INTRODUCTION

Obtaining a sample of a single lot of coal from railcars that is acceptably representative of the lot from which it was taken may present some unique problems. Factors such as variations in railcar loading equipment and procedures, lack of access to predictable portions of coal in railcars, time, weather, and the heterogeneous nature of the product can all interact, inducing unacceptable bias and variance in the sample. Therefore, considering the aforementioned and considering the sample only represents the top layer of coal in the railroad car, this sampling guide should not be considered as a substitute for a more reliable sampling method, for example, full stream mechanical sampling. Nonetheless, some laboratory measurement of a lot of coal is often required, so sampling procedures for coal in railcars must be designed which reduces variance to an acceptable level while remaining physically and economically feasible.

The procedures described herein are designed to provide guidelines for meeting this criteria. As a caution, however, it is highly recommended that any modification in sampling procedure be accompanied by an understanding of both the product and potential effects of sample interactions with those modifications.

1. Scope

1.1 This guide describes manual sampling procedures for coal contained in railroad cars. These procedures are used to provide samples for general analysis and for the determination of total moisture. The gross sample is to be crushed and further prepared for analysis in accordance with Method D 2013 and Test Methods D 2234.

1.2 This guide provides instructions for sampling only the top 18 to 24 in. of a railcar. The user is cautioned that samples so obtained do not represent material below the point of penetration.

1.3 The user is cautioned that, during the removal of increments, spillage of coal from the sampling device is to be avoided. Spillage can result in a size biasing of the various sizes in the coal.

1.4 The samples obtained by this practice under Condition D (from stationary sources, see Test Methods D 2234), shall be considered less representative than samples taken from the same lot under Conditions A, B, or C (Test Methods D 2234).

1.5 The quantities stated in either acceptable SI units or in inch-pound units shall be regarded separately as standard. The quantities stated in each system may not be exact equivalents; therefore, each system must be used independently of the other, without combining quantities in any way.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 2013 Method of Preparing Coal Samples for Analysis
D 2234 Test Methods for Collection of a Gross Sample of Coal
E 105 Practice for Probability Sampling of Materials
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. Terminology

3.1 Definitions

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

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

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

3.3 angle of repose—the greatest angle, measured from horizontal, attained by a coal so that material on the sides or top of the inverted cone or trench created in car top sampling remains stable (that is, will not shift or slide). Coal size and moisture content are contributing factors to this angle. In all cases, the coal shall be dug so that the physical angle is less than the angle of repose.

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

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

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

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

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

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

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

3.11 representative sample—a sample collected from a lot of coal in such a manner that all properties to be analyzed are proportionately represented in the sample, to within agreed upon limits of accuracy.

3.12 sample—a quantity of material taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity.

3.13 sample division—the process whereby a sample is reduced in weight without change in particle size.

3.14 sample preparation—any procedure that may include air drying, crushing, division, and mixing of a gross sample, implemented for the purpose of obtaining an unbiased analysis sample.

3.15 significant loss—any loss that introduces a change in an analysis that is of appreciable economic importance to the concerned parties.

3.16 size consist—the particle size distribution of a coal.

3.17 subsample—sample taken or prepared from another sample.

3.18 top size—the opening of the smallest screen in the series upon which is retained less than 5 % of the sample (see Test Method D 431).

4. Summary of Guide

4.1 Manual car top sampling is accomplished by collecting increments from the tops of railroad cars for the purpose of acquiring a gross sample.

5. Significance and Use

5.1 Coal in any condition of preparation, from raw run-of-mine to a washed and sized product can be transported in railroad cars. The guidelines, cautions, and suggested procedures presented here are intended to aid in the proper planning of car top sampling operations, taking into account the wide variety of conditions that may be encountered.

5.2 When agreed upon by all interested parties, these guidelines provide procedures for collecting gross samples from tops of railroad cars; these samples then being further processed to provide subsamples for required laboratory determinations.

5.2.1 Use of this guide results in human discretion often being exercised in the selection of increments, therefore, samples collected in accordance with this guide are not considered probability samples (see Practice E 105). Thus, statistical inferences cannot be drawn from these samples. No general statement concerning the reliability of these samples may be made.

5.3 This sampling method is used only when sampling by the preferred methods, (Conditions A, B, or C in Test Methods D 2234) is not feasible. Condition D, of which car top sampling is a type, is described as the least desirable method for collection of gross samples from a lot of coal.

