Parr, S. W., "Chemical Study of Illinois Coal," Bulletin No. 3, p 35, Illinois Coal Mining Investigations, State Geological Survey, Urbana, Ill. (1916).

[5] Rees, O. W., "Determining Ash in High Carbonate Coals, Study of the Modified Method," *Industrial and Engineering Chemistry, Analytical Edition*, Vol 9, 1937, pp. 307-309.

APPENDIX G

STANDARD TEST METHODS FOR TOTAL SULFUR IN THE ANALYSIS SAMPLE OF COAL AND COKE

Designation: D 3177 – 75

Standard Test Methods for TOTAL SULFUR IN THE ANALYSIS SAMPLE OF COAL AND COKE[1]

This Standard is issued under the fixed designation D 3177; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 These methods cover three alternative procedures for the determination of total sulfur in samples of coal and coke. Sulfur is included in the ultimate analysis of coal and coke.

1.2 The procedures appear in the following order.

	Sections
Method A—Eschka Method	5 to 8
Method B—Bomb Washing Method	9 to 11
Method C—High-Temperature Combustion Method	12 to 16

2. Applicable Documents

2.1 *ASTM Standards:*

D 346 Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 1193 Specification for Reagent Water[3]

D 2013 Preparing Coal Samples for Analysis[2]

D 2015 Test for Gross Calorific Value of Solid Fuel by the Abiabatic Bomb Calorimeter[2]

D 3173 Test for Moisture in the Analysis Sample of Coal[2]

D 3176 Ultimate Analysis of Coal and Coke[2]

D 3180 Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]

3. Summary of Methods

3.1 *Eschka Method*—A weighed sample and Eschka mixture are ignited together, and the sulfur is precipitated from the resulting solution as barium sulfate ($BaSO_4$). The precipitate is filtered, ashed, and weighed.

3.2 *Bomb Washing Method*—Sulfur is precipitated as $BaSO_4$ from oxygen-bomb calorimeter washings, and the precipitate is filtered, ashed, and weighed.

3.3 *High-Temperature Combustion Method*—A weighed sample is burned in a tube furnace at a temperature of 1350°C in a stream of oxygen. The sulfur oxides and chlorine formed are absorbed in a hydrogen peroxide (H_2O_2) solution yielding hydrochloric (HCl) and sulfuric (H_2SO_4) acids. The total acid content is determined by titration with sodium hydroxide (NaOH), and the amount of sodium chloride (NaCl) resulting from the titration of the HCl is converted to NaOH with a solution of mercuric oxycyanide (Hg(OH)CN). This sodium hydroxide is determined titrimetrically and used to correct the sulfur value which is equivalent to the amount of H_2SO_4 formed during combustion of the coal.

4. Sample

4.1 The sample shall be the material pulverized to pass No. 60 (250-μm) sieve in accordance with Method D 2013 or Method D 346.

4.2 A separate portion of the analysis sample should be analyzed for moisture content in accordance with Method D 3173, so that calculation to other than the as determined basis can be made.

4.3 Procedures for converting as determined sulfur values obtained from the analysis sample to other bases are described in Method D 3176 and Method D 3180.

4.4 Standard Reference Material (SRM)

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[1] These methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke.

Current edition approved May 30, 1975. Published August 1975. Originally published as D 3177 – 73. Last previous edition D 3177 – 73.

[2] *Annual Book of ASTM Standards*, Part 26.

[3] *Annual Book of ASTM Standards*, Part 31.

1631-Sulfur in Coal[4] consists of three different low-volatile coal samples, each of which has a certified sulfur content. Sulfur values obtained by analyzing these coals, using any of the three methods described in this standard, may be used for checking the accuracy of analytical results.

ALTERNATIVE PROCEDURES

Method A—Eschka Method

5. Apparatus

5.1 *Gas (Note 1) or Electric Muffle Furnace, or Burners*, for igniting the sample with the Eschka mixture and for igniting the barium sulfate ($BaSO_4$).

