ASTM INTERNATIONAL

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Standard Practice for
Sampling Industrial Chemicals[1]

This standard is issued under the fixed designation E300; 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 U.S. Department of Defense.

1. Scope

1.1 This practice covers procedures for sampling several classes of industrial chemicals. It also includes recommendations for determining the number and location of such samples, to ensure their being representative of the lot in accordance with accepted probability sampling principles.

1.2 Although this practice describes specific procedures for sampling various liquids, solids, and slurries, in bulk or in packages, these recommendations only outline the principles to be observed. They should not take precedence over specific sampling instructions contained in other ASTM product or method standards.

1.3 These procedures are covered as follows:

	Sections
Statistical Considerations	7 – 11
Simple Liquids	12 – 27
Solids	28 – 35
Slurries	36 – 41

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.* Specific precautionary statements are given in Sections 6, 19, 20, 30, 34 and 37.

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D270 Method of Sampling Petroleum and Petroleum Products[3]
D2234/D2234M Practice for Collection of a Gross Sample of Coal

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *simple liquid*—a single-phase liquid having a Reid vapor pressure of less than 110 kPa at 37.8°C (16 psi at 100°F) and a Saybolt viscosity of less than 10 000 s (2160 cSt) at 25°C.

3.1.2 *lot*—a discreet quantity of material. It may contain a single batch or several batches, or be the product of continuous process broken into units on the basis of time or shipment. It is very desirable that individual batches in a lot be specifically identified so that they may become individual or stratified units for inspection.

3.1.3 *average sample*—one that consists of proportionate parts from all sections of the container.

3.1.4 *spot sample*—a sample taken at a specific location in a tank or from a flowing stream in a pipe at a specific time.

3.1.5 *composite sample*—a blend of spot samples mixed in proportion to the volumes of material from which the spot samples were obtained.

3.1.6 *all-levels sample*—one obtained by submerging a closed sampler to a point as near as possible to the draw-off level, then opening the sampler and raising it at a rate such that it is about three fourths full as it emerges from the liquid. An all-levels sample is not necessarily an average sample because the tank volume may not be proportional to the depth and because the operator may not be able to raise the sampler at the variable rate required for proportionate filling. The rate of filling is proportional to the square root of the depth of immersion.

NOTE 1—The tube sampling procedure, 26.3, may be used to obtain an all-levels sample from a drum.

3.1.7 *upper sample*—a spot sample obtained from the middle of the upper third of the tank contents (Fig. 1).
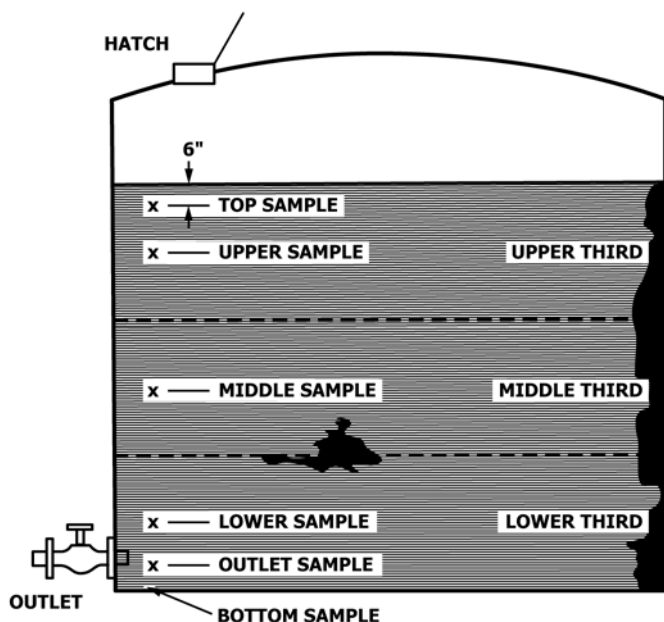
FIG. 1 Sampling Depths

NOTE 2—The taking of samples from various levels of the tank permits the detection of variation in composition of the contents caused by stratification. If it is known that the contents are not subject to this variation, the taking of samples at multiple levels may be eliminated.

3.1.8 *middle sample*—a spot sample obtained from the middle of the tank contents (Fig. 1) (Note 2).

3.1.9 *lower sample*—a spot sample of liquid from the middle of the lower one-third of the tank's content (a distance of one-half of the depth of liquid below the liquid's surface) (Fig. 1).

3.1.10 *single-tank composite sample*—a blend of the upper, middle, and lower samples. For a tank of uniform cross section, such as an upright cylindrical tank, the blend consists of equal parts of the three samples. For a horizontal cylindrical tank, the blend consists of the three samples in the proportions shown in Table 1.

3.1.11 *compartment-tank composite sample (ship, barge, etc.)*—a blend of individual all-levels samples from each compartment, which contains the product being sampled, in proportion to the volume of material in each compartment.

3.1.12 *top sample*—a spot sample normally obtained 150 mm (6 in.) below the top surface of the tank contents (Fig. 1).

TABLE 1 Sampling Instructions for Horizontal Cylindrical Tanks

Liquid Depth, Percent of Diameter	Sampling Level, Percent of Diameter Above Bottom			Composite Sample Proportionate Parts of		
	Upper	Middle	Lower	Upper	Middle	Lower
100	80	50	20	3	4	3
90	75	50	20	3	4	3
80	70	50	20	2	5	3
70	...	50	20	1	5	4
60	...	50	20	...	5	5
50	...	40	20	...	4	6
40	...	...	20	...	...	10
30	...	...	15	...	...	10
20	...	...	10	...	...	10
10	...	...	5	...	...	10

3.1.13 *outlet sample*—a spot sample normally obtained with the inlet opening of the sample apparatus at the level of the bottom of the tank outlet (either fixed or a swing line outlet) (Fig. 1).

3.1.14 *continuous sample*—a spot sample obtained from a pipeline conveying the product in such a manner as to give a representative average of the stream throughout the period of transit.

3.1.15 *jar sample*—a spot sample obtained by placing a jar into the path of a free-flowing stream so as to collect a definite volume from the full cross section of the stream.

3.1.16 *mixed sample*—a spot sample obtained after mixing or vigorously stirring the contents of the original container, and then pouring out or drawing off the quantity desired.

3.1.17 *tube or thief sample*—a spot sample obtained with a sampling tube or special thief, either as a core sample or spot sample from the specified point in the container.

3.1.18 *drain sample*—a spot sample obtained from the draw-off or discharge valve. Occasionally, a drain sample may be the same as a bottom sample, as in the case of a tank car.

