Designation: C50/C50M – 13

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 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.4 *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]

C25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime

C110 Test Methods for Physical Testing of Quicklime, Hydrated Lime, and Limestone

C400 Test Methods for Quicklime and Hydrated Lime for Neutralization of Waste Acid

C1271 Test Method for X-ray Spectrometric Analysis of Lime and Limestone

C1301 Test Method for Major and Trace Elements in Limestone and Lime by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP) and Atomic Absorption (AA)

D2234/D2234M Practice for Collection of a Gross Sample of Coal

D3665 Practice for Random Sampling of Construction Materials

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.

[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.

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[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.

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 *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–tonne [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 Practice D2234/D2234M.

7.6.3 For randomized sampling, refer to Practice D3665.

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

where:

N_1 = minimum increments required, per 1000 tonne [1000 ton] lot, and

N_2 = increments required for specified lot size rounded to the nearest whole number.

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)

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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

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

Nominal Particle Size	–6.3 mm [–1/4 in.]	+6.3 mm by 19 mm [+1/4 by –3/4 in.]	+19 mm [+3/4 in.]
Minimum number of increments	10	10	10
Minimum mass of increment, kg [lb]	2.5 [5]	5 [10]	7.5 [15]

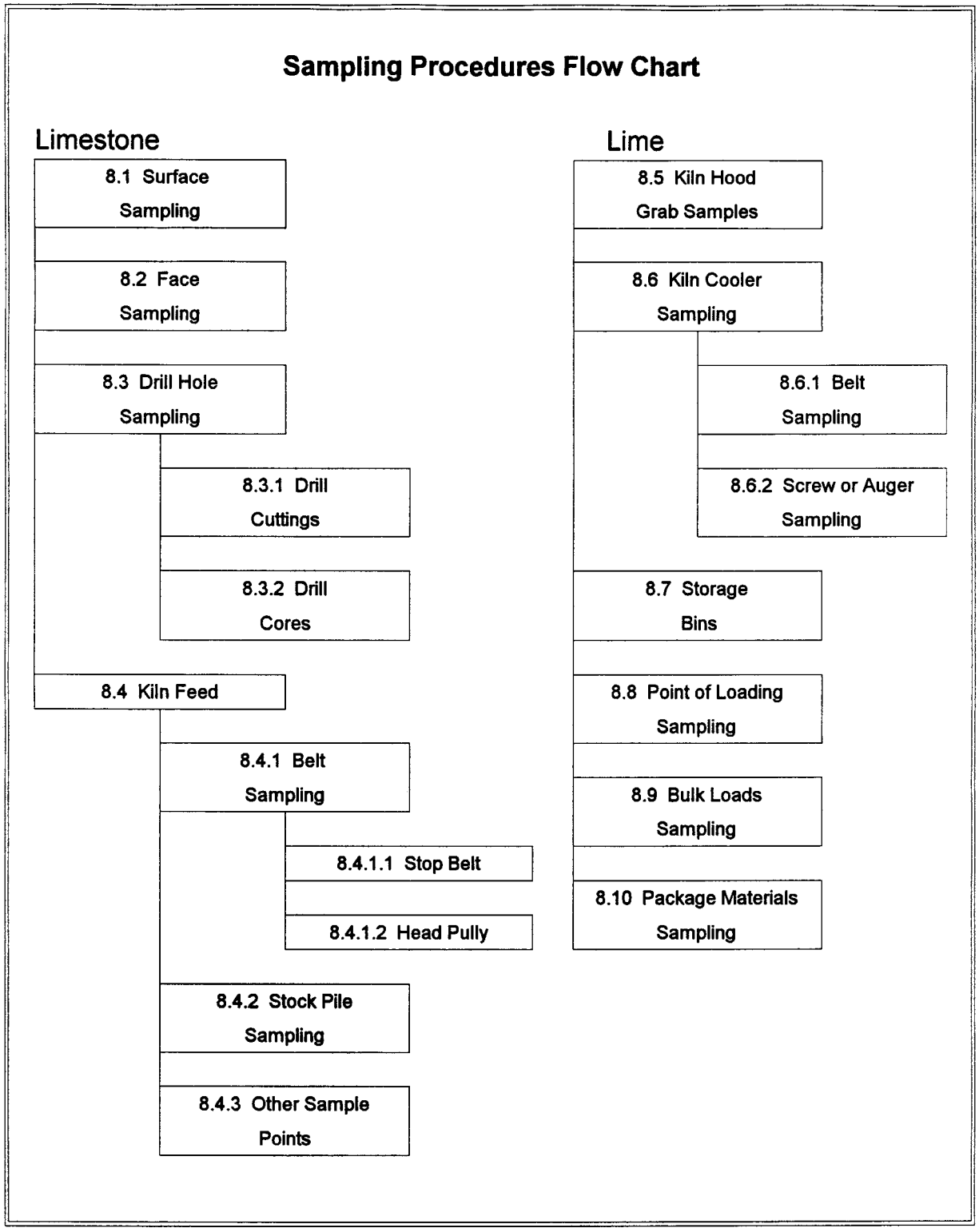

FIG. 1 Sampling Procedures Flow Chart

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 guaranteeing an unbiased sample. When sized material is stockpiled, segregation occurs, with coarser particles rolling to the outside base of the pile and finer particles sifting toward the center. It is very difficult to ensure representative samples, due to the segregation, which usually occurs when material is stockpiled, with the coarser particles rolling to the outside base of the pile. When it is necessary to sample stockpiles, every effort should be made to enlist the services of power equipment that is capable of exposing the material at various levels and locations. Separate samples shall be taken from different areas of the stockpiles to represent the material in that portion. Test results of the individual samples will indicate the extent of segregation existing in the stockpiles.

8.4.2.2 If it is necessary to sample stockpiles, numerous increments should be obtained from various points and combined to create the bulk sample. The number of increments must be sufficient to indicate the degree of variability existing within the pile. This is of particular importance when testing for material gradation.

8.4.2.3 If power equipment is available, larger increments can be obtained and then combined to form a smaller stockpile from which the bulk sample can be removed. The increments must be of a sufficient number and from various locations on the main stockpile to ensure formation of a representative and unbiased sampling stockpile. After mixing the smaller stockpile, several increments from different locations can be combined to form the bulk sample. Depending upon the size of the main stockpile, several sampling stockpiles can be formed from which bulk samples can be then obtained.