NOTE 1—Gas may contain sulfur compounds.

5.2 *Crucibles or Capsules*—Porcelain capsules, ⅞ in. (22 mm) in depth and 1¾ in. (44 mm) in diameter, or porcelain crucibles of 30-ml capacity, high or low form, or platinum crucibles of similar size shall be used for igniting the sample with the Eschka mixture. Porcelain, platinum, alundum, or silica crucibles of 10 to 15-ml capacity, shall be used for igniting the $BaSO_4$.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Available Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type IV conforming to Specification D 1193.

6.3 *Barium, Chloride Solution (100 g/litre)*—Dissolve 100 g of barium chloride ($BaCl_2 \cdot 2H_2O$) and dilute to 1 litre with water.

6.4 *Bromine Water (Saturated)*—Add an excess of bromine to 1 litre of water.

6.5 *Eschka Mixture*—Thoroughly mix 2 parts by weight of light calcined magnesium oxide (MgO) with 1 part of anhydrous sodium carbonate (Na_2CO_3). Both materials should be as free as possible from sulfur.

6.6 *Hydrochloric Acid (1 + 1)*—Mix equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) and water.

6.7 *Hydrochloric Acid (1 + 9)*—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 9 volumes of water.

6.8 *Methyl Orange Indicator Solution (0.2 g/litre)*—Dissolve 0.02 g of methyl orange in 100 ml of hot water and filter.

6.9 *Sodium Carbonate, Saturated Solution*—Dissolve approximately 60 g of crystallized sodium carbonate ($Na_2CO_3 \cdot 10H_2O$) or 22 g of anhydrous sodium carbonate (Na_2CO_3) in 100 ml of water, using a sufficient excess of Na_2CO_3 to ensure a saturated solution.

6.10 *Sodium Hydroxide Solution (100 g/litre)*—Dissolve 100 g of sodium hydroxide (NaOH) in 1 litre of water. This solution may be used in place of the Na_2CO_3 solution.

7. Procedure

7.1 *Preparation of Sample and Mixture*—Thoroughly mix on glazed paper approximately 1 g of the sample, weighed to nearest 0.1 mg, and 3 g of Eschka mixture. The amount of sample to be taken will depend on the amount of $BaCl_2$ solution required in accordance with 7.3. Transfer to a porcelain capsule or porcelain crucible, or a platinum crucible, and cover with about 1 g of Eschka mixture.

7.2 *Ignition*—Heat the crucible over an alcohol, gasoline, or gas flame as described in 7.2.1, or in a gas or electrically heated muffle as described in 7.2.2 for coal and in 7.2.3 for coke. The use of artificial gas for heating the sample and the Eschka mixture is permissible only when the crucibles are heated in a muffle.

7.2.1 Heat the crucible, placed in a slanting position on a triangle, over a very low flame to avoid rapid expulsion of the volatile matter which tends to prevent complete absorption of the products of combustion of the sulfur. Heat the crucible slowly for 30 min, gradually increase the temperature, and occasionally stir

[4] Available from the Office of Standard Reference Materials, Room B314, Chemistry Bldg., National Bureau of Standards, Washington, D.C. 20234.

[5] "Reagent Chemicals, American Chemical Society Specification," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."

until all black particles have disappeared, which is an indication of the completeness of the procedure.

7.2.2 *For Coal*—Place the crucible in a cold muffle and gradually raise the temperature to 800 ± 25°C in about 1 h. Maintain this maximum temperature until on stirring all black particles have disappeared (about 1 ½ h).

7.2.3 *For Coke*—Place the crucible in a warm muffle (about 200°C) and gradually raise the temperature to 800 ± 25°C in about 30 min. Maintain this maximum temperature until on stirring all black particles have disappeared.

