



Designation: D 346 – 90 (Reapproved 1998)

Standard Practice for Collection and Preparation of Coke Samples for Laboratory Analysis¹

This standard is issued under the fixed designation D 346; 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

Coke, especially run of oven coke or foundry coke, or both, is a difficult material to sample. It is imperative that every sample be collected and handled carefully and conscientiously and in strict accordance with the standard procedure described herein.

Gross samples of not less than the quantities designated in this practice must be taken, whether the coke to be sampled consists of a few tons or several hundred tons.

1. Scope

1.1 This practice covers procedures for the collection and reduction of samples of coke to be used for physical tests, chemical analyses, and the determination of total moisture.

1.2 The values given in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address the safety concerns 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 167 Test Method for Apparent and True Specific Gravity and Porosity of Lump Coke²
- D 293 Test Method for the Sieve Analysis of Coke²
- D 2013 Method of Preparing Coal Samples for Analysis²
- D 2234 Practice for Collection of a Gross Sample of Coal²
- D 3038 Test Method for Drop Shatter Test for Coke²
- D 3302 Test Method for Total Moisture in Coal²
- D 3402 Test Method for Tumbler Test for Coke²

3. Significance and Use

3.1 This practice may be used to provide a representative sample of the coke from which it is collected. Because of the variability of coke and the wide variety of sampling equipment,

caution must be exercised in all stages of sampling, from system specifications and equipment procurement to equipment acceptance testing and actually taking the final sample.

3.2 This practice also provides an analysis sample of coke from the gross or divided sample. The analysis samples can be used to determine the value of the coke represented, its ability to meet specifications, as well as for other purposes.

4. Place of Sampling

4.1 Sample coke while it is being loaded into or unloaded from railroad cars, ships, barges, or trucks, or when discharged from supply bins, grab buckets, belt conveyers, or other coke conveying equipment. Sample close to the point of interest.

5. Collection of Gross Sample

5.1 Collect increments regularly, systematically, and with such frequency, so that the entire quantity of coke sampled will be represented proportionately in the gross sample, and a gross sample of the required amount will be collected. The standard gross sample shall equal or exceed the quantities given in Table 1.

5.1.1 The quantity of sample to be taken will depend on the size of the coke being sampled and the amount of information to be obtained from the sample.

5.1.1.1 The size of the sieve analysis sample is governed by the homogeneity of the coke being sampled. The quantities given in Table 1 represent the minimum quantity to be collected. The sieve analysis sample will supply sufficient coke for any subsequent physical tests and laboratory analysis. Total moisture of the coke should not be determined on the sieve analysis sample.

5.1.1.2 For the drop shatter test, a minimum of 75 kg (165 lb) of coke retained on 50-mm (2-in.) square mesh sieve if furnace coke is being processed, or 140 kg (300 lb) of coke

¹ This practice is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.23 on Sampling.

Current edition approved Sept. 28, 1990. Published January 1991. Originally published as D 346 – 32 T. Last previous edition D 346 – 78.

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

TABLE 1 Minimum Gross Sample Weights of Coke, kg (lb)

Usage	Foundry Coke		Furnace Coke		Coke Breeze ^A
	Run of Oven	Sized	Run of Oven	Sized	
Sieve analysis (size consist)	364 (800)	227 (500)	227 (500)	182 (400)	45 (100)
Drop shatter test	182 (400)	159 (350)	136 (300)	91 (200)	...
Tumbler test	68 (150)	45 (100)	57 (125)	57 (125)	...
Apparent specific gravity	46 (100)	46 (100)	46 (100)	46 (100)	23 (50)
Coke moisture	46 (100)	46 (100)	46 (100)	46 (100)	23 (50)
Chemical analysis	113 (250)	113 (250)	57 (125)	57 (125)	34 (75)

^A Coke passing a 3/4-in. (19-mm) square hole sieve or smaller top size.

retained on 75-mm (3-in.) square mesh sieve if foundry coke is being processed, is required. The quantities given in Table 1 represent the minimum amounts required of sized coke.

