Proceedings of the First

COAL TESTING CONFERENCE

MARCH 2, 3, 1981
LEXINGTON CENTER
LEXINGTON, KENTUCKY

COAL TESTING CONFERENCE

Proceedings of the 1st Conference

Volume I

Lexington, Kentucky - March 2-3, 1981

Sponsored by
Standard Laboratories, Inc.
Corporate Headquarters:
3322 Pennsylvania Avenue
Charleston, WV 25302

Printed in the U.S.A., Charleston, WV 25302
Karen Gallagher, Editor

COAL TESTING CONFERENCE

Standard Laboratories, Inc.

Troy Stallard
Host & Moderator
Vice President

Gladys Berchtold
Host
President

Karen Gallagher
Conference Manager
Manager of Administrative Services

Ray Daniels
Exhibitors
General Manager of
Standard Instrumentation, Inc.

Dick Kelly
Moderator
Technical Director

Israel Broome
Moderator
President of
Precision Samplers, Inc.

Mick Samples
Moderator
Manager of
Special Projects

CONTENTS

QUALITY CONTROL

PAPERS NOT AVAILABLE AT TIME OF PUBLICATION*

Sampling Case Study — Auger Application
Jerry Sullivan

Theory and Overview
Al Cameron

Sole and Moveable Wall Ovens
H. E. Harris

H-Plant Practice and Experience
Karl Mellenger

Spark Source Mass Spectrophotometry
Dr. Leroy Jacobs

Lab Computerization
Greg Gould

Washability Testing for Prep Plant Design
Joe Leonard

Washability Test Design
Robert Quinlan

*For any further information on these papers, please contact the authors.

WELCOME

Given at March 2, 1981 Banquet

Troy F. Stallard
Vice President and
Chief Operating Officer
Standard Laboratories, Inc.

In preparing for this gala affair, I have been amazed and even a little overwhelmed by your great response to a "Coal Testing Conference." We had initially felt that the conference would be a rousing success if somehow we could get but 200 people to attend. And in looking across this room of about 600, I can virtually see the entirety of our industry in the U. S., as well as a strong contingent from Canada, and even a few people from Mexico. Almost every ton of coal mined, shipped or used has a representative at this conference. One thing is clear: we, who are here tonight, are, in essence, the Coal Testing Industry in North America.

The question that I keep asking is "Why?" Why did all you people come? Is there so much interest in coal quality? Well, tonight is proof that one heck of a lot of people care about coal testing and coal quality.

In reflecting on the meaning of about 600 persons getting together on coal testing, I am able to make a few observations. Fortunately, for you, they are short observations so I'll do my best to finish before too much more bar time is consumed.

First, we are gathered here because we have a huge responsibility. And I do mean "huge." Nothing less than the continuation of progress in the world we know is at stake. Everyone recognizes the future of our countries is heavily dependent upon coal. Coal must be used until abundant alternative energy sources can be identified and scaled up into massive production. We in this room have a very significant role to play in this scenario. Coal quality becomes increasingly vital as the demand for coal increases. More, better and faster results will be required in order to make effective real-time production/utilization decisions and these are the basic decisions which will permit our society to move forward. We have indeed a huge responsibility.

A second observation is that our profession is becoming increasingly sophisticated. We, to fulfill our responsibility, have to keep up with technological changes, and accordingly, a conference is one vehicle to help accomplish this. Coal testing has been -- and to some degree still is -- archaic as compared with some of the other testing fields. When I say this I must include myself since I've been a part of it for as long as I can remember. In the past there has not been the demand for increased sophistication as the coal industry itself was in the doldrums. No longer is this the case. To do the job we have to do, we must progress and we must progress rapidly. Quality programs must be upgraded. No longer is one split to another lab once a year sufficient for quality control. Equipment enhancement and method improvement must take place. Better training of bench personnel is essential. We as an industry may have begun the task of upgrading our technology but we have a long way to go.

My third and final observation is that fortunately we are all in this together. This is our chosen profession, and as such, I believe that we are searching for some sense of community, comradery, some occupational alliance, something that resembles the fraternal professionalism that exists in many other groups such as engineers or the AMA and its doctors. It is good that we remember that we are professionals and that we owe something to our profession. There should be a strong sense of responsibility to coal testing and to each other; perhaps as strong as our responsibility to our employers. With more opportunities such as this to gather with each other as colleagues, this sense of common purpose will grow and perhaps permit us to mature into a close-knit professional association.

In closing let me say that, all in all, we simply must begin to do a better job to get our act together, as the quality control segment of the coal industry. To do this, we in this room must:

-- recognize our vital responsibility as a very important part of the coal industry.

-- recognize our need to improve our equipment, personnel and methods.

