Designation: D 4182 – 97

Standard Practice for Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke[1]

This standard is issued under the fixed designation D 4182; 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. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Traditionally, coal testing facilities have been established to serve the specific interests or activities of one or more of the following:

(*1*) Exploration and evaluation of coal fields.

(*2*) Establishment of mining and coal preparation capabilities.

(*3*) Coal quality evaluation: (*a*) to meet contractual obligations; (*b*) to determine compliance with municipal, state, and federal regulations; and (*c*) to generate documentation used to establish power rates or rate basis.

(*4*) Confirmation of properties pertinent to coal processing and utilization, such as direct combustion or the production of coal-derived products (for example, metallurgical coke, carbons, liquids, and gas).

(*5*) Coal research pursuits.

1. Scope

1.1 This practice is limited to the evaluation of laboratories using ASTM procedures that are under the jurisdiction of Committee D-5 on Coal and Coke. They may be used to evaluate a laboratory's capability to perform the functions for which it has been established. It is not the intention that this practice be used to evaluate capabilities beyond those specifically claimed by the laboratory.

2. Referenced Documents

2.1 *ASTM Standards:*

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 409 Test Method for Grindability of Coal by the Hardgrove-Machine Method[2]

D 1989 Test Method for Gross Calorific Value of Coal and Coke by Microprocessor Controlled Isoperibol Calorimeters[2]

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 2234 Practice for Collection of a Gross Sample of Coal[2]

D 2795 Test Methods for Analysis of Coal and Coke Ash[2]

D 2961 Test Method for Total Moisture in Coal Reduced to 2.36-mm (No. 8) Mesh Top Sieve Size (Limited-Purpose Method)[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal[2]

D 3175 Test Method for Volatile Matter in the Analysis Sample of Coal and Coke[2]

D 3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke[2]

D 3178 Test Methods for Carbon and Hydrogen in the Analysis Sample of Coal and Coke[2]

D 3179 Test Methods for Nitrogen in the Analysis Sample of Coal and Coke[2]

D 3286 Test Method for Gross Calorific Value of Coal and Coke by the Isoperibol Bomb Calorimeter[2]

D 3302 Test Method for Total Moisture in Coal[2]

D 3682 Test Method for Major and Minor Elements in Coal and Coke Ash by the Atomic Absorption Method[2]

D 4239 Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods[2]

D 4326 Test Method for Major and Minor Elements in Coal and Coke Ash by X-Ray Fluorescence[2]

---

[1] This practice is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.30 on Accreditation.

Current edition approved Sept. 10, 1997. Published May 1998. Originally published as D 4182 – 82. Last previous edition D 4182 – 92.

[2] *Annual Book of ASTM Standards*, Vol 05.05.

D 4621 Guide for Accountability and Quality Control in the Coal Analysis Laboratory[2]
D 5142 Test Methods for the Proximate, Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures[2]
D 5373 Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke[2]
D 5865 Test Method for Gross Calorific Value of Coal and Coke[2]
E 548 Guide for General Criteria Used for Evaluating Laboratory Competence[3]
E 1187 Terminology Relating to Laboratory Accreditation[3]
E 1267 Guide for ASTM Standard Specification Quality Statements[4]
E 1323 Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data[3]

3. Significance and Use

3.1 This practice provides guidelines to be followed by an individual or an accrediting body in evaluating a laboratory's capability to perform those functions only for which the laboratory claims competence. A laboratory for the purpose of this practice may be considered as any facility capable of carrying out one or more of the following functions relevant to coal or coke, or both:

3.1.1 Sampling,
3.1.2 Sample preparation,
3.1.3 Chemical analysis, and
3.1.4 Physical testing.

4. Functions

4.1 The laboratory personnel should be able to demonstrate a satisfactory knowledge of Practice D 2234 for coal or Practice D 346 for coke (or both), and the laboratory should have the capability to perform sampling in accordance with the appropriate practice.

4.2 The laboratory personnel should be able to demonstrate a satisfactory knowledge of Method D 2013 for coal or Practice D 346 for coke (or both), and the laboratory should have the capability to prepare samples in accordance with the appropriate method or practice.

4.3 The laboratory personnel should be able to demonstrate a satisfactory knowledge of all testing and analysis procedures for which the laboratory claims capability, and the laboratory should have the capability to perform those tests and analyses. The laboratory should be able to confirm that the specified equipment and reagents are available and that they are routinely used for those methods.

5. Guidelines for Selection of an Evaluator

5.1 The individual(s) selected to evaluate the competence of a laboratory to perform coal analyses by ASTM standards should be chosen primarily by experience. This evaluator should be familiar with the procedures required, as well as standard laboratory practices for quality assurance, and should have sufficient technical background to be able to comprehend the application of the appropriate procedures to the analysis of coal, coke, or coal ash. The competent evaluator should be able to communicate effectively, both orally and in writing, and should be familiar with the criteria against which the laboratory is to be evaluated.

6. Deviation from Standard Method

6.1 Documentation must be available to demonstrate that any procedures or practices that deviate from those specified in ASTM standard procedures must yield results equivalent to the appropriate ASTM standard procedures.

7. Checklist

7.1 To provide uniformity, the evaluator should review the laboratory with the aid of a worksheet or guideline. An example is provided in Appendix X2. Any worksheet or checklist used by the examiner should include at least those areas covered by Sections 1 through 7 of Appendix X2. Examples are not provided for all test methods that are under the jurisdiction of ASTM Committee D-5, nor is it the intention of ASTM Committee D-5 to provide checklists for all tests and analyses that are under its jurisdiction. The examples provided may be used as templates for checklists that can be used to evaluate tests not covered by the example checklist.

7.2 Comments made by the evaluator should be keyed to item numbers in the checklist and may be made on a separate sheet, on the margins, or on the reverse of the checklist.

7.3 The numbering of the notes to accompany the example checklist that are presented in Appendix X1 corresponds to that of the checklist. The notes describe most of the procedures that define a capable laboratory and may be used by a laboratory as a guide to establishing quality procedures and good laboratory practices.