5.1.1.3 The tumbler test requires a minimum of 35 kg (75 lb) of coke that will pass a 75-mm (3-in.) square mesh sieve and can be retained on a 50-mm (2-in.) square mesh sieve, or that which will pass a 64-mm (2½-in.) square mesh sieve and can be retained on a 38-mm (1½-in.) square mesh sieve. The quantities given in Table 1 represent the minimum amount of the required sized coke.

5.1.1.4 The apparent specific gravity test required a minimum of 23 kg (50 lb) of representative coke pieces. The quantities given in Table 1 represent the minimum amounts of the required coke pieces.

5.1.1.5 The determination of total moisture of the coke requires approximately 45 kg (100 lb) of coke. The quantities given in Table 1 represent the minimum amount to be collected.

5.1.1.6 The quantities given for samples for chemical analysis represent the minimum amounts to be collected.

NOTE 1—Samples collected from the surface of coke in piles, bins, cars, ships, or barges are, in general, unreliable because of size segregation and should not be used for determining conformance to specifications unless the purchaser and the seller so agree. If necessary to collect a sample of coke from the surface of a loaded railroad car, take nine equal increments about 305 mm (1 ft) below the surface. Locate the nine points as shown in Fig. 1. The diameter of the hole must be at least 3 times that of the largest piece in the shipment.

6. Condition of Increment Collection

6.1 Four conditions of increment collection are recognized:

6.1.1 *Condition A (Stopped-Belt Cut)*, in which a loaded conveyor belt is stopped and a full cross-section cut with parallel sides is removed from the coke stream. The distance between the parallel faces shall not be less than three times the length of the largest piece.

6.1.2 *Condition B (Full-Stream Cut)*, in which a full cross section is removed from a moving stream of coke.

6.1.3 *Condition C (Part-Stream Cut)*, in which a portion, not a full cross section, is removed from a moving stream of coke.

6.1.4 *Condition D (Stationary Sampling)*, in which a portion of coke is collected from a pile, a railroad car, a barge, or a shiphold.

NOTE 2—See Section 6 of Practice D 2234.

7. Size of Increments

7.1 To collect increments, use a shovel or specially designed tool or mechanical means for taking equal increments. When increments are collected from the surface of a loaded rail car or truck shipment, the gross sample shall consist of nine increments of approximately equal quantity. When increments are taken from piles, conveyor belts, barges, and so forth, the gross sample shall consist of not less than 25, nor more than 50 increments, of approximately equal quantity.

NOTE 3—When large-size coke, such as foundry coke, is being sampled, it is likely that quantities larger than those given in Table 1 will be collected. However, the minimum number of increments collected should not be decreased unless such is agreed upon between the purchaser and the seller.

NOTE 4—If the sample for chemical analysis is obtained from the sieve analysis sample, a sample not smaller than that shown in Table 1 should be reconstituted from the size fractions obtained when the sieve analysis sample was processed.

8. Quantities Represented

8.1 Take a gross sample for each 454 Mg (500 tons) or fraction thereof, or in case of larger tonnages, for such quantities as may be agreed upon. Sample separately each lot of coke arising from a different source or known to be of different quality or size.

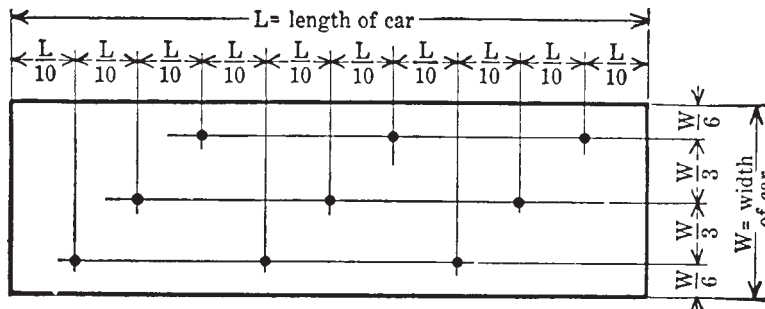


FIG. 1 Location of Sampling Points from Exposed Surface of Car

9. Processing of the Test Sample

9.1 Process samples collected for sieve analysis of coke in accordance with the procedure given in Method D 293.

9.2 Process samples collected for the drop shatter test in accordance with the procedure given in Method D 3038.

9.3 Process samples collected for the tumbler test in accordance with the procedure given in Method D 3402.

9.4 Process samples collected for the determination of apparent specific gravity and porosity in accordance with the procedure given in Test Method D 167.