7.3 *Subsequent Treatment*—Remove the crucible and empty the contents into a 200-ml beaker and digest with 100 ml of hot water for ½ to ¾ h, while stirring occasionally. By decantation, filter, and thoroughly wash the insoluble matter with hot water. After several washings in this manner, transfer the insoluble matter to the filter and wash five times with hot water, keeping the mixture well agitated. Treat the filtrate, amounting to about 250 ml, with 10 to 20 ml of saturated bromine water, make slightly acid with HCl, and boil to expel the liberated bromine. Make just neutral to methyl orange with NaOH or Na_2CO_3 solution; then add 1 ml of HCl (1 + 9). Boil again and add slowly from a pipet, while stirring constantly, 10 ml or more of $BaCl_2$ solution. The $BaCl_2$ solution must be in excess. If more than 10 ml of $BaCl_2$ solution is required, reduce the weight of sample to about 0.5 g and repeat the ignition and digestion. Continue boiling for 15 min and allow to stand for at least 2 h, or preferably overnight, at a temperature just below boiling. Filter through an ashless paper and wash with hot water until 1 drop of silver nitrate ($AgNO_3$) solution produces no more than a slight opalescence when added to 8 to 10 ml of filtrate.

7.3.1 Place the wet filter containing the precipitate of barium sulfate ($BaSO_4$) in a weighed platinum, porcelain, silica, or Alundum crucible, allowing a free access of air by folding the paper over the precipitate loosely to prevent spattering. Smoke the paper off gradually and at no time allow to burn with flame. After the paper is practically consumed, raise the temperature to approximately 925°C and heat to constant weight.

7.4 *Blanks and Corrections*—In all cases a correction must be applied. Either a reagent blank may be run exactly as described above, using the same amount of all reagents that were employed in the routine determination, or a more accurate correction may be made by analyzing a weighed portion of a standard sulfate using the prescribed reagents and operations. If the latter procedure is carried out once a week, or whenever a new supply of a reagent is used, for a series of solutions covering the approximate range of sulfur concentrations in the samples, it is only necessary to add to or subtract from the weight of $BaSO_4$ determined for the sample, the deficiency or excess found by the appropriate "check" determination. This procedure is more accurate than the simple reagent blank because, for the amounts of sulfur in question and the conditions of precipitation prescribed, the solubility error for $BaSO_4$, is probably the largest one to be considered. Barium sulfate is soluble[6] in acids and pure water, and the solubility limit is reached almost immediately on contact with the solvent. Hence, if very high-purity reagents are used or extra precaution is exercised, there may be no sulfate apparent in the "blank." In other words, the solubility limit for $BaSO_4$ has not been reached or at any rate not exceeded; consequently, some sulfate in the sample may remain in solution or redissolve.

8. Calculation

8.1 Calculate the sulfur content as follows:

$$\text{Sulfur, \% in the analysis sample} = \frac{(A-B) \times 13.738}{C}$$

where:

A = grams of $BaSO_4$ precipitated,
B = grams of $BaSO_4$ correction, and
C = grams of sample used.

Method B—Bomb Washing Method[7]

9. Reagents

9.1 *Purity of Reagents*—(See 6.1.)

9.2 *Purity of Water*—(See 6.2.)

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[6] *Journal of the American Chemical Society*, JACSA, Vol 32, 1910, p. 588; Vol 33, 1911, p. 829.

[7] Selvig, W. A., and Fieldner, A. C. "Check Determinations of Sulfur in Coal and Coke by the Eschka, Bomb-Washing and Sodium Peroxide Fusion Methods," *Industrial and Engineering Chemistry*, JECHA, Vol 29, 1927, pp. 729–733.

9.3 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH_4OH).

9.4 *Bromine Water (Saturated)*—(See 6.4.)

9.5 *Hydrochloric Acid (1 + 1)*—(See 6.6.)

9.6 *Sodium Carbonate Solution*—Dissolve 20.90 g of anhydrous sodium carbonate (Na_2CO_3) in water and dilute to 1 litre. The Na_2CO_3 should be previously dried for 24 h at 105°C.