8.4.2.4 When manually sampling a stockpile (see Note 1), its shape dictates the methodology to be used. If the stockpile has a large flat upper surface, increments can be taken from various locations on the top surface. Otherwise, increments must be taken from the side, on a line from the base to the top of the stockpile. At no time should material be taken directly from the surface, but should be obtained from at least a foot below the surface, because of segregation. When a stockpile is active, increments can be obtained from the working location.

NOTE 1—Safety is a major concern when taking this type of sample due to material slides. Caution must be used at all times, as the individual sampling is at risk. Two people should be involved in this type of sampling with one individual at a distance from the pile, mindful of the sampler's safety. An inspection of the locations must be made beforehand for

possible sliding hazards with those locations avoided. This is not a recommended practice.

8.4.2.5 When sampling from the side of a stockpile, on a line from top to bottom, select a minimum of five locations from which increments are to be taken (refer to Practice E122). Remove the surface material from the increment location to the proper depth in order to create a small working bench. Use of a shield is recommended above the sampling point to prevent further segregation and contamination. From the exposed bench remove an increment from as far inside the pile as possible. Repeat this procedure for each of the selected locations and composite increments to form the bulk sample.

8.4.2.6 When sampling on top of a flat stockpile, increment locations must be spaced over the entire surface and be of a number to ensure a representative bulk sample. Again, the increments must come from a depth of at least 30 cm [1 ft].

8.4.2.7 When sampling from the working face of an active stockpile, select a minimum of five to ten spots from the entire working face for which to withdraw an increment. The increment sites should be at least 60 cm [2 ft] from the base of the stockpile, and must not include accumulated material, which has fallen from the sides of the active face due to further segregation. Combine the increments to form the bulk sample.

8.4.3 *Other Sampling Points:*

8.4.3.1 Other sampling points include stockpile feeders, cascade feeders, chutes, and so forth. As before, the most important consideration is in obtaining a representative sample. This means obtaining a complete cross section of the material flow.

NOTE 2—Consideration must be given to the flow of material to the sampling point from a surge (from a bin, stockpile, etc.). As material goes into a surge point, segregation of material occurs. Likewise, as material flows from a surge, its character can change with time. As sample increments are obtained, an unintentional bias can be obtained relative to the bulk sample. This may not be the case if the flow of material is directly from a process or system, such as a specific screening deck. In any case, misleading results can be obtained if this consideration is ignored, even with conscientious sampling techniques.