9.5 Process samples taken for the determination of total moisture in accordance with the procedure given in Test Method D 3302 and Method D 2013.

9.6 Prepare samples taken for chemical analysis as directed in Section 10.

10. Preparation of Coke Sample for Laboratory Analysis

10.1 Crush, mix, and divide the entire gross sample in quantity to convenient size for transmission to the chemical laboratory. Crush the sample, preferably by means of jaw or roll crushers, or on a chilled iron or hard steel plate by impact of a tamper, hard bar, or sledge, avoiding all rubbing actions as otherwise the ash yield can be materially increased by the addition of iron from the sampling apparatus. Do the crushing under such conditions as to prevent loss of coke or accidental mixture of foreign matter.

10.2 Procedure A, Manual Riffing:

10.2.1 Determine the number of passes required in the riffing operation from the total volume of the gross sample and the minimum permissible weight in accordance with Table 2.

10.2.2 Divide the crushed gross sample by using a large riffle. Riffles properly used will reduce sample variability but cannot eliminate it. Pass the crushed coke through the riffle from a feed scoop, feed bucket, or riffle pan having a lip or opening the full length of the riffle. When using any of the above containers to feed the riffle, spread the crushed coke evenly in the container, raise the container, and hold it with its front edge resting on top of the feed chute; then slowly tilt it so that the coke flows in a uniform stream through the hopper straight down over the center of the riffle into all the slots, thence, into the riffle pans, one half of the sample being collected in each pan. Under no circumstances shovel the sample into the riffle, or dribble into the riffle from a small-mouth container. Do not allow the crushed coke to build up in or above the riffle slots. If it does not flow freely through the slots, shake or vibrate the riffle to facilitate even flow. The opening of the riffle slot should be 2.5 to 3 times the size of the largest particle.

10.2.3 After crushing the gross sample of not less than 14 kg (30 lb) (see Table 2) for 4.75-mm (No. 4) sieve size, using suitable pulverizing equipment, further reduce this subsample to 2.36-mm (No. 8) sieve size and divide by riffing to not less than the quantity specified in Table 3 for 2.36-mm (No. 8) sieve size.

10.2.4 With suitable pulverizing equipment, preferably a roll crusher or ball mill, reduce the 2.36-mm (No. 8) sieve size subsample to a 250- μ m (No. 60) sieve size.

10.2.5 As an alternative to the procedure given in 10.2.4, the 2.36-mm (No. 8) sieve size subsample may be reduced to pass 95 % through a 850- μ m (No. 20) sieve. Divide this subsample by riffing with the small riffle to not less than the quantity specified in Table 3, and then reduce to 250- μ m (No. 60) sieve size as described in 10.2.4.

10.2.6 Thoroughly mix the laboratory analysis sample, weighing not less than 50 g, preferably, by mechanical means, before extracting portions for analysis.

10.3 Procedure B, Hand Division:

10.3.1 The method of dividing by hand the quantity of coke in a gross sample shall be carried out as described even though the initial size of coke and impurities may be less than indicated in Table 2.

10.3.2 Do the progressive crushing and division in the weight of the sample to the quantities indicated in Table 2 by the following method, which is described and illustrated in the accompanying Fig. 2, until the maximum size of the coke is 13 mm ($\frac{1}{2}$ in.), and the quantity is 27 kg (60 lb). Crush the gross sample to a maximum size of pieces of 50 mm (1 in.).

