Designation:C50–00 (Reapproved 2006) Designation: C50/C50M – 12

Standard Practice for Sampling, Sample Preparation, Packaging, and Marking of Lime and Limestone Products[1]

This standard is issued under the fixed designation C50/C50M; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice covers procedures for the collection and reduction of samples of lime and limestone products to be used for physical and chemical tests.

1.2 This practice further covers inspection, rejection, retesting, packing, and marking of lime and limestone products as it may be used in the chemical, agricultural, and process industries.

1.3 *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:*[2]

C51 Terminology Relating to Lime and Limestone (as used by the Industry)
C702 Practice for Reducing Samples of Aggregate to Testing Size
D75 Practice for Sampling Aggregates
D2234/D2234M Practice for Collection of a Gross Sample of Coal
D3665 Practice for Random Sampling of Construction Materials
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E105 Practice for Probability Sampling of Materials
E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
E141 Practice for Acceptance of Evidence Based on the Results of Probability Sampling
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. Terminology

3.1 *accuracy*—a term generally used to indicate the reliability of a sample, a measurement, or an observation and is a measure of closeness of agreement between an experimental result and the true value.

3.2 *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.

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

3.4 *combined water*—water that is chemically bonded to calcium or magnesium oxide to form hydrate.

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

3.6 *free water*—water that is not chemically bonded to calcium or magnesium oxide.

3.7 *gross sample*—a sample representing one lot of material 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

[1] This practice is under the jurisdiction of ASTM Committee C07 on Lime and is the direct responsibility of Subcommittee C07.06 on Physical Tests.

Current edition approved Nov. 15, 2006. Published December 2006. Originally approved in 1922. Last previous edition approved in 2000 as C50–00. DOI: 10.1520/C0050-00R06.

Current edition approved July 1, 2012. Published July 2012. Originally approved in 1922. Last previous edition approved in 2006 as C50 - 00 (2006). DOI: 10.1520/C0050_C0050M-12.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

increments from the lot to make a gross sample.

3.9 *laboratory sample*—refers to the sample after the initial preparation from which the analytical sample is obtained.

3.10 *lot*—a discrete quantity of material for which the overall quality to a particular precision needs to be determined.

3.11 *precision*—a term used to indicate the capability of a person, an instrument, or a method to obtain repeatable 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 E177).

3.12 *representative sample*—a sample collected in such a manner that every particle in the lot to be sampled is equally represented in the gross or divided sample.

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

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

3.15 *sample preparation*—the process that may include crushing, dividing, and mixing of a gross or divided sample for the purpose of obtaining a representative analysis sample.

3.16 *sampling unit*—a quantity of material from which a gross sample is obtained. A lot may contain several sampling units.

3.17 *segregation variance of increment collection,* Ss^2—the variance caused by nonrandom distribution of inert material or other constituent in the lot.

3.18 *size consist*—the particle size distribution of quicklime or hydrated lime.

3.19 *standard deviation*—the square root of the variance.

3.20 *subsample*—a sample taken from another sample.

3.21 *top size*—the opening of the smallest screen in the series upon which is retained less than 5 % of the sample.

3.22 *total variance,* So^2—the overall variance resulting from collecting single increments, and including division and analysis of the single increments.

3.23 *unbiased sample*—a sample free of bias or a representative sample.

3.24 *unit variance (random variance of increment collection),* Sr^2—the theoretical variance calculated for a uniformly mixed lot and extrapolate to 0.5-kg (1-lb) increment size.

3.25 *variance*—the mean square of deviation (or errors) of a set of observations; the sum of squared deviations (or errors) of individual observations with respect to their arithmetic mean divided by the number of observations less one (degrees of freedom); the square of the standard deviation (or standard error).

4. Significance and Use

4.1 The following practices are to be used in obtaining samples that are representative of the lot being sampled. The methodology used will be dependent upon the size and type of material sampled and testing requirements.

4.2 The following practices are intended for use in obtaining samples from material that is ready for sale and are not intended as sampling procedures for quality control purposes. These practices are to be used in obtaining a laboratory sample that will yield results serving as a basis for acceptance or rejection of the lot of material sampled. This does not preclude the use of these practices for quality control purposes.

4.3 The following practices can be used to eliminate bias in sampling. The person or persons responsible for using these practices must be trained and they will be conscientious and timely in their use.

4.4 An agreement between the producer and the consumer on location of sampling, either at the producer's plant or at the destination, is encouraged. Product quality can be affected through careless handling, improper protection, and delayed shipment. It is preferable to sample at the point of loading. The consumer has the right to witness the sampling practices being used.