6. Organization and Planning

6.1 Lot Size—All interested parties shall agree before sampling begins to the size of the lot to be represented by one gross sample. Section 7 of Test Methods D 2234 is the proper guideline.

6.2 Top Size—The top size of the coal to be sampled should be ascertained to determine the size of the increment collection device (a minimum of two and one half times the top size of the coal), as well as the weight and number of increments to be taken. (Use of Table 1 is recommended to establish minimum weight and number of increments).

6.3 Sampling Pattern—The configuration of increment collection over the surface of the coal in a railcar should be agreed upon by all interested parties prior to commencement of sampling. Fig. 1 illustrates examples of typical car top sampling patterns, however, these are examples only and the user may be guided but not bound by these patterns. It is recommended that the user develop, by test, the pattern(s) that work best for the type of coal the user samples.

6.4 Storage of Increments—It is recommended that sample increments be adequately protected from moisture variation by the use of moisture-tight containers. Care must be taken to ensure that the integrity of the container, including any moisture seal, is not compromised during handling.

6.5 Stratification—Stratification can occur both vertically and horizontally within a railcar. Vertical stratification may
often be detectible visually and increments should be taken so as to proportionately represent all visible strata within the lot. Horizontal stratification may not be visually detectable from the top of the railcar. When horizontal stratification is suspected, more extensive sampling procedures are needed.

7. Apparatus and Equipment

7.1 Sampling implements and sample containers (suitable for storage and transportation) must be agreed upon by all interested parties prior to commencement of sampling. Fig. 2 illustrates some commonly used increment collection implements.

7.2 It is recommended that storage containers should be both waterproof and strong enough to be removed from the tops of the railcars and transported to the preparation facility without damage. (For example, a commonly used container is a combination of a heavy woven plastic or heavy cotton outer bag with a plastic inner liner bag of at least 4-mil thickness). The sealing of both bags (for example, with drawstrings or twist-ties) is recommended. Ten- or twelve-gallon metal containers with similar plastic bag inner liners and tight-fitting lids may also be used where appropriate.

7.3 Each storage container must be fully identified to keep it separate from any other container. Waterproof marking pens and tags are useful for this purpose. Two tags are recommended, each fully marked with all identifying information for each container, one placed inside the inner liner plastic bag and one placed on the outside of the container.

8. Personnel

8.1 Field situations often dictate on-site modifications of sampling procedure. These modifications should only be made by personnel with an understanding of and a sense of responsibility for the potential effects on the sample of variations in sampling procedures.

9. Sampling Procedure

9.1 This procedure, (classified in Test Methods D 2234, as Condition D—stationary sampling, where Type II—human discretion is employed) provides recommendations for manual sampling from tops of railroad cars.

9.2 Prior to sampling of a lot of coal, a visual inspection for size distribution and possible size segregation within each car should be performed. Increments should be collected to represent the observed size distribution of the coal.

9.3 The top size of the coal, condition of preparation, and lot size to be represented by each gross sample must be ascertained in order to determine from Table 1 the minimum number

<table>
<thead>
<tr>
<th>Top Size</th>
<th>5/8 in. (16 mm)</th>
<th>2 in. (50 mm)</th>
<th>6 in. (150 mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanically Cleaned Coal*</td>
<td>15</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>Minimum weight of increments, lb</td>
<td>2</td>
<td>6</td>
<td>15</td>
</tr>
<tr>
<td>Minimum weight of increments, kg</td>
<td>1</td>
<td>3</td>
<td>7</td>
</tr>
</tbody>
</table>

* If there is any doubt as to the condition of preparation of the coal (for example, mechanically cleaned coal or raw coal) the number of increments for raw coal shall apply. Similarly, although a coal has been mechanically cleaned, it may still show great variation because of being a blend of two different portions of one seam or a blend of two different seams. In such cases, the number of increments should be as specified for raw (uncleaned) coal.
and weight of increments.

9.4 The increment weight shall be no less than specified in Table 1, and all increments shall be of approximately equal weight.

9.5 Number of Increments:

9.5.1 For a lot of up to 1000 net tons, the number of increments collected to represent that lot shall be no less than specified in Table 1.