9.7 *Wash Solution*—Dilute 1 ml of a saturated solution of methyl orange to 1 litre with water.

10. Procedure

10.1 *Ignition*—Sulfur is determined in the washings from the oxygen-bomb calorimeter following the calorimetric determination (Method D 2015). The type of bomb, amount of water in the bomb, oxygen pressure, and amount of sample taken shall be the same as specified under the calorimetric determination (Sections 4 to 8 of Method D 2015). The bomb shall stand in the calorimeter water for not less than 5 min after firing.

10.2 *Subsequent Treatment*—Remove the bomb from the calorimeter water and open the valve carefully so as to allow the gases to escape at an approximately even rate so the pressure is reduced to atmospheric in not less than 1 min. Bombs equipped with valves other than needle valves, such as compression valves, shall be provided with a device so the valve can be controlled to permit a slow and uniform release of the gases. Open the bomb and examine the inside for traces of unburned material or sooty deposit. If these are found, discard the determination. Wash carefully all parts of the interior of the bomb, including the capsule, with a fine jet of water containing methyl orange (9.7) until no acid reaction is observed. It is essential to wash through the valve opening in the case of bombs equipped with compression valves, or other types of valves with large openings, as considerable spray may collect in such valve openings.

10.3 Collect the washings in a 250-ml beaker and titrate with standard sodium carbonate solution (9.6) to obtain the "acid correction" for the heating value, as specified under the calorimetric determination D 2015. Adjust the pH to 5.5 to 7.0 with dilute NH_4OH, heat the solution to boiling, and filter through a qualitative paper. Wash the residue and paper thoroughly five or six times with hot water. To the filtrate and washings, amounting to about 250 ml, add 1 ml of saturated bromine water (9.4) and sufficient HCl (9.5) to make it slightly acid. Boil the solution to expel the excess bromine. Adjust the acidity, precipitate, and determine the sulfur as specified under the Eschka method, Sections 5 to 8.

Method C—High-Temperature Combustion Method[B]

11. Apparatus

11.1 *Tube Furnace*—Capable of heating a tube approximately 34-mm external diameter over a length of 150 mm to a temperature of 1350°C. It is heated electrically using either silicon carbide resistance rods or a resistance wire.

NOTE 2—Induction furnace techniques may be used provided it can be shown that they meet the precision requirements of Section 17.

11.2 *Combustion Tube*—Approximately 28-mm internal diameter with a 3-mm wall thickness and 750 mm in length, which is gas tight at working temperature. A high-temperature porcelain or zircon straight refractory tube has been found most efficient. It requires a silica (12.12) adaptor with a flared end that just fits inside the combustion tube and serves as an exit for the gases. Alternatively, the combustion may be carried out in a tapered end tube that is directly connected to the elbow of the fritted gas bubbler (12.8) or to a 10/30 standard-taper ground joint which is attached to a borosilicate glass right-angle bend. The temperature at the tapered end of the tube should be high enough to prevent condensation in the tube itself.

11.3 *Oxygen Cylinder*, fitted with pressure regulator and needle valve to control flow rate of oxygen.

11.4 *Flowmeter*, for measuring an oxygen flow rate of 300 ml/min.

[B] Based on the method of Mott, R. A., and Wilkinson, H. C., "Determination of Sulfur in Coal and Coke By the Sheffield High Temperature Method," *Fuel*, FUEL B, Vol 35, 1956, p. 6. This method is designed for the rapid determination of sulfur in coal and coke. It is not applicable to coals or coal density fractions that have been subjected to treatment with chlorinated hydrocarbons because of the potentially high acidity of the combustion gases.

11.5 *Sample Combustion Boats*—Iron-free, unglazed porcelain or zircon boats. A convenient size is 100 mm long, 19 mm wide, and 11 mm deep.

11.6 *Heat-Resistant Wire*, 1.5 mm thick with bent end to remove boats from combustion tube.