-- recognize that we, indeed, are linked together professionally and that we should develop a sense of community in our segment of the coal industry.

This is what we have to do--let's hope that this conference is a symbol that we are on our way.

We thank you for your attendance, we hope that you will find the conference enjoyable and rewarding.

THE STATUS OF THE COAL INDUSTRY

Charles H. Daly
Publisher
Coal Age

I've taken on this subject, the status of the coal industry, with a fair amount of apprehension. I heard it said several years ago that he who lives by the crystal ball must be prepared to eat ground glass.

I think that I'll try not so much to forecast, but to show you the trends and let you draw your own inferences.

The most important thing about coal in the eighties is that we will be burning a lot more of it than we did in the seventies. I'm sure that most of you have heard some gloomy stories about the state of the coal industry, most of them are true. The industry does suffer from over capacity. There are thousands of miners out of work. Many mines have either shut down or reduced their production. Despite that the outlook for coal in the eighties is bright.

Now, some statistics. Actual bituminous output in 1980 weighed in at 830 million tons, an 8.3 percent increase over the 766.3 million tons produced during 1979. This increase was considerably higher than the original forecast for 1980 production.

Before I go on speaking blithely about hundreds of millions of tons of coal, I'd like to give you an idea of just how much coal a million tons is. If you could pack a million tons of coal into a neat cube, you would have a cube about three hundred feet by three hundred feet by three hundred feet. Another way to think of it is that you could get two football fields on each of the six sides of this million ton cube.

To understand the direction that coal will go in the nineteen eighties, it helps to see where coal has been. In 1940 we produced 460 million tons of coal. The customers for coal then were manufacturing, which took 25%, the steel industry took 15%, railroads took 20%, retail sales -- mostly household furnaces -- took about 20%, electric utilities used about 10% and the rest was exported.

The ups and downs of coal since 1940 are fair indicators of our country's economic history. Its use surged upward during the Second World War, downward at war's end, then experienced gradual gains with occasional abrupt declines during recessionary periods.

In 1940 coal was truly king. It accounted for more than half the energy used in the United States. It powered the railroads, heated homes, fueled industrial boilers and generated more than half our electricity.

But by 1950, coal's customers began to disappear. Railroads switched to diesel oil or electricity. Cheap natural gas and oil took over the home heating business. Not only were they cheaper, they were easier to handle and didn't track up the carpets. New methods of production reduced the steel market for coal. By 1961 coal reached the lowest level of production since the depression -- all its markets had either stabilized or declined, all but one; the electric utility industry.

The steady rise in the use of coal to generate electricity kept pulling coal production up. By 1975, coal production surpassed the record it established in 1944.

In 1940, although coal produced 55% of the country's electricity, the utilities took only about 10% of coal production. Now, utilities buy 75% of coal production, and coal generates about half of our electricity. So much coal is used because we are using 12 times as much electricity now as we did in 1940.

We can look forward to increasing use of coal by the electric utilities not only because of growing consumption of electricity, but because coal will take a larger share of the generating market. The rate of increase in electricity consumption won't be what it was in the years from '68 to '73, when it rose at 7½% a year. Conservation, plus a slowdown in economic growth, will limit the growth in consumption to about 3-1/3% a year in the eighties.

The National Electric Reliability Council predicts that coal will have an increasing share of utilities energy consumption. The Council's projection calls for coal to produce half of our electric power by 1988 and use 900 million tons of coal, as compared with 540 million tons used last year. The projection was made before the accident at the Three Mile Island nuclear generating plant. Coal could well end up producing 55% or more of total electricity by 1988.

I've averaged the forecasts for the coming 20 years of the Department of Energy, Ford Foundation, M.I.T., World Coal Study, Data Resources, Inc. and National Coal Association. The consensus is that coal production will reach 2 billion tons by the turn of the century.

Looking forward to a 2 billion ton year for those of us who earn our living in the coal industry, is a happy prospect, and it should mean prosperity for us during the 80's.

Like all pleasant business forecasts, however, the picture is not all perfect and growth will not be immediate.

In fact, for 1981, we call this a "slightly" year. Production will rise slightly, and so will utilities' consumption. Prices will rise slightly, in response to a new contract with the United Mine Workers and to rising rail rates.

The new administration will improve the operating climate slightly and metallurgical coal production will improve slightly as steel production improves, you've guessed it, "slightly."

For all the optimism generated in the coal industry by the election of Ronald Reagan, a Republican Senate and a more conservative House, it is unlikely that there is room for the kind of dramatic government action that will have a marked effect on consumption and production during 1981.

It's possible, in fact, that some of the administration's early actions will dampen our ardor for the new man in the White House.