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8.5 *Kiln Hood Grab Sample:*

8.5.1 The Kiln Hood Grab Sample is a nonrepresentative and bias sample that only indicates chemical condition of the material flow from which it was obtained. It indicates the degree of calcination, relative to particle size, at that point in time and assists the operator in making decisions concerning the operation of the kiln. Because of the nature of the kiln calcination process, it is the closest to the process and most timely indication of the material quality that can be obtained. Therefore, is very important, regardless of the bias nature of the sample.

NOTE 3—Safety issues, relative to the kiln process, involve the high-calcination temperatures and draft conditions at the time of sampling. Protective equipment must be used.

8.5.2 A scoop or large ladle attached to a long metal pole is used to obtain the sample. It is passed parallel to the axis of the kiln through the process flow to obtain all size fractions of the material. Several increments, to increase the probability of representation, should be obtained and combined to form the bulk sample. This is extremely critical as chemistry can vary with particle (pebble) size. It is also preferable that several minutes elapse between incremental addition to the bulk sample as particle size of the material flow can change. The sample should be stored in a large dust-protected container, allowed to cool to ambient temperature, and then processed for chemical analysis.

8.5.3 To increase the homogeneity, the entire bulk sample must be crushed initially before reduction in the amount of material.

8.6 *Kiln Cooler Sampling*—As with Kiln discharge samples, the purpose of sampling dictates the methods to be used. If chemical results are required as a process control, the frequency of increments and amount of material composing a bulk sample may be reduced. If the results are to be used as a tool for process control, the timing becomes critical, and a snapshot of the chemical characteristics of the process product becomes more important. The frequency of sampling is then determined by the variability of the process and the resulting product. This would be true for any of the methods used for sampling.

8.6.1 *Belt Sampling, Pans or Chutes*—Cooler sampling can involve a number of different methods. As with sampling limestones, many of the techniques are similar, such as with belts. Other techniques involve discharge points, such as chutes and feeders. For these techniques please refer to the previous sections referring to limestone sampling.

NOTE 4—When sampling at a kiln cooler, the material may be quite hot and the area very dusty. Appropriate safety precautions must be followed.

8.6.2 *Screw or Auger:*

8.6.2.1 One method involves the use of an opening placed along the axis of the screw conveyor, which is closed off by either a gate or valve. The smallest dimension of the opening must be at a minimum of three times the longest dimension of the largest particle of the material being conveyed.

NOTE 5—In obtaining samples from a screw or auger, the particle size can be altered as a result of the conveying process. Therefore a sample obtained in this fashion can not be used to determine sizing characteristics. The chemical nature of the material, though, can be determined from samples obtained in this fashion.

8.6.2.2 This is important to obtain a free flow of material into the sampling apparatus, and to ensure a representative sample relative to the material chemical characteristics. The number of increments and their amounts to compose the bulk sample must be determined beforehand, so as to obtained an unbiased, representative sample (refer to Practice E122).

8.6.2.3 A second method involves sampling the discharge of the screw or auger. In this case, the entire cross section of material flow must be obtained for proper representation. Again the number of increments and their amounts must be determined beforehand (refer to Practice E122).

8.7 *Storage Bins*—If the samples are taken from a bin, they shall be taken from the entire cross section of the flow of material as it is being discharged. At the beginning of the discharge from the bins, sufficient material should be permitted to flow to ensure normal uniformity before the sample is collected.

8.8 *Sampling at Point of Loading:*

NOTE 6—The sampling plan including sample size, frequency of sampling, and so forth (as well as possible problems with sampling, such as product degradation) should be reviewed with customers prior to purchase.

8.8.1 Bins, railcars, trucks, and barges should be sampled while being loaded. Sampling from bulk shipments (trucks, railcars, or barges) is not a recommended practice, because of the bias relative to particle size and obtaining a representative sample. If there is a bias, it is generally proportional to the particle size range. True particle representation from bulk shipments is difficult to obtain. Safety is a major concern when sampling from bulk shipments. It is preferred, whenever practical, to sample product as it is being transferred to a shipping unit.

8.8.2 Sampling may be done by cutting the stream of material going into the bin, railcar, truck, or barge with a suitable sampling device, diverting the entire product stream briefly to a sample container, or by stopping a belt and thoroughly cleaning the material into a sample container. For sampling practices involving conveyor belts, refer to 8.4.1.