9.5.2 For a lot of more than 1000 tons, where one gross sample will be used to represent that lot, the following equation may be used to determine the recommended number of increments to be collected:

\[ N_2 = N_1 \frac{\text{total lot size (tons or Mg)}}{1000 \text{ tons or } 908 \text{ Mg}} \]

where:
- \( N_1 \) = number of increments specified in Table 1, and
- \( N_2 \) = number of increments required.

9.5.3 The minimum number of increments to be collected from each railcar can be determined by dividing the total minimum number of required increments by the number of railcars in the lot. It is recommended that fractions of increments for each railcar be mathematically rounded upward to the next whole number. (Example: 19 railcars containing 90 net tons of raw coal per car would require a minimum of 46 increments or 2.42 increments per railcar, rounded upward to three increments per railcar). Rounding upward ensures that the total number of increments collected meets or exceeds the minimum requirements. In any case, a minimum of one increment per railcar is recommended.

9.6 Increment Positions:

9.6.1 Whether chosen randomly or by pattern, it is recommended that increment positions be agreed upon by all concerned parties. To determine potential positions of increments, divide the railcar length into as many equal sections as the required number of increments and divide the railcar width into three, four, or five equal sections, depending on which number is common to the required number of increments per railcar. If one increment per railcar is required, the length and width should be divided into three equal sections, varying the placement of increments in successive cars. An imaginary grid is formed dividing the surface of the railcar into equal rectangular sections which are the potential positions for the removal of increments.

9.6.2 As shown in Fig. 3, when increment positions are randomly selected, it is recommended that the procedure chosen ensures that an equal number of increments are collected from each section of the grid throughout the sampling lot.

9.6.3 When increment positions are selected by pattern as shown in Fig. 1, the pattern should be chosen so that an increment is collected from each length section of the railcar and an equal number of increments are collected from each width section of the railcar. The sampling pattern should be reversed after each pattern cycle.

9.7 According to Test Methods D 2234, the recommended size of a lot of coal to be represented by one gross sample shall not exceed 10 000 net tons (9080 metric tons) (9080 Mg). Frequently, however, daily top car sampling regimes may produce samples whose increments represent only part of a larger lot of coal, for example, ten cars out of a total of 50 cars are sampled and prepared each day for five days producing five samples representing a 50-car lot. In these cases, each sample may be composited proportionately with the other samples to represent the whole lot to be analyzed or each sample may be analyzed separately and a calculated composite analysis of the whole lot may be obtained. In either case, increments proportional to the whole lot may be collected from each smaller lot. It is recommended that each smaller lot be referred to as a sublot. When the sample representing the sublot is analyzed and reported separately, it is recommended to be referred to as a fractional gross sample.

9.8 Increments shall be collected from holes or trenches dug to a depth of at least 18 in. (46 cm), and the angles of the sides of the holes or trenches shall be less than the angle of repose. The coal removed shall be placed away from the sample area. The increment then shall be taken from the bottom of the hole or trench and put into its container, avoiding any spillage.

9.9 All increments collected should be promptly sealed in their containers and prepared in accordance with Method D 2013.

10. Exceptions and Cautions

10.1 Moisture determinations from car top samples are often biased by factors such as downward moisture migration, evaporation, and precipitation.

10.2 It is essential that the increments be removed from throughout the lot to be sampled. Therefore, the sampling pattern used shall be varied if the possibility exists that increment patterns are in phase with the sequence of coal
variability. (Example: routine car top sampling where increment patterns coincide with the “high” or “lows” in the content of fines.)

10.3 In extremely cold weather, coal can become frozen inside the railcar. Whenever possible, do not attempt to manually collect increments from frozen coal until it thaws. When sampling is necessary while coal is frozen, it will be extremely difficult or even impossible to collect increments properly.

10.4 All sampling personnel shall be trained and cautioned on all aspects of sampling safety prior to commencement of sampling.

11. Precision and Bias
   11.1 The wide variety of situations and sampling regimes that may be encountered with this practice precludes an accurate measure of precision that would apply to every case. The necessity of using human discretion for increment placement in this practice suggests that precision and bias is, in part, a function of the knowledge and abilities of the individual assigned to the task. While this procedure uses several specifications from Test Methods D 2234, it does not necessarily follow that an equivalent precision will result. No statement of precision or bias can be made for this guide.

12. Keywords
   12.1 manual sampling; railroad cars