For instance, new Budget Director, David Stockman, has recommended elimination of DOE's coal conversion office, thus ending a 3 year totally unsuccessful effort to force utilities to switch to coal. Though the conversion plan did not succeed, there are those who would favor revisions in the Clean Air Act to keep the program going.

In addition, the new administration is planning to reconstruct the Synthetic Fuels Corporation, placing more responsibility for financing on the private sector, thus reducing coal usage potential in the long term.

In the main, however, we should view the new administration as being friendly toward our industry--promises made by President Reagan, and the basic attitudes of the Republicans now chairing the various committees, tell us that the long years of frustration with impossibly strict federal regulations are over.

The Office of Surface Mining will come under review, and the result will be new rules that will not be overly restrictive and costly and jurisdiction will return to coal producing states, reducing federal participation to one of providing guidelines.

This concept was behind the Act when it passed in Congress in 1977. What emerged in subsequent years was a Democrat interpretation. What will appear over the next several years will be the Republican interpretation.

There will be considerable confusion for a while during the early stages of the new administration as many current mine regulatory programs grind to a halt. The feeling is that it will take a full year for the new attitude to be reflected.

The Clean Air Act Amendment of 1977 and the regulations it spawned are often thought of as hindering coal market development. Its reorganization will be on the early agenda

f the new Congress but changes and amendments to the Act tself won't come easy.

While the Republican party may be assumed to be generlly pro-coal in regulatory matters, this view may not be hared by the new chairman of the Senate Environment and ublic Works Committee, which handles clean air amendments. enator Robert Stafford has built a record of support of nvironmental amendments in the past, much to the consternation of coal marketers, and he has succeeded Senator ennings Randolph, a high visibility friend of our industry.

I cannot leave the Reagan administration and its attiude toward big government without attempting to underline he importance of two men, Jim Watt, the new Secretary of the nterior, and Senator Jim McClure, new Chairman of the Senate nergy Committee.

McClure is committed to the development of alternative uels, particularly synthetic fuels and geothermal reources, and, he and Secretary Watt are determined to curb overnment excesses and to restore a balanced approach to a ineral development program.

With your permission, I'd like to broaden this subject bit to consider mineral development or rather lack of it.

Senator Jack Schmitt has issued this warning: "Not far in the future awaits sudden recognition of a 'materials risis' with the possibility of more devastating effects han our current energy crisis."

Congressman Jim Santini has done his best to underline he problem, pointing out that the U.S. is dangerously reliant on foreign sources for key minerals.

Currently, we import over 50 percent of more than 20 of he minerals essential to our country. In some critical inerals, such as cobalt, manganese, chromium and platinum, e import nearly all of our annual needs.

How vulnerable are we? Consider that 98 percent of the orld's known reserves of manganese ore are in southern frica and in the U.S.S.R. Without manganese, it is virtually impossible to make iron and steel, for instance.

For chrome ore the figure is 96 percent. Without hromium we cannot produce high temperature and high pressure alloys essential for electricity generating turbines, for nuclear reactors, for advanced aircraft and for the petro-chemical industry.

Domestic sources of these minerals do not exist or their mining is severely restricted by our own environmental olicies.

It is an astounding fact that only a fraction of one ercent of U.S. land surface has ever been touched by mining nd much of this has been restored. Where minerals are found, their development sometimes requires the permanent se of the site, just as highways and shopping centers do, ut more often, mining represents only a temporary disturbnce of the land while resources are extracted. This is not abuse of the land, it is the highest use of the land.

What must be done to begin a corrective approach to our ilemma?

-- review and improve our stockpiling laws, policies and programs
-- re-evaluate our foreign policy in view of our materials dependency
-- review our public lands policy

And in that regard, we must open potentially mineral-rich areas of the U.S. for exploration and mining. We must -- with the President and his administration leading the way -- bring our environmental values and our critical mineral needs into harmony. We should, at the same time, take a hard look at regulations that restrict the domestic mining industry. We should offer special tax incentives to domestic mining companies for investing in capital equipment.

There cannot be any question that assuring supplies of critical minerals for our industries may be the most important undertaking ever to face this nation.

And now, back to coal for my conclusion...

My favorite magazine just made public in our February issue the results of a survey conducted by our annual, Keystone Coal Industry Manual. It reports on the planning for new capacity by new mines currently being developed, by older mines being expanded and by operations still on the drawing board. These added capacity figures, supplied to us by the mining companies, come to 515 million new tons by the end of 1989.

Now, all this may not happen, as a huge set of vagaries takes effect, but the numbers reflect industry optimism and the continuing conviction among both producers and suppliers that coal will provide the answers to the worldwide energy dilemma.