8. Keywords

8.1 audits; calibration; certification; evaluation; laboratory; quality assurance; quality control; quality manual; quality system; traceability

[3] Annual Book of ASTM Standards, Vol. 14.02.
[4] Withdrawn. See 1995 *Annual Book of ASTM Standards*, Vol 14.02.

APPENDIXES

(Nonmandatory Information)

X1. NOTES TO ACCOMPANY THE EXAMPLE CHECKLIST (Appendix X2)

X1.1 Quality Assurance Program

X1.1.1 There should be a Quality Assurance (QA) Manual available that documents all of the components of the quality system (see Guide E 548, Sections 3.1.11 and 6.2).

X1.1.2 The QA Manual should be updated whenever any quality system provisions are changed. A section of the manual should make explicit the procedures to be followed for making the modifications and should specify who has the authority to make them.

X1.1.3 A master document control list that defines the current revision status of all referenced documents, practices, procedures, and standards used in the laboratory should be maintained and made readily available throughout the laboratory.

X1.1.4 The QA Manual should contain a summary of the QA program and

X1.1.5 A written statement of management commitment to the QA program.

X1.1.6 A QA Coordinator or Manager (sometimes assisted by a QA Committee) should be responsible for assuring that the QA program is in place and working. Generally, the Coordinator should report directly to the manager of the laboratory.

X1.1.7 The laboratory should maintain an up-to-date list of all of the sampling, preparation, testing, and analysis procedures for which it claims competence.

X1.1.8 The QA Manual should include or reference a written Standard Operating Procedure (SOP) for each procedure and test that is conducted by the laboratory (Note X1.1). Though ASTM standards should be referenced, if the laboratory deals with international customers, international standards may be referenced. If a procedure deviates from an established standard, data must be available to demonstrate that the modified procedure provides results that are equivalent to those obtained by using the standard. Simple reference to published standards, however, is generally inadequate; such reference needs to be supplemented by additional information pertaining to quality assurance and quality control activities that are frequently not covered in test procedure standards.

NOTE X1.1—The terms "Standard Operating Procedure" and "SOP" are used in this document to designate any written procedure whether it applies to a specific test procedure or a procedure that is used in another aspect of the laboratory operation including quality elements. The laboratory may refer to such documents by a name other than Standard Operating Procedure.

X1.1.9 Calibration is the set of operations that establish, under specified conditions, the relationship between values indicated by a measuring instrument, measuring system, or values represented by a material measure and the corresponding known values of a measurand (Guide E 548).

X1.1.9.1 Where appropriate, procedures employed for calibrating the equipment used should be defined in an SOP. It should include a value for an acceptable deviation and procedures for dealing with results that exceed the acceptable deviations.

X1.1.9.2 A schedule should be maintained that designates when calibration is to be performed on each piece of equipment that requires it (see X1.1.11 following).

X1.1.9.3 Procedures should be defined for establishing traceability of reference materials (RMs, Note X1.2), including in-house control samples (Guide D 4621, Section 3.1.7 and Appendix X2), to certified reference materials (Note X1.3). See also Guide E 548, Section 3.1.13 and Guide E 1267, Sections 3.2.2 and 5.2.2 for discussions of traceability.

NOTE X1.2—Reference material: a material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to a material (Terminology E 1187).

NOTE X1.3—Certified reference material: a reference material for which one or more property values are certified by a technically valid procedure accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

X1.1.10 Procedures for dealing with instances of nonconformance when analyzing RMs or control samples should include the following issues:

X1.1.10.1 The SOP should define statistically based criteria for recognizing nonconformance, including numerical limits that require action when exceeded (namely, "action limits," Guide D 4621, Appendix X1). See also Guide E 1323 for guidance on statistical analysis practices.

X1.1.10.2 It should also define specific procedures to be followed to investigate the cause of nonconformance.

X1.1.10.3 It should define procedures to correct the SOP or equipment.

X1.1.10.4 A description of nonconformance occurrences and actions taken should be documented in a permanent manner.

X1.1.10.5 Procedures should be defined to validate that any modification does accomplish the objective and that the process is under control.

X1.1.10.6 Specific procedures should be modified or instituted to prevent recurrence of the nonconformance.

X1.1.10.7 Authority to make modifications of equipment or SOPs should rest with only one designated person (or, at most, only a few persons). This policy should be enforced to prevent casual tampering with or modification of equipment and procedures, especially when nonconforming results are detected. Authority to appoint deputies or substitutes should reside only with one individual.

X1.1.11 There should be a calendar made up in advance that lists the routine QA activities that will be undertaken, such as recalibration, audits, equipment maintenance, and so forth.

X1.1.12 There should be a written policy for continuous improvement.

X1.1.13 As a part of this activity, there should be procedures for soliciting customer feedback and dealing with customer complaints.

X1.1.14 If the laboratory is certified or registered by a recognized agency, documents attesting to same should be readily available.

X1.1.15 Provisions should be in place to protect the confidentiality of data, including data residing on any computer to which unauthorized persons have access.

X1.1.16 There should be a clearly stated policy that provides for notification of a customer whenever any reported result becomes suspect because of the detection of a problem in the laboratory.

X1.2 Staff

X1.2.1 Information about the staff should be readily available to an evaluator. The QA Manual is an appropriate place for such information. The information should include (at minimum):

X1.2.1.1 An organization chart and

X1.2.1.2 Descriptions of responsibilities, especially for quality assurance. Deputies should be specified for critical authorities.

X1.2.2 Some judgment should be applied to evaluating whether there is adequate staff to handle the routine workload.

X1.2.3 Documentation should be available to demonstrate that:

X1.2.3.1 There is a formal training program in quality assurance and quality control methods and that this knowledge has been routinely refreshed.

X1.2.3.2 There is a formal program to train the technical staff to carry out the procedures for which the laboratory claims competence and that there is an ongoing program for evaluating the staff competence.

X1.2.4 Documentation should be available defining the technical capabilities of each staff member, including formal training that each has received and years of experience.

X1.3 Facilities

X1.3.1 Facilities should be adequate to perform the activities that the laboratory claims as its capabilities, including the following areas:

X1.3.1.1 Lighting should be adequate to see equipment and read instruments clearly.