3.1.19 *bottom sample*—a spot sample obtained from the material on the bottom surface of the tank, container, or line at its lowest point (Fig. 1). (Drain and bottom samples are usually taken to check for water, sludge, scale, etc.).

3.1.20 *laboratory sample*—that portion of the sample which is sent for laboratory testing.

4. Summary of Practice

4.1 This practice describes procedures to be followed for obtaining samples of several classes of industrial chemicals. It addresses in detail the various factors which need to be considered to obtain a representative laboratory sample. This practice also covers the statistical considerations in sampling of industrial chemicals whether they are liquids, solids or slurries in bulk or in packages.

5. Significance and Use

5.1 Representative samples of industrial chemicals are required for the determination of chemical and physical properties which are used to establish standard volumes, prices, and compliance with commercial and regulatory specifications.

5.2 The objective of sampling is to obtain a small portion (spot sample) of material from a selected area within a container which is representative of the material in the area or, in the case of running or all-level samples, a sample whose composition is representative of the total material in the container. A series of spot samples may be combined to create a representative sample.

5.3 *Manual and Automatic Sampling Considerations*—The selection of manual or automatic sampling devices is part of establishing a sampling plan applied under all conditions within the scope of this practice provided that the proper sampling procedures are followed. Both types of sampling are commonly used for liquid, solid, and slurry sampling and require adherence to the following:

5.3.1 An adequate frequency of sampling must be selected.

5.3.2 The equipment to support manual or automatic sampling systems may be obtained commercially, fabricated from the designs presented in this practice, or constructed as needed to satisfy process design or other specific requirements.

5.3.3 The sampling equipment must be maintained on a regular basis, and the sampling plan adopted must be strictly followed.

6. Safety Precautions

6.1 This practice covers procedures and sampling equipment used to sample industrial chemicals that may be potentially hazardous to personnel or the environment. Accordingly, it is emphasized that all applicable safety rules, regulations, and procedures must be followed in handling and processing the chemicals. Furthermore, this practice does not purport to cover all safety aspects associated with sampling. However, it is presumed that the personnel performing sampling operations are adequately trained with regard to safe application of the procedures contained herein for the specific sampling situation.

6.2 The characteristics of the material to be sampled will govern the type of protective equipment required. Since sampling may present such hazards as splashing or spilling, protective clothing must be worn when the chemical is capable of producing eye or skin irritation or burns. During such potential exposures, chemical-type goggles or face shield and protective gloves, or combination thereof, must be worn.

6.3 Respiratory protection, where required, must be in good condition and must be suitable to protect against chemicals being handled.

6.4 When sampling chemicals that may be dangerous to life by skin absorption, oral ingestion, or by breathing the vapor, unusual precautions will be indicated. In such cases, full-body protection such as supplied by a gas-tight or one-piece air-supplied suit should be worn. A second person must be continuously present to summon help and render aid in the event of an emergency.

STATISTICAL CONSIDERATIONS[5]

7. Objectives

7.1 The sampling and testing of industrial chemicals may have one or more of the following objectives:

7.1.1 The objective may be to estimate the average quality characteristic of a given lot of material and to establish confidence limits for this average. This would be the main objective, for example, if a dollar value is to be placed on the material for customs purposes or for sale.

7.1.2 The objective may be to decide whether the average value for the lot meets a specification. This calls for an acceptance sampling plan with the criterion being related to the estimated mean of the lot.

7.1.3 The objective may be to estimate or make decisions about the variability of a quality characteristic within the lot.

7.1.4 The objective may be to obtain simultaneous estimates of the mean and variance or to make decisions about some joint combination of these estimates.

7.1.5 If the material comes in containers or can be viewed as coming in clearly demarked units, the objective may be that of estimating the number of such units outside of specifications, that is, the "fraction defective."

NOTE 3—Procedures are given below for estimating average quality and for applying acceptance sampling inspection based on the lot mean.

8. General Sampling Considerations

8.1 To obtain samples that are representative in a statistical sense, one must consider such factors as physical form, uniformity, type and number of containers, etc. All of these factors influence the choice of method for performing the sampling operation, as well as the number and location of the required samples. Two commonly used practices for selecting the sequence or location of the individual samples are described.

8.2 *Random Sampling* is achieved when every part of the lot has an equal chance of being drawn into the sample.

8.2.1 Designate all units in the lot, choosing numbers in sequence or other serial code so that sampling by random numbers can be employed.

8.2.2 Preferably, this sequence should be in direct relation to order of manufacture of packaging as an aid to observing, from the sample results, any evidence of stratification.

8.2.3 Random selection of the numbers should be accomplished by chance or preferably by the use of a table of random numbers.

8.3 *Stratified Sampling* can be employed to estimate average quality when it is known or suspected that the value of a property of the material varies in non-random fashion throughout the lot for the following typical reasons: (*a*) the lot may contain several production batches, (*b*) the lot may contain units produced by different procedures, equipment, shifts, etc., or (*c*) the lot may be non-uniform because of subsequent size segregation, moisture pickup, surface oxidation, etc. If the assumed pattern is correct, the variance of the population mean estimate will be less than that based on random sampling. If the assumptions are incorrect, the estimate of the mean may be biased. A stratified sample can be obtained as follows:

8.3.1 Based on the known or suspected pattern, divide the lot into a number of real or imaginary strata.

8.3.2 If these sections are not equal in size, the number of samples to be taken from each stratum must be proportional to the size of the various strata.

8.3.3 Further subdivide the major strata into real or imaginary subsections and select the required number of samples by chance or preferably by means of a table of random numbers.

9. Estimate of Average Quality

9.1 *Determination of the Variance of a Sample Mean*—If the material comes in, or can be viewed as coming in, realizable primary units, each of which are to be divided into realizable secondary units, and if n_b primary units are selected at random from a lot of N primary units, and if n_w secondary units are selected from each primary unit with k tests being made on

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[5] Prepared by an Ad Hoc Committee of ASTM Committee E11 on Statistical Methods.

each secondary unit drawn, then the variance of the mean of the results is given as follows (Note 4 and Note 5):

$$\sigma_{\bar{x}}^2 = (\sigma_b^2/n_b) \times [(N - n_b)/N] + [\sigma_w^2/(n_b \times n_w)] + (\sigma_t^2/n_t) \quad (1)$$

where:

$\sigma_{\bar{x}}^2$ = variance of the mean,
σ_b^2 = variance of primary units (the material in cars, tanks, cans, drums, bottles, or other containers) in the lot,
σ_w^2 = average variance of secondary units (all-level, tube, thief, or similar samples) from a primary unit,
σ_t^2 = variance of tests on a homogeneous sample,
N = number of primary units in the lot,
n_b = number of randomly selected primary units from which secondary units are drawn,
n_w = number of randomly drawn secondary units from each of the n_b primary units, and
n_t = total number of tests made on all units, including replicates.