4.5 This practice may be used to provide a representative sample of lime or limestone products. Due to the variability of limestone and lime and the wide variety of sampling equipment, caution must be exercised in all stages of sampling, from system specification and equipment procurement to equipment acceptance testing and actually taking the final sample.

5. Incremental Collection

5.1 For the number and weight of increments refer to Practice E122.

5.2 The number of samples required depends on the intended use of the material, the quantity of material involved, and the variations both in quality and size. A sufficient number of samples shall be obtained to cover all variations in the material.

5.3 The quantity of sample to be taken will depend on the size of the material to be sampled and the amount of information to be obtained from the sample. Caution must be taken to ensure a statistically correct amount of material is selected for all testing, and sufficient quantities of material retained for reserved purposes. Recommended reference documents would include Practices E105 and E122.

5.4 *Particle Size*:

5.4.1 Generally, a large range of particle sizes for a given material requires a larger bulk sample size. The amount of the sample increment is then dependent upon the largest particle size encountered. The sample amount is determined by repeated testing to determine the bias between successive increments, and then to reduce this bias to acceptable limits.

5.4.2 The chemistry may change relative to the particle size. It is important that all particle sizes proportioned relative to their distribution be in the parent material.

5.5 Large material transfer rates result in large incremental samples. The sample must be representative of the entire cross-section flow of material. The amount of sample and number of increments must be determined prior to sampling. Randomized sampling should be used where appropriate to minimize unintentional bias.

6. Random Sampling

6.1 Practices D3665, E105, and E122 can be used to minimize unintentional bias when obtaining a representative sample. Depending upon what comprises the lot of material, sampling can be extended to specific shipping units chosen on a random basis.

6.2 Collect increments with such frequency that the entire quantity of material will be represented in the gross sample. Due to the variability of lime and limestone products and the wide variety of sampling equipment, caution must exercised in all stages of sampling.

7. Sampling Plan

7.1 *Purpose*:

7.1.1 Adequate methods for obtaining representative samples for testing the chemical and physical properties of a shipment of lime or limestone are essential. The sale and use are dependent upon the chemical or physical properties, or both.

7.1.2 The sampling plan specifies the minimum weights and the number of increments required in each step of the procedure to meet the objectives of the testing.

7.1.3 The sampling plan should include the personnel doing the sampling, preservation or protection of the samples, location of sampling, the sampling procedure to be used, sample preparation required, and the tests to be performed.

7.1.4 Proper sampling involves understanding and consideration of the minimum number and weight of increments, the particle size of the material, sample preparation, variability of the constituent sought, and the degree of precision required.

7.2 *Personnel*:

7.2.1 It is imperative that a sample is collected carefully and conscientiously. If the sampling is done improperly, the sample is in error and any subsequent analysis is not representative of the lot being sampled. Further, a second sample may be impossible to obtain. If an analysis is in error, another analysis is impractical on an incorrectly obtained sample. Whereas, a second analysis is possible, if the first was in error, if the initial sampling was correct.

7.2.2 Because of the importance of proper sampling and the resulting information, individuals engaged in sampling and sample preparation must be qualified by training and experience and possess a thorough understanding of sampling practices and techniques or under the direct supervision of such an individual.

7.3 *Preservation of Sample*:

7.3.1 Due to the hygroscopic nature of quicklime, samples must be immediately stored in airtight, moisture-proof containers to avoid air-slaking and subsequent absorption of carbon dioxide.

7.3.2 Due to the generally soft characteristics of quicklime, proper handling to avoid degradation must be practiced if the sample is to be used for particle size determination.

7.4 *Location of Sampling*—The process type and the process measurements required determine the sampling location. Sites should be selected to allow for safe, easy access to a representative cross section of the process material.

7.5 *Choice of Sampling Procedure*—The choice of sampling procedure to be used is dependent on three things. First, it is necessary to define the lot or batch of material to be sampled. Second, it is necessary to determine the number of incremental samples to be taken from the lot. Third, the choice of sampling procedure needs to be determined from Section 8 utilizing the preceding criteria.

7.6 *Recommended Number and Weight of Increments*:

7.6.1 Refer to Table 1 for the recommended number and weight of increments for general purpose sampling. The number of increments required listed in Table 1 are based upon a 1000–ton lot size. To determine the number of increments recommended for a specific lot size, use Eq 1. To determine the recommended weight for a bulk sample, multiply the increment requirement times the minimum increment weight from Table 1~~–~~. The nominal particle size is assigned based on production screening.

7.6.2 The increments and weights listed in Table 1 are only recommendations and are not based upon a statistical model. For more accurate methods to determine weights and increments required, refer to Practices E105, E122, and E141 and Test Methods D2234/D2234M.