X1.3.1.2 Temperature and humidity should be controlled where it is required by a procedure.

X1.3.1.3 Space should be adequate.

X1.3.1.4 Hoods should be available where fumes are emitted.

X1.3.1.5 There should be adequate sinks and water fixtures and

X1.3.1.6 Electrical outlets.

X1.3.2 The laboratory should be clean and orderly. Equipment should be maintained in a clean fashion.

X1.3.3 Safety and health policies should be documented, and the documents should be readily available. Where appropriate, they should be posted. The evaluator should make an effort to ascertain that the staff is familiar with safety and health policies and that they practice them.

X1.3.4 To be able to fulfill commitments should some facet of the facilities become inoperable, there should be provisions to use contractor services or other facilities to make analyses. Or, there should be well-defined provisions for bringing in qualified repair persons to provide immediate repairs to the facilities.

X1.3.5 ASTM standards and other written procedures (SOPs) applicable to the laboratory's activities should be readily available to the staff.

X1.3.6 Access to and use of facilities should be defined and controlled.

X1.4 Quality Control

X1.4.1 Quality control (QC) procedures that cover at the least the following items should be established. There should be a document that summarizes the QC program.

X1.4.2 Test sanples:

X1.4.2.1 The test or analysis sample should be traceable to the submitter by a formal chain of custody from intake through the analysis or test procedures.

X1.4.2.2 There should be a permanent log containing all important information about a sample.

X1.4.2.3 Each sample should be a given a unique laboratory identifier which should appear on all subsamples and documents.

X1.4.3 All data reported to the customer should be traceable to the initial receipt of the sample. The evaluator should select a few final reports at random and determine that sample and data traceability is adequate. All worksheets or similar documents should include the sample identifier. Personnel who carried out the activities should be identified on the documents to provide a source for information should a problem be suspected. Between any two documents reviewed, there should be no deviation of information identifying the sample or preceding test results.

X1.4.4 Reference materials should be tested according to a specific schedule (see X1.1.9).

X1.4.4.1 For all test calibrations, data (including operator's identification) should be maintained in a permanent log and kept current in statistical form or control chart form or both so that anyone can see immediately what the condition of standardization is for a given test.

X1.4.4.2 Written policies should define at what point of nonconformance action needs to be taken to correct equipment or procedures to assure that a test procedure is in calibration (see previous Section X1.1.10 and Action Limits, Guide D 4621).

X1.4.5 Reference materials (RM) should be stable material of appropriate top size with an appropriate matrix and, when used as primary standards (see Guide D 4621, Section 7), should be traceable to Certified Reference Materials (CRMs). It is desirable to compare results from a new sample with the old one before the new one is used as the standard. Permanent records should be maintained of such actions in the log book. See also X1.1.9 and X1.4.4.

X1.4.6 Proficiency test:

X1.4.6.1 The laboratory should participate in at least one interlaboratory proficiency testing (PT) program, sometimes referred to as a "round robin" testing program. Permanent

records should be maintained of proficiency tests and other sample exchange or interlaboratory programs.

X1.4.6.2 There should be an SOP that defines how the participation in an interlaboratory proficiency testing program is conducted and administered.

X1.4.6.3 Reproducibility data from such programs which may indicate whether the laboratory produces accurate (unbiased) results should be maintained in a permanent file and summarized in statistical form or on control charts or both. Examine data for evidence of any persistent deviations from proficiency test averages that could indicate a bias. Breathing a sigh of relief that the laboratory's results are within some arbitrary range of the proficiency test mean value on a month-to-month basis is not a satisfactory use of proficiency test data.

X1.4.6.4 Written policies should define the statistically based level of nonconformance at which action needs to be taken to modify or recalibrate a procedure based on performance in the proficiency test program (see Section X1.1.10). Records of such actions should be documented. Responsibilities should be explicitly defined.

X1.4.7 Control samples:

X1.4.7.1 Internal control samples should be tested on a routine basis (see Guide D 4621, Appendix X2). A control sample might be a RM, a sample that has a known composition traceable to a RM, a customer sample analyzed in replicate, or a fresh proficiency test sample. Control samples should be stable materials of appropriate top size with an appropriate matrix. Retesting a sample analyzed the previous day or shift is also recommended.

X1.4.7.2 The procedures for using a control sample should be specified in an SOP.

X1.4.7.3 Data from control samples should be preserved in a permanent log or file and summarized in statistical or control chart form. Data from replicate analyses of control samples (or customer samples) are used to define precision (repeatability) performance, and these measures should be routinely updated and readily available.

X1.4.7.4 Written policies should define statistically significant nonconformance levels requiring action to be taken to investigate the accuracy of a procedure (see Guide D 4621, Appendix X1). Responsibility for reviewing control sample results should be explicitly specified, as should responsibility for taking appropriate corrective actions. Records of such actions should be documented.

X1.4.8 There should be a written policy that defines how long data should be retained. This policy should cover:

X1.4.8.1 Calibration and control data and

X1.4.8.2 Routine test data.

Generally, one year is considered adequate for routine test data, longer for calibration and control data.

X1.4.9 Calculations:

X1.4.9.1 For ease of reference and to facilitate auditing, a single document that shows all calculation procedures used for all tests should be included in the QA Manual. There should be a process to ensure that the procedures are rigidly followed. If calculations are performed on a computer, a printout should be available of the appropriate parts of the program or of the results of a test program to demonstrate that the calculations are programmed correctly.

X1.4.9.2 Unless equipment sends data directly to a computer, there should be a specific worksheet used to record data for each test procedure.

X1.4.9.3 Some procedure should be used to ensure accurate data input to the worksheet or computer.

X1.4.10 Date review, validation, and retesting:

X1.4.10.1 All final data should be reviewed and validated before being transmitted to the customer. Often, the validity of data can be checked by assessing whether different parameters are consistent with each other. For instance, laboratories that analyze coal from one source can check whether specific energy (calorific value) calculated to a dry, ash-free basis is consistent with an average from earlier analyses. Determined specific energy should agree with SE calculated from elemental analyses.