11.7 *Silica Pusher or Heat-Resistant Rod*, with a disk end for pushing the combustion boat into the hot zone. The pusher passes through a T-piece which is fitted into a rubber stopper at the inlet end of the combustion tube. The open end of the T-piece is sealed with a rubber tube or one-holed stopper to permit movement of the pusher and prevent escape of oxygen which enters at the side limb of the T. The rubber stopper or tube should be changed periodically to avoid leakage.

11.8 *Gas Absorption Bottles with Fritted Disk*, 125-ml capacity, for gas absorption. Fritted glass end porosity should be 15 to 40 μm. The bottle should be of such a diameter that the fritted end is covered by peroxide solution to a depth of at least 50 mm. The bottles are fitted, in a series of two per combustion tube, to the outlet end of a combustion tube. Alternatively a single narrow gas absorber may be used so that the fritted bubbler is covered to a depth of at least 90 mm.

11.9 *Vacuum Regulating Bottle*, containing mercury with an open-ended tube dipping into it.

11.10 *U-tube*, packed with soda-asbestos.

11.11 *Vacuum Source.*

11.12 *Silica Adaptor*, 300 mm long by 8 mm in outside diameter and flared at one end to 26 mm.

12. Reagents

12.1 *Purity of Reagents*—(See 6.1.)

12.2 *Purity of Water*—(See 6.2.)

12.3 *Aluminum Oxide* (Al_2O_3), finely divided and dried at 1350°C.

12.4 *Hydrogen Peroxide* (H_2O_2) *Solution*—One volume percent (50 ml of 30 % H_2O_2 with 1450 ml of water). The pH is adjusted (using NaOH or H_2SO_4 as appropriate) to that which is used for the end point in the titration. Solutions should be discarded after 2 or 3 days.

12.5 *Indicator*—Indicators that change color (titration end point) between pH 4 and 5 are recommended, but in no case should the pH exceed 7. Adequate lighting and stirring to ensure proper detection of the end point is essential. A choice of indicators or use of a pH meter is permitted. Directions for preparing two acceptable mixed indicators are as follows:

12.5.1 Mix 1 part methyl red solution (dissolve 0.125 g in 60 ml of ethanol and dilute to 100 ml with water) with 3 parts bromcresol green solution (dissolve 0.083 g in 20 ml of ethanol and dilute to 100 ml with water). Discard the mixed solution after 1 week.

12.5.2 Mix equal volumes of methyl red solution (dissolve 0.125 g in 60 ml of ethanol and dilute to 100 ml with water) and methylene blue solution (dissolve 0.083 g in 100 ml of ethanol and store in a dark glass bottle). Discard the mixed solution after 1 week.

12.6 *Mercuric Oxycyanide Hg(OH)CN* (*Note 2*)—One g/80 ml of water. Prepare fresh solution every 2 or 3 days.

NOTE 4—This is a highly poisonous substance and will explode when touched with a flame or by percussion.

12.7 *Soda-Asbestos*, 8 to 20 mesh.

12.8 *Sodium Hydroxide* (*NaOH*) *Solution, 0.050 N.*

12.9 *Sulfuric Acid* (H_2SO_4), *0.050 N.*

13. Procedure

13.1 Raise the temperature of the furnace to 1350°C at such a rate that the combustion tubes will withstand the thermal shock. Measure 100 ml of 1 % H_2O_2 (12.4) into two gas absorption bottles so that at least 50 mm of the fritted disk is covered in the first bottle, or pour the whole amount into a single absorption bottle. Assemble the apparatus as shown in Fig. 1 except do not connect the rubber tube from the oxygen supply to the soda-asbestos U-tube. Draw air through at about 350 ml/min. The rate of flow can be adjusted by changing the depth of penetration into the mercury of the open-ended glass tube in the vacuum regulating bottle. Connect the oxygen supply to the U-tube and adjust the rate of flow of oxygen to 300 ml/min. This flow rate, at a temperature of 1350°C, will prevent the formation of oxides of nitrogen. The preliminary adjustment to 350 ml/min of air ensures that the connections at the outlet end of the combustion tube are under

slightly reduced internal pressure and no leak of combustion products will occur.