10.3.2.1 Repeat the alternate-shovel method of dividing the gross sample until the sample is divided to approximately 57 kg (125 lb) and take care to observe before each division in quantity that the sample has been crushed to the fineness specified in Table 2. Shovel the crushed coke into a conical pile (Fig. 2 (2)) by depositing each shovelful of coke on top of the preceding one, and then form into a long pile in the following manner: Take a shovelful of coke from the conical pile and spread it out in a straight pile (Fig. 2 (3A)) having a width equal to the width of the shovel and a length of 1.5 to 3 m (5 to 10 ft). Spread the next shovelful directly over the top of the first shovelful, but in the opposite direction, and so on back and forth, piling and, occasionally flattening the pile, until all the coke has been formed into one long pile, proceeding as follows: Beginning on one side of the pile, at either end, and shoveling from the bottom of the pile, take one shovelful (Fig. 2 (4), shovelful No. 1) and set it aside; advancing along the side of the pile a distance equal to the width of the shovel, take a second shovelful (shovelful No. 2) and discard it; again advancing in the same direction one shovel width, take a third shovelful (shovelful No. 3) and add it to the first. Take the

TABLE 2 Weights of Coke Samples with Corresponding Crushing Sizes

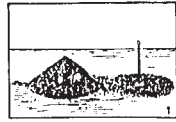
Weight of Sample to Be Divided		Largest Size of Coke and Impurities Allowable in Sample Before Division	
kg	lb	mm	in.
113 or over	250 or over	25	1
57	125	19	$\frac{3}{4}$
27	60	13	$\frac{1}{2}$
14	30	5	4 mesh

TABLE 3 Preparation of Sample for Chemical Analysis

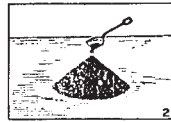
Crush to Pass at Least 95 % Through Sieve	Divide to Minimum Weight, g
4.75 mm (No. 4)	2000
2.36 mm (No. 8)	500
850 μ m (No. 20)	250
250 μ m (No. 60) (100 % through)	50

NOTE
 SELECT A HARD, CLEAN SURFACE, FREE OF CRACKS AND PROTECTED FROM RAIN, SNOW, WIND, AND BEATING SUN. DO NOT LET CINDERS, SAND, CHIPPINGS FROM FLOOR, OR ANY OTHER FOREIGN MATTER GET INTO THE SAMPLE. PROTECT SAMPLE FROM LOSS OR GAIN IN MOISTURE

FIRST STAGE in the Preparation of a 227-kg (500-lb) Sample



Crush 227-kg (500-lb) on hard, clean surface to 25 mm (1 in.)



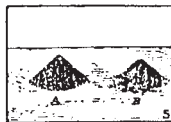
227-kg (500-lb) sample crushed to 25 mm (1 in.) and coned



Mix by forming long pile
 A-Spreading out first shovelful
 B-Long pile completed



Halving by alternate shovel method. Shovelfuls 1,3,5, etc. reserved as 5A; 2,4,6, etc. rejected as 5B

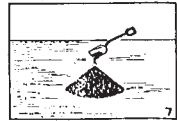


Long pile divided into two parts; A-Reserve; B-Reject

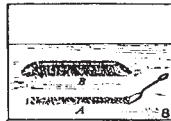


Crush 113-kg (250-lb) Sample (Fig. 5A) to 19-mm (3/4 in.) size

SECOND STAGE



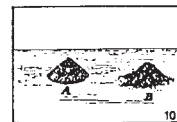
113 kg (250 lb) crushed to 19 mm (3/4 in.) and coned



Mix by forming long pile
 A-Spreading out first shovelful
 B-Long pile completed



Halving by alternate shovel method. Shovelfuls 1,3,5, etc. reserved as 10 A; 2,3,5, etc. rejected as 10 B



Long pile divided into two parts; A-Reserve; B-Reject

FIG. 2 Standard Hand Method of Crushing and Reducing the Gross Sample Quantity of Coke

fourth (shovelful No. 4) in a like manner and discard, retain the fifth (shovelful No. 5), and so on, advancing always in the same direction around the pile so that its size will be gradually divided in a uniform manner. When the pile is removed, about half of the original quantity of coke should be contained in the new pile formed by the alternate shovelfuls that have been retained (Fig. 2 (5A) shows the retained half and (5B) the rejected half).

10.3.3 After the gross sample has been divided by crushing and by the alternate-shovel method to approximately a 27-kg (60-lb) sample of 13-mm (1/2-in.) coke, proceed using the methods in 10.2.3.

10.4 The report of analysis should include a brief description of the method of taking the sample, by such characteristic expressions as *belt sample*, *top-of-car*, and so forth. Information should be given as to how the gross sample is crushed and divided in the description that accompanies the sample sent to the laboratory.

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