X1.4.10.2 There should be one individual responsible for this validation activity, and that individual should also have the authority, if incorrect data are suspected, to institute retesting or to check calibrations.

X1.5 Audits

X1.5.1 Quality assurance and quality control activities should be formally audited or evaluated on a routine basis. There should be a document in the QA Manual that describes the audit policy.

X1.5.2 Internal:

X1.5.2.1 Internal audits should be conducted by personnel not directly involved with the activities. Internal audits can be carried out with this checklist.

X1.5.2.2 They should be carried out on a fairly frequent basis (for example, quarterly).

X1.5.3 Audits by external examiners can be conducted on a more long-term basis (for example, yearly).

X1.5.4 There should be a policy that provides for an annual review of the quality assurance program by the laboratory management. This is commonly done in conjunction with an audit. The policy should be written and should define specifically the procedures that management should follow to maximize the effectiveness of the review.

X1.5.5 A written procedure should define who is responsible for taking action when an audit identifies a problem. This procedure should also define who is responsible for assuring that the action is taken. There should be a specific "sign-off" process.

X1.5.6 Records should be permanently retained of the results of audits. Specific actions taken to correct deficiencies should be documented.

X1.6 Equipment and Supplies

X1.6.1 There should be a single file system documenting all critical supplies and equipment, including identification information (for example, inventory numbers, serial numbers), date of acquisition, source, date and condition when received, repair history, maintenance history, and so forth.

X1.6.2 Manuals for all pieces of equipment should be kept in a well-defined repository so that they are not lost.

X1.6.3 Critical parts and supplies should be inventoried, and an updated inventory list should be readily available.

X1.6.4 Provisions should be made to assure that the inventory list is used to reorder critical parts and supplies in time to keep the operation functional.

X1.6.5 To keep the laboratory operating if equipment breaks down, there should be provisions to use contractor services or facilities to perform tests or analyses. Alternatively, there should be provisions for repair persons to be called upon to provide immediate repairs.

X1.6.6 Maintenance:

X1.6.6.1 Scheduled maintenance should be listed on a calendar, performed in a timely fashion, and permanently documented when completed.

X1.6.6.2 A checklist of maintenance activities should be available near the equipment, and an additional document should be preserved in a central repository.

X1.6.7 A copy of calibration and check or control sample data should be kept near the equipment to which they apply. The status of calibration should be indicated on all equipment.

X1.6.8 A strictly enforced policy should prevent unauthorized tampering with or modification of equipment and procedures, especially when nonconformance is suspected. Modifications should be performed only after a well-defined statistically valid measure of nonconformance is detected, and modifications must be approved by only specified supervisory personnel, who must document the problem and their approval.

X1.6.9 There should be a written policy that describes the procedure for removing faulty equipment from service, for repairing or replacing it, and for recertifying repaired equipment.

X1.6.10 Balances:

X1.6.10.1 Balances should have sufficient sensitivity and accuracy to be appropriate for the activities in which they are used.

X1.6.10.2 Balance calibration should be checked routinely, and documents maintained of the results.

X1.7 Samples and Sample Preparation

X1.7.1 Staff familiarity:

X1.7.1.1 If the laboratory carries out sampling, it should be able to demonstrate that the staff engaged in sampling is adequately trained to accomplish the tasks according to ASTM methods or practices (for example, Practice D 2234).

X1.7.1.2 The staff should demonstrate the capability to prepare samples according to Method D 2013 (for coal) or Practice D 346 (for coke) or both.

X1.7.2 Samples:

X1.7.2.1 Samples should be expeditiously handled.

X1.7.2.2 Labels should be applied in such a manner that they remain legible and cannot be inadvertently removed, switched, or misapplied.

X1.7.3 The sample preparation area should be:

X1.7.3.1 Clean and

X1.7.3.2 Draft free.

X1.7.3.3 If the climate warrants, temperature and humidity should be controlled to minimize inadvertent changes in moisture content. Under no circumstances should samples be prepared in direct sunlight or precipitation.

X1.7.4 Equipment:

X1.7.4.1 Equipment for size reduction and sample division should be appropriate for the task. Riffles should be tightly enclosed to prevent dust loss and have correct chute size (see Method D 2013).

X1.7.4.2 All sample material should be recovered from equipment such as riffles, crushers, and pulverizers to prevent creation of a biased sample.

X1.7.4.3 Size-reduction and sample division equipment should be thoroughly cleaned after each use, preferably by vacuuming (the use of an air blast to clean equipment is not recommended). Thorough cleaning is especially important where only small quantities of sample are being processed.

X1.7.5 Sample integrity should be preserved while samples are being processed or while they are awaiting processing. In particular, samples should be protected from temperature extremes, moisture changes, and oxidation.

X1.7.6 Air dry procedure:

X1.7.6.1 In some laboratories, when samples are known to be fairly consistent, it has become common practice to carry out air drying for a fixed time interval rather than by measuring the rate of weight loss. In those instances, data should be available to demonstrate that the time interval is appropriate for achieving stable moisture content with that coal.

X1.7.6.2 The air-drying oven temperature should be less than 104°F (40°C).

X1.7.6.3 The oven temperature should be monitored by some method independent of that used to control the temperature.

X1.7.6.4 There should be an adequate flow of air (1 to 4 volume exchanges per minute) across the samples.

X1.7.6.5 The air-drying pan should be noncorroding, and the coal depth in the pan should not exceed 1 in. (25 mm).

X1.7.7 Although not mandatory, it is highly desirable to conduct tests to assess the magnitude of any incidental moisture changes that might occur during routine sample preparation. The protocol for such tests is left to the individual laboratory. If tests are conducted, results should be documented and made accessible in an easily understood form.

X1.7.8 Tests should be performed to measure the magnitude of the variance attributable to routine sample preparation procedures (see Annexes in Method D 2013).

X1.7.9 Weights of samples at each stage of division must be in accord with Method D 2013.

X1.7.10 Particle sizes of crushed or pulverized samples should be checked on a routine basis to assure that samples meet the appropriate size criteria.

X1.7.11 Pulverized samples:

X1.7.11.1 Pulverized samples should be mixed on a mixing wheel (to improve mixing, some laboratories place a child's toy jack or "jack rock" in the container).