So, there we have a brief look at coal's past, its short and long term future with emphasis on the impact of the new administration with its fresh attitude toward conditions effecting our industry.

Coal is at a crossroads. On the one hand, government cooperation can trigger the biggest production surge in our history -- on the other hand, public and environmentalist pressure can bring about governmental back-tracking, thus denying us the opportunity to contribute a solution to energy problems.

As business men and women, we must set realistic goals. We must work toward these goals with enthusiasm and courage. We must become good communicators, taking our story to the public in place of our tendency to talk among ourselves, convincing the convinced.

Our industry leaders, individually and through state and national associations, must be more visible, must set performance examples, providing inspiration and motivation.

We must take advantage of our newly found opportunity, for the rewards can be great -- and rewards in life don't go to the dreamers and the talkers and the wishers -- rewards go to the doers, and believe me, this great coal industry, a cornerstone of civilization, has a lot to get done!

MARY

The quality assurance component of a laboratory ality control program is one of the most vital parts. It ould be developed along with the quality and quality con- ol components so that all three components may be inte- ated into an effective program.

The quality assurance function of a quality control ogram is comprised of the following components:

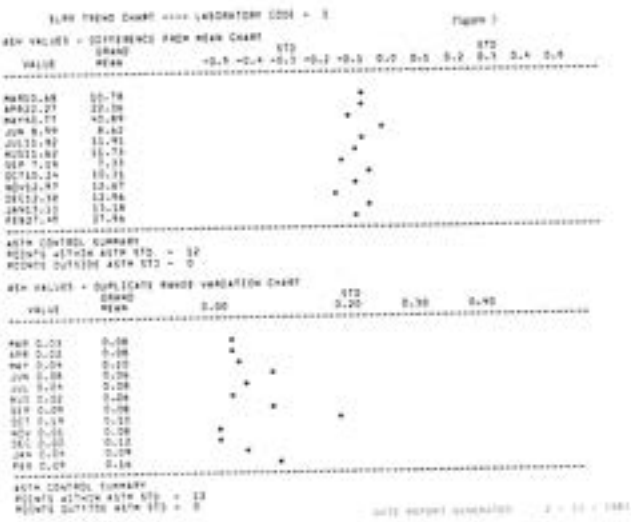
1. Information -- which identifies the laboratory's level of accuracy and precision.
2. Interpretation -- of the information leads to the decision of in-control, problem, and out-of-control.
3. Response -- the actions initiated as a result of the interpretation.

Each of the three components of the quality assurance nction -- information, interpretation, and response -- ust be developed with accuracy, timeliness, and simplicity mind.

These components and attributes, when effectively ntegrated, will allow the quality control program to ffectively influence the laboratory's results with the east interruption to the laboratory's main role -- the roduction of test data.

Figure 1

Figure 2



INTERLABORATORY QUALITY CONTROL

R. D. Graham
Manager, Amax Coal Company
Midwest Area Laboratory

INTRODUCTION

The goal of every analytical laboratory and of every analyst is to produce highly precise and accurate analyses in a timely and cost effective manner. Quality assurance must be an integral part of any laboratory's standard operating procedures to insure the precision and accuracy of these results.

The subject of interlaboratory quality assurance is an important one. Within the temporal and spatial limitations of this presentation the necessity for and the results of an active quality assurance program can be discussed only briefly. Therefore, a general overview will be presented with a bibliography which hopefully will be of value in developing, improving or expanding a quality control/quality assurance program.

In this text the terms quality assurance and quality control are used almost interchangeably. It would perhaps be best to differentiate between these two terms as has Hauser: Quality control is defined as those internal operations performed during the measurement process to document the quality of data whereas quality assurance are those components performed on a more occasional basis, usually by someone outside of normal operations, to gain an independent assessment of monitoring operations and data assessment. These are good definitions but it is difficult to categorize some quality elements under either one or the other definition. Therefore, these terms will not be mutually exclusive as they are used in this text.

MISCONCEPTIONS ABOUT QUALITY ASSURANCE PROGRAMS

Through lack of knowledge, misunderstanding, or by design, misconceptions about quality assurance programs abound, especially among those uninitiated as to the fundamental statistical concepts of sampling and sample analysis.

Three such misconceptions are: 1. The primary aim of the quality control program is to chastise individuals or groups of individuals whenever a process or a product fails to meet specification. 2. Individuals who are found out by these quality control groups are subject to ridicule, scorn and embarrassment. 3. A quality control program is too time consuming, utilizing too much of the laboratory's overall resources and, thus, detracting from the productive analysis of actual test samples.