8.8.3 If loading directly from a chute or a bin, it may not be practical to sample the stream, because of the volume, weight and speed of the material. For sampling practices involving bins, refer to 8.7.1.

8.8.4 Sampling may also consist of intermittent cutting of a stream with a suitable automatic sampling device to yield a representative composite sample. Mechanical sampling systems must be checked to ensure that the particle size is not degraded.

8.8.5 If necessary, fine lime products can be sampled from bulk shipments with the use of special devises that allow sampling through the depth of the shipping unit. These devises generally consist of two cylindrical hollow spears, one inside the other with consistent openings. The narrowest dimension of the openings must allow free flow in the material being sampled. As one spear is rotated the openings close or open, relative to the positions of both spears. The spear is forced down through the depth of the product with the openings closed. At maximum depth, the one spear is rotated to allow the product to flow into the inner spear and thus to collect a sample. The spear is rotated to close the openings and the sampling device is withdrawn from the material. The sample is collected from the top of the hollow spears. It is recommended that a number of increments be obtained from different equally spaced locations across the shipping unit to compose the bulk sample relative to the size of the shipping unit and sampling plan.

8.9 *Sampling of Bulk Loads:*

8.9.1 If necessary, for methods of sampling directly from bulk shipments use those practices in accordance with 8.4.2.

8.9.2 Separate samples shall be taken from as many points in the shipment unit as necessary to represent the material, realizing the probability of segregation as the material was loaded. These separate samples will usually be combined to form a composite sample. This sample shall be reduced, but if information on variation is desired, the separate samples shall be tested.

8.10 *Sampling of Packaged Material:*

8.10.1 *Random Sample*—Random sample may be taken using a combination of methods. Bags may be taken at random from within the pallet or among multiple pallets. A sample may then be taken from each bag. A composite of all bags is then formed and tested.

8.10.2 *Thief Sample*—Material to be taken from a bag may be taken from the available opening (see 8.8.5).

8.10.3 Samples must not be taken from packages whose integrity has been compromised.

9. Preparation of Laboratory Samples

9.1 Sample preparation is the reduction of the bulk sample in both particle size and amount of material to the suitable laboratory sample, so as to maintain representation of the initial bulk sample. Procedures as outlined must be followed in a conscientious and thorough manner in order to maintain that representation. The method of sample preparation to be used depends upon the type of material, tests to be performed, and the characteristics of the bulk sample in relation to particle size and quantity of material.

9.2 Generally when a screen analysis is required, a larger bulk sample is needed compared to a chemical analysis. But the key objective is to maintain representation of the bulk sample, so that the final test values reflect the characteristics of the initial material. Working with coarse materials, segregation of the different size fractions is a major problem, as different size fractions will have varying properties. Segregation must be avoided through the conscientious use of splitting devices and strict adherence to procedure.

9.3 Splitting gross samples can be accomplished in multiple stages of reduction in both size and amount. Reduction in the particle size of the material or crushing should precede the lowering in the amount or splitting of the gross sample to retain the representative nature of the initial sample, and is relative to the amount of material present at each stage of reduction.

NOTE 7—High-moisture content in limestone causes problems relative to reduction of the bulk sample (splitting) to the test sample, as well as, in the sizing operations. In this case drying becomes a necessity.

9.4 *Splitting Devices:*

9.4.1 *Riffle Splitter*—A riffle splitter is a device that should have an equal number of adjacent chutes of identical widths opening into adjacent buckets or collection pans. The bucket or pan feeding an open splitter should be of an identical width to the chutes. The width of the chutes should be at least three times the width of the nominal top size of the material being split. Some splitters have a catch basin, which holds the material before reduction. In this case, the material must be spread across the entire width of the splitter. The important consideration is that each chute has an equal selection probability.

9.4.2 *Sectorial Splitter*—A sectorial splitter is a radial device with either a revolving or stationary feeder, useful for obtaining identical samples for comparative testing. With this type of device, the revolving speed and material flow rate must be as constant and uniform as possible. All sectors of the

splitter must be radial and equal in size with no escape of material from any sector.

9.4.3 *Coning and Quartering*—Coning and quartering is an old method that can be used for small or large lots, in which a conical pile is created and quartered to obtain a sample.

9.4.3.1 Mix and spread the sample material on a clean surface, and then heap into a conical pile by placing shovels of material onto the apex of the cone. Flatten the cone symmetrically from the center to form flat circular pile.