X1.7.11.2 Pulverized samples should be mixed and maintained in well-sealed bottles or other containers until analyzed. It is desirable to perform some tests to demonstrate that the container lids do seal well, thereby preventing moisture changes and oxidation. A convenient test is to determine, by weighing, if water evaporates when sealed in a typical container.

X1.7.11.3 Samples should not be unduly shaken or tapped after mixing to minimize subsequent size segregation.

X1.7.12 There should be a written policy for sample retention procedures. The routine execution of the policy should be demonstrable to the evaluator.

X1.7.13 The laboratory should be able to demonstrate that retained samples maintain their integrity for an appropriate period of time. All retained samples will deteriorate during storage, and this fact must be recognized. Plastic bags, in particular, are poor storage vehicles because they are permeable, allowing oxidation and moisture changes. Changes can be minimized by storing samples in well-sealed solid containers (see Section X1.7.11.2) or Mylar- or foil-lined bags.

X1.8 Moisture in the Analysis Sample

X1.8.1 The procedure should conform to Test Methods D 2961, D 3173, D 3302, or D 5142.

X1.8.2 The drying oven should have a minimum of free air space.

X1.8.3 Atmosphere:

X1.8.3.1 When requested by a customer, some laboratories use nitrogen as the atmosphere in the drying oven.

X1.8.3.2 The flow rate should renew the atmosphere at the rate of at least two times per minute.

X1.8.3.3 The atmosphere should be dried through a desiccant and reheated.

X1.8.4 Temperature:

X1.8.4.1 The temperature in the drying oven should be calibrated by a temperature-measuring device that is independent of that used routinely to monitor or control temperature. This calibration should be carried out on a fairly frequent basis (for example, weekly). Records should be maintained.

X1.8.4.2 The uniformity of temperature across the oven should be assessed.

X1.8.4.3 Every day (or while each batch of samples is being dried), the temperature should be monitored with a permanently installed thermometer or thermocouple and a record kept of the results. It is inadequate to rely solely on the temperature control device as a monitoring procedure.

X1.8.4.4 The temperature recovery rate for the oven should be determined.

X1.8.5 Samples should be handled expeditiously to minimize exposure to ambient air once they are removed from the sample container.

X1.8.6 Dried samples should be rapidly placed in a desiccator or desiccator cabinet for cooling.

X1.8.7 Some form of control sample should be analyzed on a routine basis to assure that the procedure provides accurate data (see Section X1.4.7 for further information on the use of control samples). Some laboratories analyze a control sample with each batch of customer samples. Data should be documented and summarized in statistical or control chart form or both.

X1.8.8 Results from interlaboratory proficiency tests (PT) of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.8.9 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard.

X1.9 Ash in the Analysis Sample

X1.9.1 The procedure should conform to Test Methods D 3174 or D 5142.

X1.9.2 The air flow rate in the ashing furnace should change the atmosphere from 2 to 4 times per minute.

X1.9.3 The method used to increase the temperature in the ashing furnace should be consistent with the requirements of Test Method D 3174 and should be consistent from test to test.

X1.9.4 Temperture:

X1.9.4.1 The temperature in the furnace should be calibrated routinely according to the procedure described in Section 6.1 of Test Method D 3174. The temperature should be calibrated by a temperature-measuring device that is independent of that used routinely to monitor or control temperature. This calibration should be carried out on a fairly frequent basis (for example, weekly). Records should be maintained.

X1.9.4.2 Every day (or while each batch of samples is being burned) the temperature should be monitored with a permanently installed measuring device (for example, thermocouple) and a record kept of the results. It is inadequate to rely solely on the temperature control device as a monitoring procedure.

X1.9.5 Sample:

X1.9.5.1 Some laboratories remove the sample, stir it, and replace for final combustion. This procedure is useful for samples that are difficult to combust.

X1.9.5.2 All combusted samples should be checked for unburned carbon.

X1.9.5.3 Provisions should be in place for reburning the sample or discarding it when carbon is detected.

X1.9.6 Burned samples should be handled and cooled in a manner to minimize absorption of moisture from the atmosphere.

X1.9.7 Some form of control sample should be analyzed on a routine basis to assure that the procedure provides accurate data (see Section X1.4.7 for further information on the use of control samples). Some laboratories analyze a control sample with each batch of customer samples. Data should be documented and summarized in statistical or control chart form or both.

X1.9.8 Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.9.9 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM Standard.

X1.10 Volatile Matter in the Analysis Sample

X1.10.1 The procedure should conform to Test Methods D 3175 or D 5142. The modified method should be used where appropriate.

X1.10.2 Some laboratories use crucibles other than platinum as specified in Test Method D 3175. The lids must fit tight enough to prevent access of air which could burn some of the fixed carbon. It is desirable to test this in an experimental program.

X1.10.3 Temperature:

X1.10.3.1 The temperature should be calibrated routinely by a temperature-measuring device that is independent of that used routinely to monitor or control. This is commonly accomplished with a thermocouple immersed in sand in a crucible. The calibration check should be carried out on a fairly frequent basis (for example, weekly). Records should be maintained.

X1.10.3.2 The temperature should be monitored every day (or more frequently) with a permanently installed thermocouple and a record kept of the results. It is inadequate to rely solely on the temperature control device as a monitoring procedure.

X1.10.4 Some form of control sample should be analyzed on a routine basis to assure that the procedure provides accurate data (see Section X1.4.7 for further information on the use of control samples). Data should be documented in statistical or control chart form or both.

X1.10.5 Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.10.6 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard.

X1.11 Sulfur in the Analysis Sample

X1.11.1 The procedure should conform to Test Methods D 3177 or D 4239.

X1.11.2 Reference materials (RMs, either primary or secondary) should be analyzed to assure that the procedure yields accurate results or to calibrate the instrument (Test Method D 4239). Data from RMs should be available for inspection.

X1.11.3 For the instrumental method, there should be a policy in place for monitoring and replacing absorbents in the system.

X1.11.4 If blanks are run, data should be documented, and the record preserved.