In actuality: 1. The primary purpose of the quality control group is to keep individuals or groups of individuals out of trouble through its inspection and auditing roles. This involves the monitoring of systems and procedures, evaluating whether or not the written procedures are being followed, establishing the validity of generated data and areas of concern. Deviations are reported and documented leading to improvements in methods and technique with a concurrent improvement in accuracy and precision.

2. A good quality control/quality assurance program will minimize the occurrences that may lead to an embarrassing situation and will keep this cause of embarrassment to a least critical level, i.e., the problem will be found and corrected before it gets completely out of control and jeopardized the product's, institution's or the individual's reputation or livelihood.

3. It can be more expensive in the long run not to have a good quality control program than it costs to have one, even if the program takes 20-30% of the available time and resources. Whenever data is generated without sufficient control, its quality may become suspect. When this happens the old data is typically discarded, and new samples are obtained and analyzed. Thus the initial effort is lost and additional effort must be expended to repeat the process.

Additionally, there can be indirect costs associated with an inadequate quality control program: 1. loss of respect, reputation or trust in the analyst or lab, 2. loss of self confidence on the part of the individual analysts, 3. heightened apprehension and tension from being under increased pressure to produce more accurate analyses, 4. unnecessary rechecks or, worse yet, nonperformance of necessary rechecks and 5. increased maintenance costs from repairs which could have been detected early through an adequate quality control program.

DEVELOPMENT OF A QUALITY ASSURANCE PROGRAM

The development and institution of a productive quality assurance program is no small project. It is not something which can develop overnight but requires the dedication, involvement and commitment of all individuals. This commitment must start with upper management and be passed down to the analysts actually performing the bench work.

For a laboratory which has no formal quality assurance program, its development can seem to be an insurmountable project. For a laboratory with an existing program, it is a continuous, ongoing battle to maintain and update the program.

INDIVIDUAL COMPONENTS OF A GOOD INTERLABORATORY QUALITY CONTROL/QUALITY ASSURANCE PROGRAM

I. Standardized Procedures

The first element of a good quality assurance program is that of standardized methods. Some tests are so empirical in nature that without exact duplication of equipment, operating conditions and measurement technique there could be no agreement between even the most conscientious individuals (e.g., volatile matter or moisture analysis of low-rank coals). Thus while objectivity may require that in compliance testing the participatory factions obtain their respective analyses independently, common sense will require that their initial action be agreement upon common or standard procedures by which each shall independently procure, prepare and analyze the material in question.

Several types of protocols must be established:

A. Sampling Procedures
B. Sample Preparation Procedures
C. Analytical Procedures

A. Sampling Procedures

Improper sampling or sample preparation has a far greater potential of introducing bias into the analytical results than does the analysis itself. Considering the technique necessary to properly take a representative sample from ten thousand tons of a material as heterogeneous as coal, dividing and reducing this representative sample into 50-100 grams of laboratory sample suggests the validity of this statement.

B. Sample Preparation

Reproducible methods of receiving samples, logging and storing them must also be included as a part of the preparation procedures. These should allow for tracing the history and custody of the sample for later reference (chain of custody). The method of logging and storing samples as they are received can also be applicable to logging and storing reserve splits and analytical samples after analysis.

C. Analytical Procedures

Accurate chemical analyses cannot be based solely upon the response of a device - be it an instrument, human eye or "black box". One needs to consider the change of the response of the test instrument to the characteristic properties of the sought-for-component as it changes with concentration, time, temperature, humidity and other "environmental factors". The effect of other properties of the component being sought and the properties of other components within the matrix of the sample being tested must also be considered.

Therefore, standard analytical procedures for calibration and standardization of the instrument must be established.

1. Equipment Set-Up Procedures

a. At least two copies of the instruction manual, including set-up instructions should be received.
b. One is for general use, the other is for the files.
b. Copies of repair manuals, wiring diagrams, parts lists, etc., should be obtained.
c. The set-up procedures should be based upon those of the manufacturer.

2. Calibration Procedures

a. All essential calibration materials (weights, thermocouples, galvonometers, etc.) should be traceable to National Bureau of Standards.
b. Written procedures for periodic reverification of the accuracy of the calibration materials should be established.
c. The individuals doing the reverification of the calibration materials should use NBS calibrating materials and furnish certificates of recalibration.
d. A written history of when calibration materials were purchased, recalibrated, etc., and by whom should be maintained.