9.1.1 Eq 1 is also applicable when the $n_b \times n_w$ secondary units are composited into a single sample before testing. If there is no compositing and k tests are made on each secondary unit, $\bar{X}$ will be an arithmetic average of $n_t = k \times n_b \times n_w$ test results. If the secondary units are composited and k_c tests are made on the composite sample, $\bar{X}$ will be an arithmetic average of $n_t = k_c$ results.

Note 4—Uniform quantities (weight or volume, as appropriate) in the primary units and in the secondary units are assumed. If the departure from uniformity is such that a material error would be introduced by using a simple mean, a weighted average should be used or, if the secondary units are composited, proportional compositing must be adhered to.

Note 5—The factor $(N - n_b)/N$ is the correction for sampling from a finite population. A corresponding correction is generally not necessary for secondary units and tests.

9.1.2 For homogeneous liquids $\sigma_w^2 = 0$, so that Eq 1 reduces to Eq 2:

$$\sigma_{\bar{x}}^2 = (\sigma_b^2/n_b) \times [(N - n_b)/N] + (\sigma_t^2/n_t) \quad (2)$$

9.1.3 If $n_b = N$, Eq 1 and Eq 2 reduce, respectively, to Eq 3 and Eq 4:

$$\sigma_{\bar{x}}^2 = [\sigma_w^2/(n_b \times n_w)] + (\sigma_t^2/n_t) \quad (3)$$

$$\sigma_{\bar{x}}^2 = \sigma_t^2/n_t \quad (4)$$

9.2 *Determination of* n_b, n_w, *and* n_t *When Basic Variances are Known*—When reliable estimates of the variances σ_b^2, σ_w^2, and σ_t^2 are available from experience with lots of the type involved, a set of equivalent combinations of n_b, n_w, and n_t may be calculated from Eq 1, each combination based on the same desired or specified variance of the mean, $\sigma_{\bar{x}}^2$. Similarly, sets of equivalent combinations may be calculated from Eq 2 and Eq 3.

Note 6—If the precision of the test method has been properly evaluated in accordance with Practice E180, an adequate estimate of σ_t^2 can be obtained from the repeatability standard deviation (s_a) based on approximately 30 degrees of freedom.

9.2.1 Choice of a particular combination in a set for a specific lot is optional. In general, one combination in a set is most economical under given cost conditions and is therefore to be preferred.

9.3 *Procedure When Basic Variances are Unknown:*

9.3.1 Select at random a likely or convenient number, n_1 (10 or more), of primary units from the lot, take one secondary unit from each, and test each secondary unit. Estimate the variance of a measurement of a primary unit, s_1^2 (a variance that includes between and within unit variability as well as test variability), using Eq 5:

$$s_1^2 = \sum (X - \bar{X}_1)^2/(n_1 - 1) \quad (5)$$

where $\bar{X}_1$ is the mean of the individual test results on the n_1 primary units, with one secondary unit per primary unit and one test per secondary unit.

9.3.2 Decide to estimate the mean of the lot from single tests on single secondary units from n_2 primary units where $n_2 > n_1$ and the n_2 units include the n_1 preliminary units, the value on n_2 being determined from Eq 6:

$$n_2 = s_1^2/T_{S^2_{\bar{x}}} \quad (6)$$

where $T_{S^2_{\bar{x}}}$ is the target value of an estimate of the variance of $\bar{X}$. The target value $T_{S^2_{\bar{x}}}$ will depend on the width of the desired confidence interval. If it is hoped to have a 0.95 confidence interval of width 2Δ, then for $n_2 > 30$, $T_{S^2_{\bar{x}}}$ should be taken as $(\Delta/1.96)^2$. For smaller values of n_2, the 1.96 should be replaced by the 0.025 values from a t-table.

9.3.3 Estimate the variance of the mean after n_2 tests from Eq 7:

$$s_{\bar{x}}^2 = \sum (X - \bar{X})^2/n_2(n_2 - 1) \quad (7)$$

9.4 *A Confidence Limits for the Mean of the Lot:*

9.4.1 If the basic variances are known and two-stage sampling (primary and secondary units) is employed, then 0.95 confidence limits for the mean of the lot μ are given by Eq 8:

$$0.95 \text{ confidence limits for } \mu = \bar{X} \pm 1.96\, \sigma_{\bar{x}} \quad (8)$$

where $\sigma_{\bar{x}}$ is obtained from the $\sigma_{\bar{x}}^2$ value given by Eq 1.

9.4.2 If the basic variances are unknown and the variance of $\bar{X}$ is estimated as in 9.3 (n_s sample primary units with one secondary unit per sample primary unit and one test per secondary unit), then 0.95 confidence limits for the mean of the lot μ are given by Eq 9:

$$0.95 \text{ confidence limits for } \mu = \bar{X} \pm t_{0.025} s_{\bar{x}} \quad (9)$$

where $s_{\bar{x}}$ is obtained from the $s_{\bar{x}}^2$ value given by Eq 7 and $t_{0.025}$ can be taken as equal to 1.96 if n_2 is greater than 30, but otherwise should be taken from a table of t-values for $n_2 - 1$ degrees of freedom.

10. Acceptance Sampling for a Lot Mean—Basic Variances Unknown

Note 7—This section describes a simple random sampling plan for the acceptance inspection of an isolated lot and provides for buyer's and seller's risks of making a wrong decision. If a series of lots is to be inspected and knowledge of the basic variances is available, significant savings may be realized by testing composites.

10.1 *Introduction*—If a specification requires, for example, that the average purity or assay of a lot be no less than 98.0 %, it it sometimes assumed that the sampling and testing plan will accept all lots of 98.0 % or higher, but will detect or reject any

4

lot falling below this value. This ideal situation is not statistically realistic, as the required degree of discrimination can be *approached* only if the lot units are essentially uniform and the test procedure is capable of attaining a very high level of precision. It is necessary, therefore, that the contracting parties realize that any sampling plan based on a low probability of rejecting a lot which, in fact, is 98.0 % or higher in purity, may also permit acceptance of some lots below this specification minimum. Accordingly, such specifications must be viewed as incorporating both a buyer's and seller's risk. The following procedures are based on this concept.

10.2 *Single Lower Specification Limit (L); Simple Random Sampling from a Large Lot:*

10.2.1 *Procedure:*

10.2.1.1 *Step 1*—Note the value of the lower specification limit for average lot quality and designate it by L. Assume this value to represent a quality level for which the probability of acceptance should be high and the risk of rejection low. In this procedure, the seller's risk is taken to be 0.05.