X1.11.5 Some laboratories routinely replicate all analyses, a good practice.

X1.11.6 Some form of control sample should be analyzed on a routine basis to ensure that the procedure provides accurate data (see Section X1.4.7 for further information on the use of control samples).

X1.11.7 Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.11.8 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard.

X1.12 Carbon, Hydrogen, and Nitrogen in the Analysis Sample

X1.12.1 The procedure should conform to Test Methods D 3178, D 3179, or D 5373.

X1.12.2 Temperature:

X1.12.2.1 The temperature in the combustion furnace should be calibrated routinely by a temperature-measuring device that is independent of that used routinely to monitor or control. This calibration should be carried out on a fairly frequent basis (for example, weekly). Results should be preserved in a permanently maintained document.

X1.12.2.2 The temperature should be monitored every day with a permanently installed thermocouple, and a record kept of the results. It is inadequate to rely solely on the temperature control device as a monitoring procedure.

X1.12.3 Reference materials (RMs, either primary or secondary) should be analyzed to assure that the procedure yields accurate results or to calibrate the instrument (Test Method D 5373). Data from RMs should be available for inspection.

X1.12.4 There should be a policy in place for monitoring and replacing absorbents in the system.

X1.12.5 If blanks are run, data should be documented, and the record preserved.

X1.12.6 Some form of control sample should be analyzed on a routine basis to assure that the procedure provides accurate data (see Section X1.4.7 for further information on the use of control samples).

X1.12.7 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard. Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.13 Gross Calorific Value in the Analysis Sample

X1.13.1 The procedure should conform to Test Methods D 1989, D 2015, D 3286, or D 5865.

X1.13.2 This test should be run only by operators who have had adequate training and experience.

X1.13.3 Standardization:

X1.13.3.1 The calorimeter heat capacity should be checked once per month at a minimum (many laboratories check on a more frequent basis).

X1.13.3.2 The policy described under the section entitled“ Standardization,” “Restandardization,” or “Heat Capacity Check” in the appropriate standard method should be followed to indicate when it is necessary to determine a new heat capacity value. If a different policy is used, it should meet at least the stringency of the procedure defined in the standard method.

X1.13.4 The calibration reference material should be certified for its calorific value by the issuing organization.

X1.13.5 There should be a policy in place that assures that a sufficient quantity of reference material is always available.

X1.13.6 If sooty deposits or unburned carbon are detected after the bomb is opened, the results should be discarded, and the sample should be reanalyzed.

X1.13.7 Acid correction:

X1.13.7.1 The titrant should be prepared from reagent grade chemicals and

X1.13.7.2 May be tested for strength by titrating against another standard solution.

X1.13.8 Firing medium:

X1.13.8.1 Optional forms of ignition media are permitted.

X1.13.8.2 Unless palladium or platinum wire is used, a correction should be made for the heat of combustion of the ignition medium.

X1.13.9 Net CV:

X1.13.9.1 Frequently, a value for the net calorific value is calculated by the laboratory. If so, the method should conform to the procedure specified in ASTM standards. If a different procedure is used, it should be described in the report.

X1.13.9.2 In some instances, the hydrogen content of the sample may be estimated for use in the net calorific value calculation. The procedure to do so should be described in the report.

X1.13.10 Bombs should be tested (inspected) on a routine basis by a qualified agency, and a record should be maintained of the results.

X1.13.11 If control samples are run on a routine basis, the data should be available for inspection.

X1.13.12 Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.13.13 Repeatability and reproducibility data achieved by the laboratory should be updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard.

X1.14 Elements in Ash

X1.14.1 The procedure should conform to Test Methods D 2795, D 3682, or D 4326.

X1.14.2 If the digestion procedure differs from that described in Test Methods D 2795 or D 3682, it should be demonstrated that it solubilizes all of the ash.

X1.14.3 Reference materials (RMs, either primary or secondary) should be analyzed routinely to assure that the procedure yields accurate results or to calibrate any instrument used.

X1.14.4 Data from RMs, summarized statistically, should be available for inspection.

X1.14.5 If control samples are run on a routine basis, the data should be summarized statistically and available for inspection.

X1.14.6 Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.14.7 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard.

X1.15 Fusibility of Ash

X1.15.1 The proportion of reducing gas should be between 20 and 80 % by volume of the atmosphere in the furnace.

X1.15.2 The flow rate of the atmosphere should be 1.3 to 1.5 furnace volumes per minute.

X1.15.3 The rate of temperature rise in the furnace should be 15 ± 5°F (8 ± 3°C).

X1.15.4 The furnace temperature-measuring device should be checked at least once during each week of operation with gold and nickel wire. Results should be maintained in a log.

X1.15.5 If control samples are run on a routine basis, the data should be available for inspection.

X1.15.6 Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.15.7 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard.

X1.16 Grindability by the Hardgrove Method

X1.16.1 Equipment:

X1.16.1.1 Equipment must be appropriate, clean, and in working condition.

X1.16.1.2 Screens used in the grindability test should be dedicated to that use only.

X1.16.2 Calibration:

X1.16.2.1 Test Method D 409 requires calibration whenever equipment is new, modified, repaired, or suspected of being defective. Reference materials (RMs) used in calibration should be less than 18 months old.

X1.16.2.2 It is good practice to check the calibration periodically with one or more RMs.

X1.16.3 The sample must be air dried strictly in accord with procedures specified in Test Method D 2013. Overdrying or underdrying will produce inaccurate results.

X1.16.4 Because HGI can vary with the moisture content of some coals, it is good practice to determine and report the moisture content of the sample as it is run in the Hardgrove apparatus.

X1.16.5 If control samples are run on a routine basis, the data should be summarized statistically and available for inspection.

X1.16.6 Results from interlaboratory proficiency tests of the procedure should be summarized statistically and available for inspection (see Section X1.4.6).

X1.16.7 Repeatability and reproducibility data achieved by the laboratory should be routinely updated and displayed in a statistically useful fashion and should fall within specifications defined in the appropriate ASTM standard.

X2. CHECKLIST FOR EVALUATING A COAL/COKE ANALYSIS LABORATORY

X2.1 Fig. X2.1 is a checklist for evaluating a coal/coke analysis laboratory.