7.6.3 For randomized sampling, refer to Practice D3665.

$$N_2 = N_1 \text{ [specific lot size (tons) / 1000 tons]}^{1/2} \quad (1)$$

C0050_C0050M-12_1

TABLE 1 Recommended Number and Weight of Increments for General Purpose Sampling

Nominal Particle Size	– 1/4 in.	+ 1/4 by – 3/4 in.	+ 3/4 in.
Minimum number of increments	10	10	10
Minimum weight of increment, lb	5	10	15

where:
N_1 = minimum increments required, per 1000 ton lot, and
N_2 = increments required for specified lot size.

7.7 *Mechanical Sampling Devices*—There are several different types of mechanical sampling devices available for many of the sampling procedures mentioned in Section 8. Due to the variety of types, it is impractical to specifically identify each device. Prior to using any mechanical sampling device, it needs to be determined that the device is capable of taking an unbiased, representative sample of the material in question.

8. Sampling Procedures (See Sampling Procedure Flow Chart (Fig. 1) for Location of Specific Methods)

LIMESTONE

8.1 *Surface Sampling*:

8.1.1 Surface sampling is limited in use due to the nonrepresentative sample obtained. For exploration purposes, a surface sample can produce information with respect to the characteristics of a deposit. It is critical to remember that a surface sample is not representative and can only be used to determine if more detailed sampling and testing may be justified.

8.1.2 Obtain the necessary information to determine a suitable location for sampling. Choose sites that will best satisfy the purpose of sampling. Describe and record observations on the characteristics of the portion of the deposit being sampled to the extent required by the sampling plan. It is imperative for the sample collected to be of sufficient size to perform any required testing.

8.2 *Face Sampling*—Describe and record observations on the characteristics of the portion of the face being sampled to the extent required by the sampling plan. With suitable marking equipment, identify the sampling site in accordance with the sampling plan. It is imperative for the sample collected to be of sufficient size to perform any required testing.

8.3 *Drill Hole Samples*—The type of drilling equipment required will be determined by the sampling plan. Sample the drill hole in intervals as specified in the sampling plan.

8.3.1 *Drill Cuttings*:

8.3.1.1 Drill cuttings are deposited on the surface by the drilling equipment. Many drills use compressed air to blow the drill cuttings out of the drill hole. These cuttings collect on the surface in a circular mound surrounding the hole. Collect a crosscut representative sample of the drill cuttings, taking care not to contaminate with surface material.

8.3.1.2 Recirculated drill cuttings are produced from another type of drilling equipment using compressed air to blow the drill cuttings through the hollow center of equipment drill steel into a collection chamber. Empty this chamber at intervals determined in the sampling plan.

8.3.2 *Drill Core*:

8.3.2.1 Some drilling equipment cuts and removes solid cylindrical cores of material from a drill hole. Sample these drill cores at intervals determined in the sampling plan.

8.3.2.2 Drill cores are split as determined by the sampling plans. One portion of the split core is preserved intact, maintaining its orientation and order as it is removed from the drill hole. These samples are invaluable for historical purposes and are often saved for the life of the quarry.

8.4 *Limestone Kiln Feed Sampling*:

8.4.1 *Belt Sampling*—Two conditions exist from which samples can manually be obtained from a conveyor belt.

8.4.1.1 *Stop-Belt Sample*:

(1) Before stopping, the conveyor must be loaded with a constant flow of material in order to be sampled. The conveyor must then be secured consistent with proper safety procedures.

(2) Carefully remove the sample increment of material from completely across the belt, removing all material in the selected area including fines with, for example, a brush. Templates, whose bottom edge are shaped to match the belt contour, are useful in bracketing the sample location, thus preventing contamination of sample from material adjacent to the sampling area. It is important that the sample increment is composed of the entire cross section of material flow. Repeat the preceding process to remove the number of increments necessary to compose the bulk sample.

8.4.1.2 *Head-Pulley Sample*:

(1) When looking at a granular or pebble material conveyed with a belt, the fines tend to sift though the coarser material and ride on the bottom and toward the middle of the belt. The coarse and fines thus become segregated to an extent dependent upon gradation and physical conditions of the material being sampled. As the material is projected from the head-pulley, the coarser material is thrown slightly further with the fines dropping closer to the head-pulley. The most important considerations, therefore, in sampling at a head-pulley is that the entire cross section of material flow (fines and coarse) is obtained with the pass of the sampling apparatus. And further, that the movement of the sampling apparatus is accomplished in a timely manner, so as to reduce any bias in sampling from the lateral movement of the sampler.

(2) Head-pulley sampling can only be accomplished manually if the flow of material is at a minimum for safety reasons, otherwise an automatic sampler is recommended.

8.4.2 *Stockpile Sampling*:

8.4.2.1 Sampling from stockpiles, although occasionally necessary, is not recommended, because of the difficulty in