10.2.1.2 *Step 2*—Establish a lower value for the barely tolerable lot quality for which the level of acceptance should be low and designate it by $L - \Delta$. Here, this buyer's risk is taken to be 0.10.

10.2.1.3 *Step 3*—Take a preliminary sample of n_1 (equals 10 or more) units at random from the lot and compute

$$\bar{X} = \sum_{i=1}^{n_1} X_i/n_1, \text{ and} \tag{10}$$

$$s_i = \sqrt{\sum_{i=1}^{n_1} \left(X_i - \bar{X} \right)^2/(n_1 - 1)} \tag{11}$$

$$\text{Set } \hat{\sigma}_1 = s_1 \tag{12}$$

10.2.1.4 *Step 4*—Note the value of Δ agreed to in Step 2. Compute $\lambda_1 = \Delta / \hat{\sigma}_1$ and find from Table 2 the value of n that comes closest to that given by the computed value of λ_1. Call this n_2.

10.2.1.5 *Step 5*—Randomly select $n_2 - n_1$ additional units from the lot. Compute

$$\bar{X}_2 = \sum_{i=1}^{n_2} X_1/n_2, \text{ and} \tag{13}$$

$$s_2 = \sqrt{\sum_{i=1}^{n_2} \left(X_i - \bar{X} \right)^2/(n_2 - 1)} \tag{14}$$

TABLE 2 Values[A] of Sample Size (*n*) for Agreed Upon Values of Δ

$\lambda = \Delta/\sigma$	Sample Size (n)
2.76	3
2.16	4
1.61	5
1.26	7
1.00	10
0.79	15
0.68	20
0.54	30
0.42	50
0.33	75
0.29[B]	100

[A] Values of λ were read from Fig. 13.31 of Bowker and Lieberman, *Handbook of Industrial Statistics.*
[B] For larger size samples, take $n = (2.927)^2/\lambda^2 = 8.57/\lambda^2$.

10.2.1.6 *Step 6*—Check on the adequacy of n_2 by taking $\hat{\sigma}_2 = s_2$. Compute $\lambda_2 = \Delta / \hat{\sigma}_2$. Enter Table 2 and find the value of n corresponding to λ_2. Call this n_3. If n_3 is much greater than n_2, for example, more than 20 %, randomly select $n_3 - n_2$ additional units from the lot and return to Step 5. If n_3 is not much greater than n_2, proceed with Step 7.

10.2.1.7 *Step 7*—Using the final values obtained above, calculate the following and accept the lot if

$$\left[\left(L - \bar{X} \right)/\left(s_{\bar{x}} \sqrt{n} \right) \right] \leq t_{0.05} \tag{15}$$

where $n = n_1$, n_2, or n_3, whichever is applicable, $t_{0.05}$ is the upper 0.05 point of a t-distribution for $n - 1$ degrees of freedom, and $s = s_2$ or s_1 whichever is applicable. Otherwise, reject the lot.

10.2.2 *Example:*

10.2.2.1 Assume that a contract covered the purchase of a packaged material with a minimum purity specification of 98.0 %. The buyer and seller agreed that the probability of rejecting a lot of 98.0 % purity should be no greater than 0.05 and that of accepting a lot as low as 97.0 % should be no greater than 0.10. In this case, the pertinent levels are:

$$L = 98.0 \tag{16}$$

$$L - \Delta = 97.0$$

$$\Delta = 1.0$$

10.2.2.2 On testing samples from ten units, selected at random, the lot standard deviation was estimated to be:

$$s_{\bar{x}} = s_1 = 0.8 \tag{17}$$

The values for $\bar{X}$ and λ_1 were also calculated:

$$\bar{X} = 97.5\% \tag{18}$$

$$\lambda_1 = \Delta/s_1 = 1.0/0.8 = 1.25$$

10.2.2.3 Entering Table 2, the sample size n for $\lambda_1 = 1.25$ is found to be 7. Accordingly, no further sampling is required.

10.2.2.4 Substituting the above values in Eq 15:

$$\left(L - \bar{X} \right)/\left(s\bar{x}/\sqrt{n} \right) = (98.0 - 97.5)/\left(0.8/\sqrt{10} \right) \tag{19}$$

$$= \left(0.5 \times \sqrt{10} \right)/0.8 = 1.97$$

Since 1.97 is greater than 1.833 (the value for the upper 0.05 point of the t-distribution for 9 degrees of freedom), the lot should be rejected.

10.3 *Single Upper Specification Limit (U); Simple Random Sampling from a Large Lot*—The procedures of 10.2 will apply here except that U will replace L and $U + \Delta$ will replace $L - \Delta$. The criterion for acceptance will be:

$$\left(\bar{X} - U \right)/\left(s_{\bar{x}}/\sqrt{n} \right) \leq t_{0.05} \tag{20}$$

10.4 *Both Lower and Upper Specification Limits: Simple Random Sampling from a Large Lot*—Use the following sampling plan: Determine n, $\bar{X}$, and s as in 10.2.1. Accept the lot if

$$\left(L - \bar{X} \right)/\left(s_{\bar{x}}/\sqrt{n} \right) \leq t_{0.05}, \text{ and} \tag{21}$$

5

$$\left(\bar{X} - U \right) / \left(s_{\bar{x}} / \sqrt{n} \right) \leq t_{0.05} \qquad (22)$$

for $n - 1$ degrees of freedom. Otherwise, reject the lot.

10.5 *General Remarks:*

10.5.1 If Δ is small relative to the lot standard deviation, a large sample size will be required to attain the low 0.10 consumer's and 0.05 producer's risks.

10.5.2 If the estimate of the lot standard deviation is less than the true lot standard deviation, the sample size given by the above procedures will produce a sampling plan whose risks will be different from those planned for. There will be a greater seller's risk of having a lot rejected whose mean is equal to the desired L level. Also, the buyer's risk of accepting a lot, whose mean is below the $L - \Delta$ level for barely acceptable quality, will also be greater than 0.10 (how much greater depends on how far off the estimate of the lot standard deviation may be).

10.5.3 If the estimate of the lot standard deviation is greater than the true lot standard deviation, then the above procedures will give a sample size (n) that is greater than necessary to yield the agreed upon risks. It will thus unnecessarily increase sampling costs.

10.5.4 The risks stated in this practice are based on the assumption that variability among units of the lot follows a normal distribution and that the total quantity of material in subsamples taken for testing does not exceed 10 % of the total quantity in the lot. If variability among units shows evidence of considerable skewness, the logarithms of the data (or other transformation) should be used.

10.5.5 If the sample units are taken from bulk material by a given sampling device, these risks are also based on the assumption that the sampling device is used in taking both the preliminary sample and the total sample.