13.2 Weigh about 0.5 g of the analysis sample (Note 3) to the nearest 0.1 mg and spread evenly in a combustion boat previously lined with a thin layer of Al_2O_3 (0.02 to 0.05 g); then cover with approximately 0.5 g of Al_2O_3.

NOTE 3—It may be necessary to grind coals of high mineral matter content to pass through a No. 100 (150-μm) sieve.

13.3 Put the charged boat into the inlet end of the combustion tube so that the center of the boat is 270 mm from the center of the combustion tube hot zone, and if necessary readjust the rate of flow of oxygen to 300 ml/min. Move the boat forward a distance of 30 mm at the beginning of each minute with the exception of the sixth minute, for the next 10 min. The boat should be left at the fifth minute position until the seventh minute to ensure a slow heating rate. At the end of the 10-min period the combustion boat will be in the center of the hot zone. Withdraw the pusher after each movement to prevent distortion of the rod. Keep the boat in the hot zone for an additional 4 min. Disconnect the gas absorption bottles and withdraw the boat onto a sheet of asbestos. This heating program has been established for all types of coal, and where it is shortened for a particular coal, results should be checked against those obtained by using the longer heating schedule.

13.4 Pour the content of the absorption bottles into a suitable titration flask. Wash the bottles and the interior of the silica adaptor with water (12.2) and add the washings to the flask. Add 5 or 6 drops of indicator solution and titrate with 0.050 *N* NaOH solution (12.8). The total acidity, due to oxides of sulfur and chlorine, is given according to the following reactions:

$$SO_2 + H_2O_2 \rightarrow H_2SO_4$$
$$Cl_2 + H_2O_2 \rightarrow 2\,HCl + O_2$$

13.5 After titration, the chloride ion is present in solution as NaCl. Convert the NaCl to NaOH by adding 20 ml of Hg(OH)CN (13.6) solution (sufficient for coals containing up to 1.2 % chlorine):

$$NaCl + Hg(OH)CN \rightarrow HgClCN + NaOH$$

13.6 Titrate the liberated NaOH with the 0.050 *N* H_2SO_4 (12.9). Make a blank determination in the same manner but without sample.

14. Calculation

14.1 Calculate the percentage sulfur in coal as follows:

$$S = \frac{1.603\,[F_1\,(a\text{-}a_1) - F_2\,(b\text{-}b_1)]}{W}$$

where:

S = percent sulfur in coal,
a = millilitre of NaOH solution used in full determination,
a_1 = millilitre of NaOH solution used in blank determination,
b = millilitre of H_2SO_4 used in full determination,
b_1 = millilitre of H_2SO_4 used in blank determination,
F_1 = normality of the NaOH solution,
F_2 = normality of the H_2SO_4 solution, and
W = grams of coal taken.

15. Report

15.1 The results of the sulfur analysis may be reported on any of a number of bases, differing from each other in the manner by which moisture is treated.

15.2 Use the percentage of moisture in the sample passing a No. 60 (250-μm) sieve to calculate the results of the analysis sample to a dry basis.

15.3 Procedures for converting the value obtained on the analysis sample to other bases are described in Specifications D 3176 and D 3180.

PRECISION

16. Eschka and Bomb-Washing Methods

16.1 *Repeatability*—Results of two consecutive determinations carried out on the same sample in the same laboratory by the same operator using the same apparatus should not differ more than the following:

	%
Coal containing less than 2 % sulfur	0.05
Coal containing 2 % sulfur or more	0.10
Coke	0.03

16.2 *Reproducibility*—The means of results of duplicate determinations carried out by different laboratories on representative samples

taken from the same bulk sample after the last stage of reduction should not differ by more than the following:

	%
Coal containing less than 2 % sulfur	0.10
Coal containing 2 % sulfur or more	0.20
Coke	0.05

17. High Temperature Combustion Method

17.1 *Repeatability*—Results of two consecutive determinations carried out on the same sample in the same laboratory by the same operator using the same apparatus should not differ more than 0.05 % sulfur for all coal and coke.