CHECKLIST FOR EVALUATING A COAL/COKE ANALYSIS LABORATORY

Evaluator_____ Date_____

Laboratory Identification_____

Address_____

Contact_____ Phone_____

X1.1 QUALITY ASSURANCE (QA)

X1.1.1 QA manual current_____ X1.1.2 Provisions for modifying_____ X1.1.3 Document control list_____

X1.1.4 Written QA policy_____ X1.1.5 Managment committed to QA_____

X1.1.6 Quality coordinator/committee_____

X1.1.7 List of competencies claimed_____ X1.1.8 Stand. Operat. Procs. (SOPs) written_____

X1.1.9 Calibration: X1.1.9.1 SOPs_____ X1.1.9.2 Schedule available_____ X1.1.9.3 Ref. mater. traceable_____

X1.1.10 Dealing with nonconformance during calibration or tests with control samples:

X1.1.10.1 Statistical criteria_____

X1.1.10.2 Identify causes_____ X1.1.10.3 Policy to correct_____

X1.1.10.4 Documented_____ X1.1.10.5 Assure conforming now_____ X1.1.10.6 Recurrence prevented_____

X1.1.10.7 Authority to modify equipment/SOPs limited to:_____

X1.1.11 Calendar for routine QA activities_____

X1.1.12 Continuous improvement policy_____ X1.1.13 Customer input policy_____

X1.1.14 Certified/registered by_____

X1.1.15 Confidentiality protected_____ X1.1.16 Policy to notify Customers of errors_____

X1.2 STAFF

X1.2.1 Staff information:

X1.2.1.1 Organization chart_____ X1.2.1.2 Responsibilities defined_____

X1.2.2 No. Supervisors_____ No. Analysts_____ Adequate for volume_____

X1.2.3 Documentation:

X1.2.3.1 QA training program/refreshers_____ X1.2.3.2 Formal technical training/testing_____

X1.2.4 Staff qualifications/training documented_____ Yrs. experience_____

X1.3 FACILITIES (THE ENVIRONMENT)

X1.3.1 Adequate: X1.3.1.1 Light_____ X1.3.1.2 Temp./humid_____ X1.3.1.3 Space_____

X1.3.1.4 Hoods_____ X1.3.1.5 Sinks_____ X1.3.1.6 Electrical outlets_____

X1.3.2 Housekeeping_____

X1.3.3 Safety/health policies written and followed_____

X1.3.4 Alternatives if facilities temporarily unusable_____

X1.3.5 ASTM standards available_____ X1.3.6 Lab access limited_____

FIG. X2.1 Checklist for Evaluating a Coal/Coke Analysis Laboratory

X1.4 QUALITY CONTROL

X1.4.1 Quality control program documented_____

X1.4.2 Test samples: X1.4.2 Traceable_____ X1.4.2.2 Logged_____ X1.4.2.3 Unique ID_____

X1.4.3 Data traceable_____

X1.4.4 Calabrations: X1.4.4.1 Log_____ Stat._____ Charts_____ X1.4.4.2 Action limits/policy defined_____

X1.4.5 RMs are appropriate_____

X1.4.6 Proficiency test: X1.4.6.1 With_____ X1.4.6.2 SOP:_____

X1.4.6.3 File_____ Stat._____ Charts_____ X1.4.6.4 Action limits/policy defined_____

X1.4.7 Control samples: X1.4.7.1 Types_____ X1.4.7.2 SOP:_____

X1.4.7.3 Log_____ Stat._____ Charts_____ X1.4.7.4 Action limits/policy defined_____

X1.4.8 Data maintained how long: X1.4.8.1 QC/QA_____ X1.4.8.2 Test data_____

X1.4.9 Calculations: X1.4.9.1 documented_____ X1.4.9.2 Procedure worksheets_____ X1.4.9.3 Accurate input_____

X1.4.10 Data review, validation, retesting:

X1.4.10.1 Written procedures defined/followed_____

X1.4.10.2 Authority/responsibility defined_____

X1.5 AUDITS

X1.5.1 Audit policy: X1.5.1.1 Defined_____ X1.5.1.2 SOP_____

X1.5.2 Internal: X1.5.2.1 Who_____ X1.5.2.2 Freq._____

X1.5.3 External: X1.5.3.1 Who_____ X1.5.3.2 Freq._____

X1.5.4 Annual management review_____

X1.5.5 Action policy/authority written_____ X1.5.6 Document available_____

X1.6 EQUIPMENT AND SUPPLIES

X1.6.1 List available_____ X1.6.2 Manuals nearby_____

X1.6.3 Inventory record_____ X1.6.4 Reorder provisions_____

X1.6.5 Alternatives if equipment unusable_____

X1.6.6 Maintenance: X1.6.6.1 Schedule_____ X1.6.6.2 Records_____

X1.6.7 Calibration/check data kept with equipment_____

X1.6.8 Modification/adjustment OK restricted to:_____

X1.6.9 Policy for removing and recertifying defective equipment_____

X1.6.10 Balances X1.6.10.1 Appropriate_____ X1.6.10.2 Checked routinely_____

FIG. X2.1 *(continued)*

X1.7 SAMPLES AND SAMPLE PREPARATION

X1.7.1 Staff familiar with: X1.7.1.1 Sampling procedures_____ X1.7.1.2 Preparation procedures_____

X1.7.2 Samples: X1.7.2.1 Expeditiously handled_____ X1.7.2.2 Well labeled_____

X1.7.3 Prep. area: X1.7.3.1 Clean_____ X1.7.3.2 Draft-free_____ X1.7.3.3 Temp./hum. controlled_____

X1.7.4 Equipment: X1.7.4.1 Appropriate_____ X1.7.4.2 Emptied_____ X1.7.4.3 Clean_____

X1.7.5 Sample integrity maintained_____

X1.7.6 Air dry procedure X1.7.6.1 If timed, data indicate appropriateness_____

X1.7.6.2 Oven temp. <104°F (40°C)_____ X1.7.6.3 Monitored how/freq._____

X1.7.6.4 Air exchange 1–4 vol./min_____ X1.7.6.5 Pan/coal depth (<1 in./25mm)_____