11. Acceptance Sampling for the Mean of a Lot from a Stream of Batched Material for Which the Basic Variances Have Been Previously Estimated

11.1 *Some Basic Considerations*—To understand the recommendations of this section, it is helpful to review briefly the nature of an operating characteristic (OC) curve for an acceptance sampling plan.

11.1.1 The OC curve of acceptance sampling plan gives the probability of acceptance of a lot with reference to a hypothetical stream of lots. Two types of streams are generally considered. These are designated as Type A and Type B. A Type A stream is a stream of lots that are identical in every respect to the lot currently being inspected. A Type B stream of lots of the same size as the lot currently being inspected that would be generated by a controlled process. When we are faced with the inspection of an isolated lot, it seems appropriate to view the risks of the sampling inspection with reference to a Type A stream. We have little or no knowledge of the process from which the lot came and a decision on the lot would seem best based on data from that lot alone. This is the case considered in Section 10 of this practice; the isolated lot with unknown standard deviation.

11.1.2 In the present section, reference is to a process that is producing a stream of lots in batches. We assume that the within-batch and between-batch variations are independent and random with constant variances and on the basis of these assumptions we run a pilot study of variances that we take to hold valid for subsequent lots from the process. The current lot being inspected is recognized from the start as being one of the stream of lots coming from the given process and, as such, we are willing to use information about within-batch and between-batch variances obtained in the pilot study as part of the total information on which a decision about the lot is based. In this section, therefore, the probability of acceptance will be with reference to a Type B stream of lots, that is, with reference to a stream of lots from a controlled process. It follows in this case that the variance of a sample lot mean will be a function of both the within-batch and between-batch variances.

11.1.3 The recommended procedures of 11.2 call for compositing of increments and reduction for laboratory testing. As in the case of the batch variability, a preliminary study is made of the compositing and reduction processes and preliminary estimates are made of the reduction variance and the testing variance. It is again assumed that these same variances continue valid for the reduction and testing procedure employed in the inspection of the *current* lot. Recommended procedures for estimating the batch variances and the reduction and testing variances are given in the Annex. In the sections that follow, it will be assumed these estimates have been made.

11.1.4 *A Word of Advice*—Before a particular program is instituted, it would be desirable to review it with a statistician to be sure that the recommendations of Section 11 are thoroughly understood.

11.2 *Acceptance Tests Based on Current Samples:*

11.2.1 *Introduction*—With knowledge of the basic variances for the product and for the method of reduction and testing, the acceptability of a *current* lot from the given stream of material can be determined as follows:

11.2.2 *Formation of Composite Samples*—For the purpose of determining the acceptability of a current lot from the given stream of lots, proceed as follows: Let the lot consist of n_1 batches of material where n_1 is an integer. Presumably n_1 is determined by the needs of the purchaser with respect to his inventories, production, etc. (Note 9). Let n_2 increments of material be taken at random from each of the n_1 batches that make up the given lot and let n_2 be an even number. (The determination of n_2 is discussed in 11.2.4). If the batches are not distinct, take $n_1 n_2$ increments at random from the lot. Form a composite of all the odd numbered increments and another composite of all the even numbered increments. Call the first composite A, the second composite B. Reduce each composite separately and under uniform conditions run two tests on each composite.

NOTE 8—A fraction of a batch should be treated as a whole batch in determining n_1.

11.2.3 *Variance Formula*—The variance formula for the mean ($\bar{X}$) of the two composite samples with two tests per composite is

$$\sigma_{\bar{x}}^2 = \frac{\hat{\sigma}_b^2}{n_1} + \frac{\hat{\sigma}_w^2}{n_1 n_2} + \frac{\hat{\sigma}_r^2}{2} + \frac{\hat{\sigma}_t^2}{4} \qquad \cdots \qquad (23)$$

where:

$\hat\sigma_b{}^2$ = estimate made in the preliminary study of the between-batch variance,
$\hat\sigma_w{}^2$ = estimate of the within-batch variance,
$\hat\sigma_r{}^2$ = estimate of the reduction variance, and
$\hat\sigma_t{}^2$ = estimate of the testing variance.

11.2.4 *Determination of the Value of n_2 with a Single Lower Specification Limit (L)*—For a single lower specification limit, the procedure for determining the value of n_2 is as follows:

11.2.4.1 *Step 1*—Note the value of the lower specification limit for average product quality and designate it by *L*. Assume this value to represent a quality level for which the probability of lot acceptance should be high and the risk of lot rejection low. In the procedure for determining n_2, the seller's risk is taken to be 0.05.

11.2.4.2 *Step 2*—Determine a barely tolerable product quality for which the probability of lot acceptance should be low and designate this by $L - \Delta$. Here the buyer's risk is taken to be 0.10.

11.2.4.3 *Step 3*—Take n_2 as the even integer just greater than

$$n_2 = \frac{\hat\sigma_w{}^2}{n_1\left[(\Delta^2/8.5673) - (\hat\sigma_b{}^2/n_1) - (\hat\sigma_r{}^2/2) - (\hat\sigma_t{}^2/4)\right]} \quad \ldots \tag{24}$$

This n_2 will for the stated variances make the probability of lot acceptance for product quality *L* equal approximately to 0.95 and the probability of lot acceptance for product quality $L - \Delta$ equal to 0.10.

11.2.5 *Determination of the Value of n_2 with a Single Upper Specification (U)*—The procedure is the same as that of 11.2.4 except that *U* replaces *L* and $U + \Delta$ replaces $L - \Delta$. The formula for n_2 is the same.

11.2.6 *Determination of the Value of n_2 with Both a Lower and Upper Specification Limit*—The procedure is exactly the same as that of 11.2.4 and the formula for n_2 is the same. It is assumed that the spread between specification limits is at least $3\,\sigma_{\bar x}$.

11.2.7 *Sample Checks on the Basic Variances*—Before using Eq 1 in an acceptance test, a check should be made to see if the values previously determined for $\hat\sigma_b{}^2$, $\hat\sigma_w{}^2$, $\hat\sigma_r{}^2$, and $\hat\sigma_t{}^2$ are still valid. To check on $\hat\sigma_t{}^2$, compute the difference between the two tests for composite *A* and also the difference between the two tests for composite *B* and plot the two differences on an extension of Control Chart (*4*) described in the Annex. Proceed only if both of the two differences fall within the control limits. To check the remaining variances, set up a chart called Control Chart (*5*); the limits for which shall be

$$0 \ \text{and} \ 3.686\left(\frac{\hat\sigma_b{}^2}{n_1} + \frac{2\hat\sigma_w{}^2}{n_1 n_2} + \hat\sigma_r{}^2 + \frac{\hat\sigma_t{}^2}{2}\right)^{1/2} \tag{25}$$

and the central line on which shall be

$$1.128\left(\frac{\hat\sigma_b{}^2}{n_1} + \frac{2\hat\sigma_w{}^2}{n_1 n_2} + \hat\sigma_r{}^2 + \frac{\hat\sigma_t{}^2}{2}\right)^{1/2} \tag{26}$$

Plot on this chart the absolute value of the difference between the mean of composite *A* and the mean of composite *B*. Again proceed only if the difference falls below the upper

limit and does not, with previous points, yield a run of seven or more above the central line.