3. Standardization Procedures

a. The purity of standards, solvents and reagents should be checked. They must be checked for strength, for deterioration with time, temperature, humidity and other environmental factors, for contamination and for expiration dates.
b. The purchasing procedures should be standardized. Everyone in the laboratory system should use the same materials from the same vendors.
c. Only standard reference materials, traceable to NBS Standards, should be used.
d. Prepare reagents carefully. Procedures for preparation, use, storage and restandardization of reagents should be documented.
e. Only high quality glassware and equipment should be used during preparation, standardization and use of standards.
f. The container should be labeled as to when the standard was prepared and standardized and by whom. The standard should be discarded before the container is completely empty to avoid possible contamination.
g. Secondary or tertiary standards can be used for restandardization. They are less expensive and plentiful. These may consist of:
   i. a previously analyzed round robin sample
   ii. a purchased secondary standard
   iii. a previously analyzed test sample in which the value of the components of interest are known with a high degree of accuracy.

4. Quality Assurance Check Procedures

Quality assurance procedures should include the analysis of uniformly distributed check samples. Some should be known by the analyst in order to provide him/her with immediate feedback. Others should be known only at an appropriate time after the analyses have been submitted for review in order to obtain an unbiased measure of the performance of the analyst and of the test.

a. Blind duplicates should be analyzed. Two types are recommended:
   i. unknown to the analysts
   ii. unknown to the laboratory - submitted by the sampler and only known after completion of the analysis and the submittal of the analytical report.
b. Spiked samples should be included.
c. Blanks and spiked blanks should be incorporated into the program.
d. Round robin or split samples should be analyzed regularly.
e. Synthetic, "junk" samples or tertiary standards should be a part of the daily routine.
f. Certified standards should be run regularly.
g. Proficiency samples are an important requirement for the program.

5. Maintenance Procedures

Procedures must be established prescribing certain maintenance functions.

a. Periodic preventative maintenance or equipment checkout should include:
   i. lubricating, changing filters
   ii. cleaning, dusting, washing
   iii. replacement of worn parts
   iv. recalibration
b. The equipment should be monitored routinely:
   i. monitor wear, e.g., of hammermills by screening the product
   ii. monitor for and correct loose bolts, guards
   iii. monitor for unusual noises, speed, appearance or condition
c. The equipment should be repaired as needed and good maintenance records must be kept.
d. A standardized equipment list or inventory should be maintained because:
   i. this allows for easier troubleshooting, repair, or replacement of equipment
   ii. the central lab can act as a supply warehouse for spare parts, accessories, materials and supplies
   iii. this will minimize bias caused by different types of equipment

The following lists information which should be on file for all laboratory equipment:

1. The name of the manufacturer
2. The equipment model and serial number
3. The properties subject to standardization
4. The range of operation and the range of calibration
5. A reference to a recognized calibration procedure

6. The frequency of calibration
7. Allowable tolerances or maximum sensitivity
8. The source of verification
9. A chronological history of repairs, modifications or substitutions
10. Traceability of reference standards to the National Bureau of Standards or accepted values of natural physical constants.

6. Approved Measurement Procedures

a. Acceptable measurement procedures must incorporate the basic concepts of calibration, standardization and maintenance discussed above.
b. Most standard procedures have their basis in standard methods from EPA, NIOSH, other regulatory and governmental authority or by ASTM, ANSI, API or other industrial consensus groups. If the laboratory's standard operating practice is a variant of one of these standard procedures the standard procedure should be referenced and the deviations noted.
c. Statistical data should be noted stating the expected repeatability and reproducibility of the test data.
d. Proper methodology for weighing, measuring, cleaning are essential.
e. Cautions and safety considerations should be listed.
f. Procedures for performing calculations need to be listed. The data review procedure should be discussed.
g. Reporting protocol should be noted.

SAMPLE EXCHANGE PROGRAMS

The laboratory should engage in more than one proficiency testing program, especially in those sponsored by federal agencies such as EPA, NIOSH, Department of Commerce, etc. These provide an economic source of high quality "standards" and under some circumstances participating can be paramount to "certification".

A special type of proficiency sample is that from a program set up for interlaboratory comparison. These split sample or round robin samples can be done routinely to monitor results produced by different labs or it can be done whenever two or more labs produce disparate results.

Whenever undertaken for the latter reason the exchange should be preceded by a visit to each lab by members of the other lab's staff in order to eliminate any obviously nonstandard practices or to select potential sources of error which will be monitored by the exchange. Whether the bias is caused by something within the lab or whether the bias has been introduced in the sampling or preparation steps of reduction and division could be determined by such a visit. This could be less expensive than conducting a sample exchange that may otherwise tell nothing.

If the source of the bias is not located by the exchange visits the length of the sample exchange, the type of sample exchanged (its state of preparation, top size, etc.) and analyses determined will depend upon the nature of the suspected problem.

The sample exchange program must be well thought out in advance in order to guarantee that once the exchange is complete, meaningful results will be obtained which will either locate and identify the source of the bias or will give direction as to what to do next.