17.2 *Reproducibility*—The means of results of duplicate determinations carried out by different laboratories on representative samples taken from the same bulk sample after the last stage of reduction should not differ more than the following:

	%
Coals containing less than 2 % sulfur	0.15
Coals containing 2 % sulfur or more	0.25
Coke	0.15

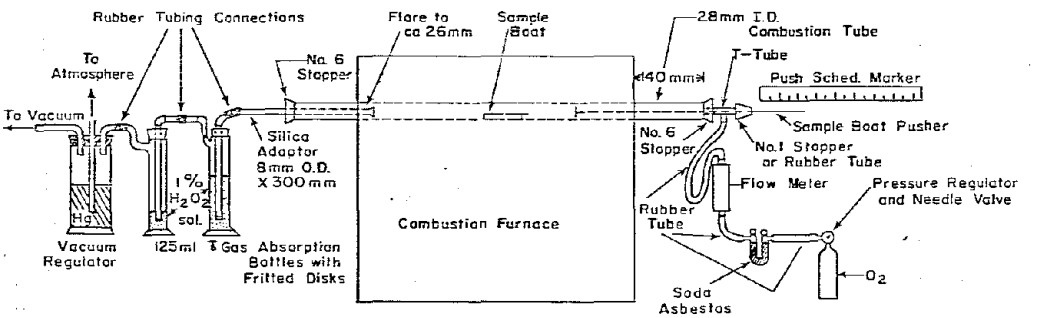

FIG. 1 High-Temperature Combustion Tube Furnace for the Determination of Total Sulfur in Coal.

APPENDIX H

ANALYSES PERFORMED IN THE MOBILE FIELD LABORATORY

ALKALINITY

Method 310.1 (Titrimetric, pH 4.5)

STORET NO. 00410

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml.
   - 1.3 Automated titrimetric analysis is equivalent.
2. Summary of Method
   - 2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.
3. Comments
   - 3.1 The sample should be refrigerated at 4°C and run as soon as practical. Do not open sample bottle before analysis.
   - 3.2 Substances, such as salts of weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
   - 3.3 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in:
     Annual Book of ASTM Standards, Part 31, "Water", p 115, D-1067, Method D, (1976).
   - 3.4 Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.
4. Apparatus
   - 4.1 pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensation is not provided, make titration at 25 ±2° C.
   - 4.2 Use an appropriate sized vessel to keep the air space above the solution at a minimum. Use a rubber stopper fitted with holes for the glass electrode, reference electrode (or combination electrode) and buret.
   - 4.3 Magnetic stirrer, pipets, flasks and other standard laboratory equipment.
   - 4.4 Burets, Pyrex 50, 25 and 10 ml.
5. Reagents
   - 5.1 Sodium carbonate solution, approximately 0.05 N: Place 2.5 ±0.2 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and dilute to the mark.

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where:

A = ml standard acid

N = normality standard acid

7.2 Potentiometric titration of low alkalinity:

$$\text{Total alkalinity, mg/1 } CaCO_3 = \frac{(2B - C) \times N \times 50{,}000}{\text{ml of sample}}$$

where:

B = ml titrant to first recorded pH

C = total ml titrant to reach pH 0.3 units lower

N = normality of acid

8. Precision and Accuracy

8.1 Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/liter, $CaCO_3$	Precision as Standard Deviation mg/liter, $CaCO_3$	Accuracy as Bias, %	Accuracy as Bias, mg/l, $CaCO_3$
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	- 8.19	-9.3
119	5.36	- 7.42	-8.8

(FWPCA Method Study 1, Mineral and Physical Analyses)

8.2 In a single laboratory (EMSL) using surface water samples at an average concentration of 122 mg $CaCO_3$/1, the standard deviation was ±3.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 278, Method 403, (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", p 113, D-1067, Method B, (1976).