NOTE 9—If a point falls above the upper limit, this means that the purchaser's testing variance is probably greater than $\hat\sigma_t{}^2$. An estimate of the former based on additional data would consequently have to be made. The acceptance procedure could thus continue with the purchaser's test variance in place of the original $\hat\sigma_t{}^2$. This new estimate should be based on at least 20 degrees of freedom.

11.2.8 *Acceptance Test when there is a Single Lower Specification Limit(L):*

11.2.8.1 *Step 1*—Compute

$$\bar X_{La} = L - 1.645\left(\hat\sigma_b{}^2/n_1 + \hat\sigma_w{}^2/n_1 n_2 + \hat\sigma_r{}^2/2 + \hat\sigma_t{}^2/4\right)^{1/2} \ \ldots \tag{27}$$

11.2.8.2 *Step 2*—Accept the lot if $\bar X \geq \bar X_{La}$.

11.2.9 *The Acceptance Test when there is a Single Upper Specification Limit(U)*

11.2.9.1 *Step 1*—Compute

$$\bar X_{Ua} + U + 1.645\left(\hat\sigma_b{}^2/n_1 + \hat\sigma_w{}^2/n_1 n_2 + \hat\sigma_r{}^2/2 + \hat\sigma_t{}^2/4\right)^{1/2} \ \ldots \tag{28}$$

11.2.9.2 *Step 2*—Accept the lot if $\bar X \leq \bar X_{Ua}$.

11.2.10 *Acceptance Test when there are both a Lower Specification Limit(L) and an Upper Specification Limit (U):*

11.2.10.1 *Step 1*—Note whether $U - L$ is greater than

$$3\left(\hat\sigma_b{}^2/n_1 + \hat\sigma_w{}^2/n_1 n_2 + \hat\sigma_r{}^2/2 + \hat\sigma_t{}^2/4\right)^{1/2} \tag{29}$$

If it is, continue to Step 2. If it is not, do not continue.

11.2.10.2 *Step 2*—Compute $\bar X_{L_a}$ and $\bar X_{U_a}$ as in 11.2.8 and 11.2.9.

11.2.10.3 *Step 3*—Accept the lot if $\bar X_{La} \leq \bar X \leq \bar X_{Ua}$.

SIMPLE LIQUIDS

12. Scope

12.1 This procedure covers the sampling of industrial chemicals which are single-phase liquids under the conditions of sampling.

NOTE 10—This procedure is based on Method D270.

13. Summary

13.1 Samples of simple liquids are examined using various ASTM methods for the determination of physical and chemical characteristics. It is accordingly necessary that the samples be truly representative of the simple liquids in question. The precautions required to ensure the representative character of the samples are numerous and depend upon the type of product being sampled, the tank, the carrier or container from which the sample is being obtained, the type and cleanliness of the sample container, and the sampling procedure that is to be used. A summary of the sampling procedures and their application is presented in Table 3. Each procedure is suitable for sampling a number of specific products under definite storage, transportation, or container conditions. The basic principle of each procedure is to obtain a sample or a composite of several samples in such manner and from such locations in the tank or other container that the sample or composite will be truly representative of the product. Although single-phase liquids are homogeneous by definition, it may be desirable to check for this condition by sampling from various sections of the container.

Type of Container	Type of Sampling	Section
Storage tanks (trucks, cars, ships, barges, stationary)	Bottle sampling, thief sampling	22, 23
Storage tanks (trucks, cars, stationary)	Tap sampling	24
Pipe lines, filling lines, transfer lines	Continuous sampling	25
Drums, carboy, cans, bottles	Tube sampling	26
Free or open-discharge streams	Jar sampling	27

14. Sampling Equipment

14.1 *General Requirements*—all sampling apparatus and closures shall be clean, dry, free of contaminants, and constructed of materials that are inert to the product to be sampled. The sampling container and closure shall be clean, dry, and inert to the material being sampled.

14.2 *Bottles and Jars*—Bottles and jars may be made of clear or brown glass or polyethylene with necks shaped to receive a glass stopper or a screw cap made of metal or plastic material. Use of unprotected corks as closures is not recommended for general use. Where safety indicates (such as for peroxides) use corks covered with materials inert to the sample, such as cellophane, polyethylene, or aluminum foil. Clear glass is advantageous because the container may be examined visually for cleanliness and the sample may be visually inspected for foreign matter. Brown glass affords some protection for light-sensitive materials. Before using a bottle or jar, examine it to see that it is scrupulously clean. A variety of methods for cleaning glass containers may be used: washing with detergents, water, acetone, etc. The specific method used will depend upon the material to be sampled. Care should be taken that all of the cleaning agents are removed from the container prior to use. Dry the container either by passing a current of clean warm air through the container or by placing it in a dust-free cabinet at 40°C or higher. Close containers as soon as they are dry.

14.3 *Screw-Neck and Press-Cover Cans*—Cans of tin plate with seams soldered on the outside must be used. The neck should be shaped to receive a screw cap or pressed cover. Take care to ensure that cans are clean, even when new. They may be cleaned by washing with low-boiling, nonflammable solvents and blowing dry with clean air. Cap the containers as soon as they are dry.

15. Time and Place of Sampling

15.1 *Finished Products*—When loading or discharging finished products, take samples from both shipping and receiving tanks, and from the pipeline, if required.

15.2 *Ship or Barge Tanks*—Sample each product immediately after the vessel is loaded, or just before discharging.

15.3 *Tank Cars*—Sample the product immediately after the car is loaded, or just before unloading.

16. Number and Location of Samples

16.1 *Bulk Containers (Tanks, Tank Cars etc.)*—Simple liquids in bulk containers are frequently found to be homoge-neous and only limited sampling is usually required. Upper, middle, and lower samples (22.3) or top and outlet samples (22.5) can be individually tested to confirm this, by means of simple physical tests such as refractive index, density, viscosity, etc. Complete testing can then be performed on a composite prepared as described in 22.4.