If the exchange is a periodic interlaboratory comparison mutually agreed upon in advance by the parties, it is important to establish what methods will be used by each participant. Different methods can yield slightly different results. A slightly positive or slightly negative bias by one method as compared to another is not to be unexpected. The report form should contain space to state which method is used or how the method used varies from the standard procedure.

Some round robin programs are available commercially and are especially useful to laboratories who have inadequate time or expertise to formulate their own program or to those who want an independent third-party basis for their quality control program.

II. Documentation

The keystone of any quality assurance program is documentation. Each component of the program must be adequately documented in order to provide the historical perspective which is necessary in detecting trends which can be helpful in taking corrective actions. Formal standard operating procedures must be written and all procedures should be dated as to issue and review date. All calibration and restandardization actions should be dated and noted. Results obtained from quality assurance samples must be recorded. Repairs and other maintenance related events, temperature, humidity or other environmental factors should be noted. A chain of custody should be maintained. All lab bench sheets should be dated and initialed; all reports should be dated and signed. If there is any question as to whether a number or event is important, it should be recorded.

An especially valuable method of documenting data for which the data trend is as important as the data itself is the Control Chart. ASTM Manual STP 15D is a valuable tool for suggesting various types of such control charts. Figures 1-8 also show various applications of control charts.

III. Data Management

Data management consists of handling the data which is generated. It should be in a format that allows it to be readily accessible for use. Manual methods such as the control charts mentioned above, bound laboratory note books, file cabinets and microfilm are much in use today. A decision should be made as to how long each type of document will be retained on file.

More and more, however, computers are taking over the management of data files. The hook-up of instrumentation with the appropriate interface to computers allows for direct acquisition of the raw data into the computer memory. This eliminates much of the sources of error which could occur in data handling, e.g., transcription errors, calculation errors and typographical errors. The physical requirements for data file storage is reduced, data is easily accessible and can be produced in almost any format desired, either as raw or calculated data.

IV. Auditing

Auditing involves ascertaining that all data generated and all reports are complete, clear and concise. All quality assurance procedures are reviewed to see that they are followed exactly and completely. On-site surveys of satellite labs or independent performance audits of a laboratory system is a very important part of a quality assurance program. At times an "outsider" can detect deviations from good laboratory practice that have been overlooked by the employees of the site.

For all procedures, there needs to be a formal protocol for review, approval, audit, update, recall and distribution of the established procedures. Inaccurate or outdated prodedures can cause quality problems. It is important to keep a record of all existing and former standard procedures, as it may be important to know how the procedure used to generate today's data differs from the procedure used to generate similar data size six years ago.

V. Training

Implementation, documentation and utilization of a

quality assurance program will not be effective if the individuals using the system do not understand it. If the formal documents described above were presented all at once to a typical analyst he would be overwhelmed just by the sheer volume of the material. The formal standard operating procedures should primarily be used as a reference. Instruction programs referencing the formal standard operating procedures should serve as the main training instrument for the analyst. Shortened bench sheet instructions should be available for routine daily use.

An adequate training program must be an integral part of a quality assurance program. Subjects for discussion should include: sampling, sample preparation and analysis procedures (including set-up, calibration and standardization), quality assurance principles, safety and legal regulations governing laboratory analyses.

Laws such as the Clean Water Act, Resource Conservation and Recovery Act, Occupational Safety and Health Act, and others establish penalties to individuals and/or companies for violations of these acts. Inasmuch as these guidelines affect the analyst, this should be communicated to him.

FINAL CONSIDERATIONS

Organizations and governmental agencies are pursuing various accreditation programs. The rebirth of coal utilization as an increased energy source and as a feedstock for other technologies has increased the necessity for increased quality control at laboratories engaged in coal and coal-related testing. This is partly due to the continually rising world-wide energy costs and the economic incentives associated with compliance testing. Stricter environmental regulations make it essential that required analyses are precise and accurate. Adding to the difficulty in obtaining "accurate" analyses are the demands made by an ever increasingly more technologically oriented society that, because of great strides and achievements made by technology in other areas, insist that analyses always be done more quickly and more accurately at lower and lower concentrations. Potential accreditation programs need to be scrutinized to determine if such programs would be beneficial in dealing with these demands placed upon laboratories engaged in coal and coal-related testing.

BIBLIOGRAPHY

Books

Berman, Gerald A., Editor, Testing Laboratory Performance: Evaluation and Accreditation, National Bureau of Standards Special Publication Number 591, U.S. Government Printing Office, Washington, D.C., August 1980, 178 pages.

Committee E-11 (prepared by), ASTM Manual on Presentation of Data and Control Chart Analysis, ASTM Print Shop, Lancaster, Pennsylvania, October 1976, 162 pages.