9.4.3.2 The divide the pile into equal and identical quarters with a shovel or straightedge. Randomly select the subsample from one or more of the quarters. To further divide the subsample, the coning and quartering process must be repeated.

9.5 *Sizing Samples for Laboratory Analysis:*

9.5.1 As necessary, crush the sample (Note 8) by a suitable means to a size small enough to be easily handled by the laboratory's pulverizing equipment or to a size required by the analytical method.

NOTE 8—If physical testing is to be carried out on the sample, it is recommended that a second sample be sent to the laboratory solely for the purpose of physical testing. If screening of the sample is required to determine physical size characteristics, the second sample, or a representative subsample of the original sample, must not be further prepared from "as received" by the laboratory.

9.5.2 Utilizing suitable pulverizing equipment, further reduce the sample (or subsample) to an appropriate size for the analytical procedure to be performed on the sample. (Note 9). Please refer to Table 2 as a reference for pulverizing.

NOTE 9—When pulverizing sample to a specific mesh sieve, it is incorrect to screen a sample on the desired sieve and discard the fraction of the sample retained on the sieve. It is imperative that the whole (total) sample be used. Through previous experience with the particular grinding equipment, the grind produced should already be known so that no actual screening is required.

10. Rejection

10.1 Specific circumstances between the manufacturer and the purchaser may dictate that contractual agreements will supersede the recommendations of rejection.

10.2 Rejection of material based on failure to pass tests prescribed in the specification shall be reported to the manufacturer within 1 week after tests have been completed or within 30 days after shipment has been received, and the cause for rejection shall be stated.

10.3 The samples that represent rejected material shall be kept in airtight, moisture-proof containers for at least 2 weeks from the date of the original test report as reported to the manufacturer.

11. Retesting

11.1 Specific circumstances between the manufacturer and the purchaser may dictate that contractual agreements will supercede the recommendations of retesting.

11.2 Either of the contracting parties may make claim for a retest within 2 weeks of the date of the original test report. The expense of the retest shall be borne by the party demanding such retest.

11.3 Should the contracting parties be unable to reach a mutually satisfactory agreement based upon the results of the original test, the third sample of the material shall be delivered unopened to a mutually satisfactory referee laboratory for test. The results of this referee test shall be binding on both parties.

12. Packaging

12.1 Lime and limestone products may be shipped in bulk or in containers agreed upon between the manufacturer and the purchaser.

12.2 All packages shall be in good condition at the time of inspection.

13. Marking

13.1 Unless otherwise agreed upon between the manufacturer and the purchaser, each package shall have legibly marked thereon the name of the product, the net weight of its contents, the name of the manufacturer, the place of manufacture, and the brand name, if any.

13.2 In addition to the preceding information, the following may be marked on each package of shipment: "The contents meet the requirements of Practice C50/C50M."

TABLE 2 Sample Preparation Equipment[A,B,C]

Sample Size	Particle Size of Analytical Sample (Nominal Top Size)	Type of Equipment	Applicable Test Procedures
> 50 grams	<3.35 mm (No. 6 sieve)	Jaw Crusher Hammermill Roll Crusher Disc Mill	C110
> 1 gram < 50 grams	300 µm (No. 50 sieve)	Others- if adjustable Hammermill (with appropriate discharge screen) Disc Mill	C25 C110 C400
> 0.5 gram < 1 gram	150 µm (No. 100 sieve)	Mechanical Mortar and Pestle[D] Ring and Puck Mill	C25
< 0.5 gram	<75 µm (<No. 200 sieve)	Mechanical Mortar and Pestle[D] Ring and Puck Mill	C25 C1271[E] C1301[E]

[A] The Precision, associated with specific analytical procedures in relation to the sample weight, can be greatly effected by the particle size of the analytical sample. Therefore this chart is provided as a guide to obtain the desired particle size with the equipment suggested.
[B] Depending upon the testing requirements, such as for color (whiteness) or trace elements (iron, aluminum, chrome, nickel, tungsten, etc.), contamination may result from the preparation equipment, particularly when grinding to finer than 50 mesh. Therefore, materials of equipment construction must be considered in regard to testing requirements and sample particle size.
[C] The above list is not complete as comparable equipment may exist.
[D] Due to the hydroscopic nature of quicklime and the time involved in pulverizing, use of a hand Mortar is not recommended.
[E] Because of the nature of the test described within this standard, a finely pulverized homogenous sample is required regardless of the analytical sample size.