16.2 *Packaged Materials (Drums, Cans, Bottles, etc.)*—In the case of lots of drums, bottles, and cans, the homogeneity of the lot cannot be assumed, and the required number of samples should be determined in accordance with Sections 7 and 8. The specific containers to be sampled for individual testing should be chosen by means of a table of random numbers.

17. Sampling Operations

17.1 Procedures for sampling cannot be made explicit enough to cover all cases. Extreme care and good judgment are necessary to ensure samples are obtained which represent the general character and average condition of the material. Clean hands are important. Clean gloves may be worn but only when absolutely necessary, such as during cold weather, or for reasons of safety. Select wiping cloths so that lint is not introduced, thus contaminating samples.

17.2 Since the vapors of some industrial chemicals are toxic and flammable, avoid breathing them, igniting them from an open flame, burning embers, or a spark produced by static electricity. All safety precautions specific to the material being sampled must be followed.

17.3 When sampling relatively volatile products, the sampling apparatus shall be filled and allowed to drain before drawing the sample. If the sample is to be transferred to another container, this container shall have been cleaned and dried as described in Section 14 and also be rinsed with some of the volatile product and then drained. When the actual sample is emptied into this container, the sampling apparatus should be upended into the opening of the sample container and remain in this position until the contents have been transferred so that no unsaturated air will be entrained in the transfer of the sample.

17.4 When sampling non-volatile liquid products, the sampling apparatus shall be filled and allowed to drain before drawing the actual sample. If the actual sample is to be transferred to another container, this container shall have been cleaned and dried as described in Section 14 and also be rinsed with some of the product to be sampled and drained before it is filled with the actual sample.

17.5 A sample shall be considered suspect under any of the following circumstances and should be referred to the appropriate supervisor before analysis:

17.5.1 The sample container is damaged or defective.

17.5.2 There is any doubt as to the nature of the contents of the sample container: for example, because of the presence of an old label, incorrect markings, or insufficient identification.

17.5.3 There is evidence of an unexpected lack of uniformity; for example, a separate layer or suspended matter.

17.5.4 Obvious and unusual variations are apparent in the sample.

17.5.5 The container closure is loose, whether or not there is evidence of leakage.

17.5.6 Evidence that the closure or liner has been attacked.

18. Size of Sample

18.1 The quantity of sample should be as specified by the test instructions, or at least three times greater than the minimum necessary for the actual tests.

19. Precautions

19.1 *Volatile Samples (Reid vapor pressure 14 to 110.3 kPa at 37.8°C (2 to 16 psi at 100°F))*—It is necessary to protect volatile samples from evaporation. Transfer the product from the sampling apparatus to the sample container immediately. Keep the container closed except when material is being transferred. After delivery to the laboratory, it is recommended to cool the containers before they are opened.

19.2 *Light-Sensitive Samples*—It is important that samples sensitive to light be kept in the dark if testing is to include the determination of such properties as color, inhibitor content, stability tests, or neutralization values. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately. It is a definite advantage to use covered metal or cardboard containers into which the sample bottles may be placed immediately after collection.

19.3 *Materials of High Purity*—Protect highly refined products from moisture and dust by placing paper, plastic, or metal foil over the closure and the top of the container.

19.4 *Container Outage*—Never completely fill a sample container, but allow adequate room for expansion, taking into consideration the temperature of the liquid at the time of filling and the probable maximum temperature to which the filled container may be subjected.

20. Shipping Precautions

20.1 To prevent the loss of liquid during shipment and to protect against moisture and dust, cover the closure of the glass bottle with plastic caps which have been swelled in water, wiped dry, placed over the top of the stoppered bottle, and allowed to shrink tightly in place. Screw-top bottles are recommended. The cap must be lined with material inert to the sample. The screw caps must be secured by use of adhesive tape or similar material.

NOTE 11—Shipping of any chemical must comply with current federal, state, and local regulations for the specific material being shipped.

21. Labeling Sample Containers

21.1 Label the container immediately after a sample is obtained. Use waterproof and oil-proof ink or a pencil hard enough to dent the tag, since soft pencil and ordinary ink markings are subject to obliteration from moisture, oil smearing, and handling. If gummed labels are used, they should be further secured with transparent sealing tape. Sufficient detail should be written on the label to completely identify the sample. The following information is frequently desired:

21.1.1 Date and time (and for continuous and dipper samples the hour and minute of collection),

21.1.2 Name of sampler,

21.1.3 Name or number and owner of the vessel, car, or container,

21.1.4 Brand name, grade of material, and code number, and

21.1.5 Reference symbol and necessary identification number.

21.1.6 Hazard ratings.

22. Bottle Sampling

22.1 The bottle sampling procedure is applicable for sampling simple liquids in tank cars, tank trucks, shore tanks, ship tanks, and barge tanks. A suitable sampling bottle, as shown in Fig. 2, is required. The diameter of the openings in the bottles should be 19 mm (¾ in.). Stopper and label bottles immediately after taking them and deliver them to the laboratory in the original sampling bottle.

NOTE 12—The designs and dimensions which follow are intended only as guides to the form that the sampling apparatus may take. When metal is required for construction of the sampling apparatus, a corrosion-resistant type steel should be selected (Type 316L may be suitable). If flammable materials are to be sampled, a nonmagnetic low-spark generating stainless steel is required. When sampling flammable liquids, exercise extreme care not to sharply strike the container being sampled with the sampling apparatus. Alternative procedures may be used if a mutually satisfactory agreement has been reached by the parties involved.

22.2 *All-Level Sample*—Lower the weighted, stoppered bottle as near as possible to the draw-off level, pull out the stopper with a sharp jerk of the twine or chain (spark-proof) attached to the stopper, and raise the bottle at such a rate that it is about three-fourths full as it emerges from the liquid.
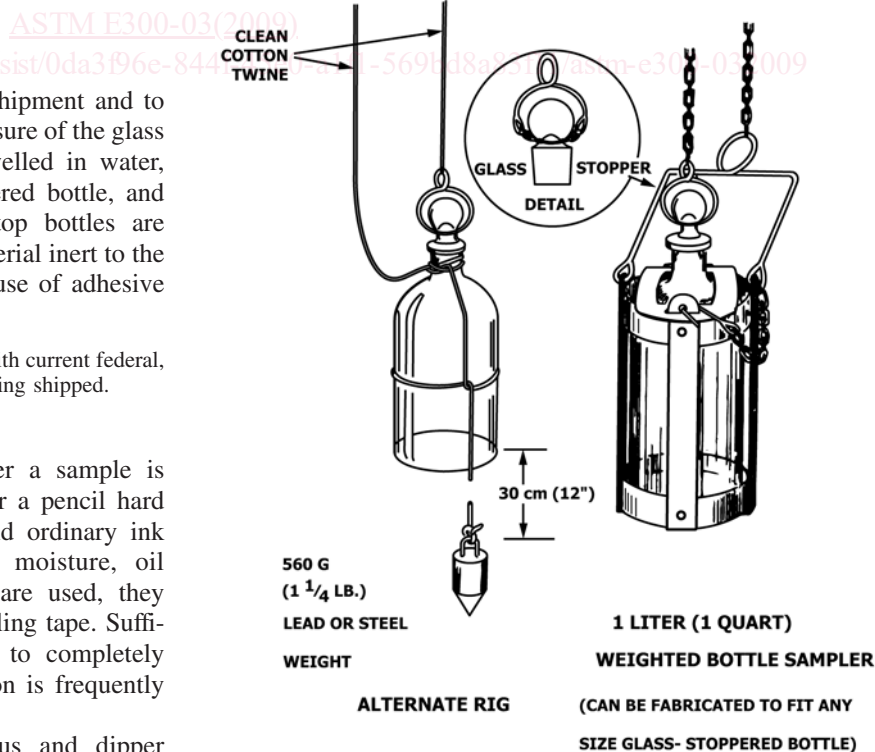