Periodicals

Amore, Francis, "Good Analytical Practices," Analytical Chemistry, Volume 51, Number 11, September 1979, pp. 1105A-1110A.

Amorosi, Roger J., "American Association for Laboratory Accreditation: All About AALA," ASTM Standardization News, Volume 8, Number 11, November 1980, pp. 21-24.

Berman, Gerald A., "ASTM Committee E-36: Participation in Laboratory Evaluation and Accreditation," ASTM Standardization News, Volume 8, Number 12, December 1980, pp. 8-10.

Booth, Robert L., "EPA's Laboratory Certification Program," Analytical Chemistry, Volume 51, Number 8, July 1979, pp. 861A-862A.

Federal Register, Part II, December 22, 1978, "Good Laboratory Practice Regulations," pp. 59986-60013.

Fischbeck, Robert D., "Good Laboratory Practice Regulations," American Laboratory, September 1980, pp. 125-130.

Hauser, Thomas R., and Dwight G. Ballinger, "Quality Assurance Update," Environmental Science & Technology, Special Report, Volume 13, Number 11, November 1979, pp. 1356-1366.

Hayes, Janan M., "Licensing Registration and Certification: What Does It Mean to You as a Chemist?", Analytical Chemistry, Volume 51, Number 3, March 1979, pp. 333A-334A.

Horwitz, William, "Good Laboratory Practices in Analytical Chemistry," Analytical Chemistry, Volume 50, Number 6, May 1978, pp. 521A-524A.

Kirkpatrick, Diana and Jeffrey Horlick, "Proficiency Testing: An Essential Element of Laboratory Accreditation," ASTM Standardization News, Volume 8, Number 12, December 1980, pp. 14-17.

Krashes, David, "National Accreditation: A Laboratory Owner's View," ASTM Standardization News, Volume 8, Number 12, December 1980, pp. 18-20.

Locke, John W. and John A. Glimour, "Australia, New Zealand, and United States: Laboratory Accreditation Systems Compared," ASTM Standardization News, Volume 8, Number 11, November 1980, pp. 14-17.

Marsh, Joe L., John W. Gable, Jr., John E. Regnier, "Safe Drinking Water Act: Laboratory Certification Trends," American Laboratory, December 1980, pp. 55-59.

Miller, Stanton, "Trace Analysis of Environmental Pollutants," Environmental Science and Technology, Volume 14, Number 12, December 1980, pp. 1434-1435.

Monaghan, N. F., "National Association of Testing Authorities: Australia's Laboratory Accreditation System," ASTM Standardization News, Volume 8, Number 11, November 1980, pp. 10-13.

Morrison, G. H., "Analytical Controversies," Analytical Chemistry, Volume 52, Number 12, October 1980, p. 1793.

Oatess, William E. "Establishment of Accreditation Programs for Environmental Labs," Environmental Science and Technology, Volume 12, Number 10, October 1978, pp. 1124-1127.

Regnier, John E., John W. Gable, Jr., Joe L. Marsh, "Laboratory Certification," Environmental Science and Technology, Volume 13, Number 1, January 1979, pp. 40-45.

Rice, James K., "Analytical Issues in Compliance Monitoring," Environmental Science & Technology, Volume 14, Number 12, December 1980, pp. 1455-1457.

Ronsen, Bruce, "Safe Drinking Water Act, Quality Assurance and Compliance for the Water Supply Laboratory," American Laboratory, December 1978, pp. 53-61.

Ruggiero, Michael A., "EPA's Industry Assistance Office," Environmental Science and Technology, Volume 13, Number 1, January 1979, pp. 46-48.

Schock, Harvey E., "Laboratory Evaluation: An Opportunity for Profit," ASTM Standardization News, Volume 8, Number 12, December 1980, pp. 11-13.

Seeber, Allen J., "Laboratory Quality Assurance," Environmental Science and Technology, Volume 12, Number 10, October 1976, pp. 1128-1130.

Smith, Robert V., "Good Laboratory Procedures for Technicians in Biopharmaceutical Analysis," American Laboratory, August 1976, pp. 47-51.

Unger, Peter S., "NVLAP Accreditation," ASTM Standardization News, Volume 8, Number 11, November 1980, pp. 18-20.

Wening, Robert J., "The Role of the Quality Control Manual in the Inspection and Testing Laboratory," Testing Laboratory Performance: Evaluation and Accreditation, National Bureau of Standards Special Publication Number 591, August 1980, pp. 99-103.

ASTM Committee D-22 (sponsored by), Standard ANSI/ASTM D3614-77, "Criteria for Laboratories Engaged in Sampling and Analysis of Atmospheres and Emissions."