X1.7.7 Test for inadvertent drying conducted_____

X1.7.8 Variance of division data_____

X1.7.9 Weight appropriate: 4 m (2 or 4 kg)_____ 8 m (0.5 or 1 kg)_____ 60 m (50 g)_____

X1.7.10 Size checked from: 4 m/8 m mills_____ 60 m Pulverizer_____

X1.7.11 Pulv. samples: X1.7.11.1 Mixed on wheel_____ X1.7.11.2 Sealed well_____ X1.7.11.3 Not shaken_____

X1.7.12 Sample retain policy_____

X1.7.13 Sample retains adequately sealed/protected_____

8. MOISTURE ANALYSIS

X1.8.1 Method(s) employed_____

X1.8.2 Oven type/free space_____

X1.8.3 Atmosphere: X1.8.3.1 Air_____ N2___ X1.8.3.2 Exchange 2 vol/min_____ X1.8.3.3 Dried_____ X1.8.3.4 Reheated_____

X1.8.4 Temp: X1.8.4.1 Calibrated how/freq._____ X1.8.4.2 Uniform_____

X1.8.4.3 Monitored how/freq._____ X1.8.4.4 Recovery_____

X1.8.5 Handled rapidly_____ X1.8.6 Desiccator OK_____

X1.8.7 Control sample freq./results_____ X1.8.8 PT[1] results_____

X1.8.9 Repeatability_____ Reproducibility_____

X1.9 ASH ANALYSIS

X1.9.1 Method(s)_____ X1.9.2 Air exchange 2–4 vol/min/min_____

X1.9.3 Temp.: to 450–500°C in 1 hr_____ 700–750°C in 2 hrs_____

X1.9.4 Temp.: X1.9.4.1 Calibrated how/freq._____

X1.9.4.2 Monitored how/freq._____

Note: 1: PT — Proficiency Test

FIG. X2.1 *(continued)*

X1.9.5 Sample: X1.9.5.1 Stirred_____ X1.9.5.2 Check for Carbon_____ X1.9.5.3 Reburned/discarded_____

X1.9.6 Handled rapidly_____ Cooled how_____

X1.9.7 Control sample freq./results_____ X1.9.8 PT results_____

X1.9.9 Repeatability_____ Reproducibility_____

X1.10 VOLATILE MATTER ANALYSIS

X1.10.1 Procedure_____ X1.10.2 Crucible type_____

X1.10.3 Temp.: X1.10.3.1 Calibrated how/freq._____

X1.10.3.2 Monitored how/freq._____

X1.10.4 Control sample freq./results_____ X1.10.5 PT results_____

X1.10.6 Repeatability_____ Reproducibility_____

X1.11 SULFER ANALYSIS

X1.11.1 Method(s)_____

X1.11.2 RMs used/freq._____

X1.11.3 Absorber fill policy_____

X1.11.4 Blanks run_____ X1.11.5 Samples duplicated_____

X1.11.6 Control sample freq./results_____ X1.11.7 PT results_____

X1.11.8 Repeatability_____ Reproducibility_____

X1.12 C/H/N ANALYSIS

X1.12.1 Method(s)_____

X1.12.2 Temp.: X1.12.2.1 Calibrated_____ X1.12.2.2 Monitored_____

X1.12.3 RMs used/freq._____

X1.12.4 Absorber fill policy_____

X1.12.5 Blanks run_____ Samples duplicated_____

X1.12.6 Control sample freq/data_____

X1.12.7 Precision:

	Repeatability	*Reproducibility*	*Proficiency test*
Carbon	_____	_____	_____
Hydrogen	_____	_____	_____
Nitrogen	_____	_____	_____

X1.13 GROSS CALORIFIC VALUE

X1.13.1 Method(s)_____

X1.13.2 Operator experience_____

FIG. X2.1 *(continued)*

X1.13.3 Standardization check: X1.13.3.1 Frequency_____ X1.13.3.2 Procedure_____

X1.13.4 Calibration sample_____ X1.13.5 Inventory policy_____

X1.13.6 Checked for unburned carbon_____

X1.13.7 Acid correction X1.13.7.1 Titrant_____ N=_____ Finish_____

X1.13.7.2 Titrant standardized_____

X1.13.8 Firing medium X1.13.8.1 Material_____ X1.13.8.2 Correction made_____

X1.13.9 Net CV: X1.13.9.1 Formula_____

X1.13.9.2 Hydrogen estimated_____

X1.13.10 Bomb test frequency_____ X1.13.10.1 Maintenance record available_____

X1.13.11 Control sample freq./results_____ X1.13.12 PT results_____

X1.13.13 Repeatability_____ Reproducibility_____

X1.14 ASH ELEMENTS

X1.14.1 Method(s)_____

X1.14.2 Digestion procedure_____

X1.14.3 Calibrate how/freq._____ X1.14.4 RMs_____

X1.14.5 Control samples: X1.14.5.1 Type_____ X1.14.5.2 Freq._____

X1.14.6 Proficiency test results_____

X1.14.7 Repeatability_____

Reproducibility_____

X1.15 ASH FUSION

X1.15.1 Reducing gas 20–80 vol %_____ X1.15.2 Flow 1.3–1.5 exchange/min_____

X1.15.3 Temp. rise/min. 15.5±5°F (8±3°C)_____ X1.15.4 Temp. check freq._____

X1.15.5 Control sample freq./results_____ X1.15.6 PT results_____

X1.15.7 Repeatability_____ Reproducibility_____

X1.16 GRINDABILITY

X1.16.1 Equipment: X1.16.1.1 Appropriate_____ X1.16.1.2 Dedicated screens_____

X1.16.2 Calibration: X1.16.2.1 RM source/date_____ X1.16.2.2 Frequency_____

X1.16.3 Air drying appropriate_____ X1.16.4 Moisture reported_____

X1.16.5 Control sample freq./results_____ X1.16.6 PT results_____

X1.16.7 Repeatability_____ Reproducibility_____

FIG. X2.1 *(continued)*