FIG. 2 Assembly for Bottle Sampling

22.3 *Upper, Middle, and Lower Samples*—Lower the weighted, stoppered bottle to the proper depths (Fig. 1), which are as follows:

Upper sample	middle of upper third of the tank contents
Middle sample	middle of the tank contents
Lower sample	middle of lower third of the tank contents.

Pull out the stopper with a sharp jerk of the twine or chain (spark-proof) attached to the stopper and allow the bottle to fill completely at the selected level, as evidenced by the cessation of air bubbles. When full, raise the bottle, pour off a small amount, and stopper immediately.

22.4 *Composite Sample*—Prepare a composite sample in the laboratory (not in the field) by mixing portions of all-levels samples as specified in 3.1.11 or by mixing portions of the upper, middle, and lower samples as specified in 3.1.10.

22.5 *Top and Outlet Samples*—Obtain these samples (Fig. 1) in the same manner as specified in 3.1.12 and 3.1.13, but at the following depths:

Top sample	150 mm (6 in.) below the top surface of the tank contents
Outlet sample	opposite the tank outlet (either fixed or swing line outlet)

23. Thief Sampling

23.1 The thief sampling procedure is applicable for obtaining bottom samples (Fig. 1), of liquids with Reid vapor pressure of 14 kPa at 37.8°C (2 psi at 100°F) or less, in tank cars and storage tanks.

23.2 *Thief*—The thief shall be designed so that a sample can be obtained within 13 mm (½ in.) of the bottom of the car or tank. Two types of thiefs are illustrated in Fig. 3. One type is lowered into the tank with valves open to permit the liquid to flush through the container. When the thief strikes the bottom of the tank, the valves shut automatically to trap a bottom sample. The other type has a projecting stem on the valve rod which opens the valves automatically as the stem strikes the bottom of the tank. The sample enters the container through the bottom valve and air is released simultaneously through the top. The valves snap shut when the thief is withdrawn.

23.3 *Procedure*—Lower the clean, dry thief through the dome of the tank car or tank hatch until it strikes the bottom. When full, remove the thief and transfer the contents to the sample container. Close and label the container immediately, and deliver it to the laboratory.

24. Tap Sampling

24.1 The tap sampling procedure is applicable for sampling simple liquids in tanks which are equipped with suitable taps or lines. The assembly for tap sampling is shown in Fig. 4.

24.2 *Tank Taps*—The tank should be equipped with at least three sampling taps placed equidistant throughout the tank
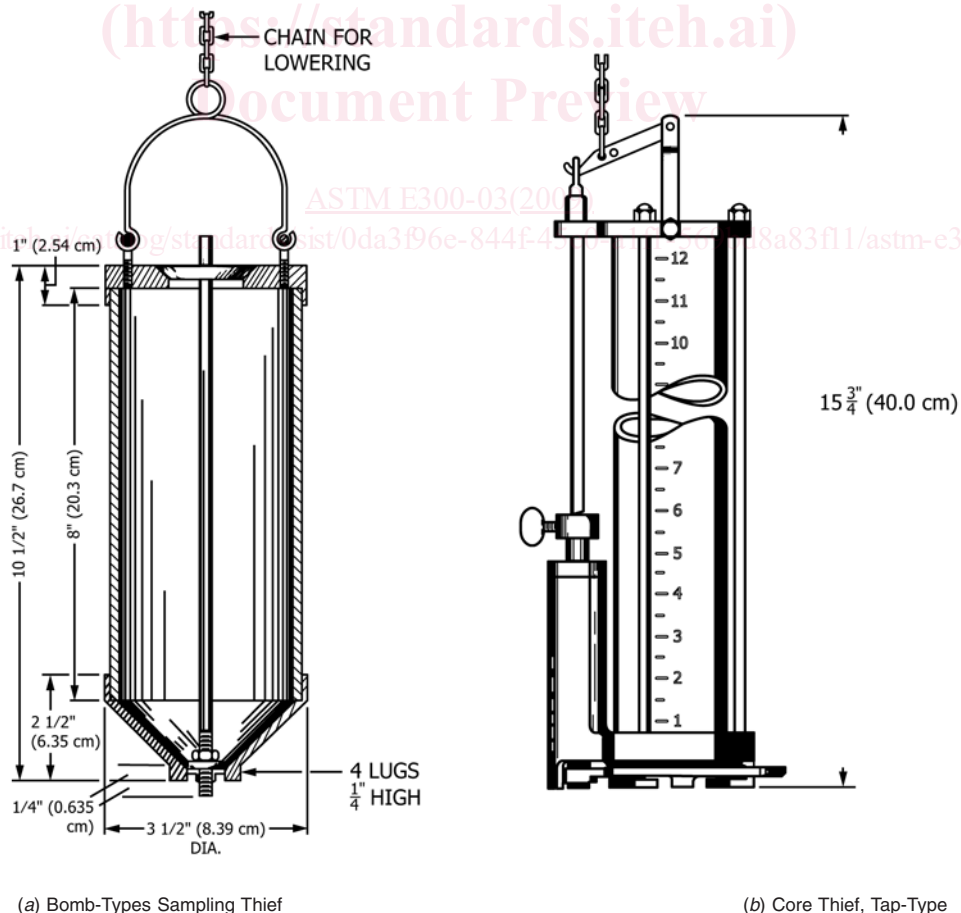

(a) Bomb-Types Sampling Thief

(b) Core Thief, Tap-Type

FIG. 3 Sampling Thiefs