ACIDITY

Method 305.1 (Titrimetric)

STORET NO. 70508

1. Scope and Application
   1.1 This method is applicable to surface waters, sewages and industrial wastes, particularly mine drainage and receiving streams, and other waters containing ferrous iron or other polyvalent cations in a reduced state.
   1.2 The method covers the range from approximately 10 mg/1 acidity to approximately 1000 mg/1 as $CaCO_3$, using a 50 ml sample.
2. Summary of Method
   2.1 The pH of the sample is determined and a measured amount of standard acid is added, as needed, to lower the pH to 4 or less. Hydrogen peroxide is added, the solution boiled for several minutes, cooled, and titrated electrometrically with standard alkali to pH 8.2.
3. Definitions
   3.1 This method measures the mineral acidity of a sample plus the acidity resulting from oxidation and hydrolysis of polyvalent cations, including salts of iron and aluminum.
4. Interferences
   4.1 Suspended matter present in the sample, or precipitates formed during the titration may cause a sluggish electrode response. This may be offset by allowing a 15–20 second pause between additions of titrant or by slow dropwise addition of titrant as the endpoint pH is approached.
5. Apparatus
   5.1 pH meter, suitable for electrometric titrations.
6. Reagents
   6.1 Hydrogen peroxide (H_2O_2, 30% solution).
   6.2 Standard sodium hydroxide, 0.02 N.
   6.3 Standard sulfuric acid, 0.02 N.
7. Procedure
   7.1 Pipet 50 ml of the sample into a 250 ml beaker.
   7.2 Measure the pH of the sample. If the pH is above 4.0, add standard sulfuric acid (6.3) in 5.0 ml increments to lower the pH to 4.0 or less. If the initial pH of the sample is less than 4.0, the incremental addition of sulfuric acid is not required.
   7.3 Add 5 drops of hydrogen peroxide (6.1).
   7.4 Heat the sample to boiling and continue boiling for 2 to 4 minutes. In some instances, the concentration of ferrous iron in a sample is such that an additional amount of hydrogen peroxide and a slightly longer boiling time may be required.

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RESIDUE, FILTERABLE

Method 160.1 (Gravimetric, Dried at 180°C)

STORET NO. 70300

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 The practical range of the determination is 10 mg/1 to 20,000 mg/1.
2. Summary of Method
   2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.
   2.2 If Residue, Non-Filterable is being determined, the filtrate from that method may be used for Residue, Filterable.
3. Definitions
   3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.
4. Sample Handling and Preservation
   4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
   5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.
   5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.
6. Apparatus
   6.1 Glass fiber filter discs, 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E, or equivalent.
   6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
   6.3 Suction flask, 500 ml.
   6.4 Gooch crucibles, 25 ml (if 2.1 cm filter is used).
   6.5 Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).
   6.6 Steam bath.
   6.7 Drying oven, 180°C ±2°C.
   6.8 Desiccator.

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RESIDUE, NON-FILTERABLE

Method 160.2 (Gravimetric, Dried at 103–105°C)

STORET NO. 00530

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 The practical range of the determination is 4 mg/1 to 20,000 mg/1.
2. Summary of Method
   2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103–105°C.
   2.2 The filtrate from this method may be used for Residue, Filterable.
3. Definitions
   3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103–105°C.
4. Sample Handling and Preservation
   4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
   4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
   5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.
6. Apparatus
   6.1 Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeves Angel 934-AH, Gelman type A/E, or equivalent.
   NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.
   6.2 Filter support: filtering apparatus with reservoir and a coarse (40–60 microns) fritted disc as a filter support.

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7.6 Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103–105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

8. Calculations

8.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = ml of sample filtered

9. Precision and Accuracy

9.1 Precision data are not available at this time.

9.2 Accuracy data on actual samples cannot be obtained.

Bibliography

1. NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.