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## Standard Practice for SAMPLING INDUSTRIAL CHEMICALS<sup>1</sup>

This standard is issued under the fixed designation E 300; 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.

### 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 Consideration ..... | 5 to 9   |
| Simple Liquids .....            | 10 to 25 |
| Solids .....                    | 26 to 34 |
| Slurries .....                  | 35 to 40 |

NOTE 1—It is intended to add sections on viscous liquids; multiphase liquids, partly solidified solids and liquefiable solids; liquefied gases; slurries; and gases, when available.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Sections 4, 18, 19, 29, 33 and 36.

### 2. Applicable Documents

#### 2.1 ASTM Standards:

D 270 Method of Sampling Petroleum and

#### Petroleum Products<sup>2</sup>

D 2234 Method for Collection of a Gross Sample of Coal<sup>3</sup>

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals<sup>4</sup>

### 3. Significance and Use

3.1 This practice outlines the principles to be observed when sampling several classes of industrial chemicals. This practice also covers the statistical considerations in the sampling of industrial chemicals whether they are liquids, solids, slurries, and in bulk or packages.

### 4. Safety Precautions

4.1 This practice covers procedures and sampling equipment used to sample industrial chemicals that may be potentially hazardous to personnel. Accordingly, it is emphasized that all applicable safety rules, regulations, and procedures must be followed in handling and processing the chemicals.

4.2 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 should 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

<sup>1</sup> This practice is under the joint jurisdiction of ASTM Committee E-15 on Industrial Chemicals and is the direct responsibility of Subcommittee E15.05 on Sampling.

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<sup>2</sup> Discontinued, see 1983 Annual Book of ASTM Standards, Vol 05.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 05.05.

<sup>4</sup> Annual Book of ASTM Standards, Vol 15.05.



combination thereof, should be worn.

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

4.4 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 man should be continuously present to summon help and render aid in the event of an emergency.

#### STATISTICAL CONSIDERATIONS<sup>5</sup>

### 5. Objectives

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

5.1.1 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.

5.1.2 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.

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

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

5.1.5 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 2—Procedures are given below for estimating average quality and for applying acceptance sampling inspection based on the lot mean.

### 6. General Sampling Considerations

6.1 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 influence the choice of method for performing the mechanical 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.

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

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

6.2.2 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.

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

6.3 *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 nonrandom 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 nonuniform 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:

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

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

6.3.3 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.

### 7. Estimate of Average Quality

7.1 *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

<sup>5</sup> Prepared on an Ad Hoc Committee of ASTM Committee E-11 on Statistical Methods.



secondary units, then 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 each secondary unit drawn, then the variance of the mean of the results is given as follows (Notes 3 and 4):

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

where:

$\sigma_{\bar{x}}^2$  = variance of the mean,

$\sigma_b^2$  = variance of primary units (the material in cars, tanks, cans, drums, bottles, or other containers) in the lot,

$\sigma_w^2$  = average variance of secondary units (all-level, tube, thief, or similar samples) from a primary unit,

$\sigma_t^2$  = 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.

7.1.1 Equation 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 3—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 4—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.

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

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

7.1.3 If  $n_b = N$ , Eq 1 and 2 reduce, respec-

tively, to Eq 3 and 4:

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

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

7.2 *Determination of  $n_b$ ,  $n_w$ , and  $n_t$  When Basic Variances are Known*—When reliable estimates of the variances  $\sigma_b^2$ ,  $\sigma_w^2$ , and  $\sigma_t^2$  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 3.

NOTE 5—If the precision of the test method has been properly evaluated in accordance with Practice E 180, an adequate estimate of  $\sigma_t^2$  can be obtained from the repeatability standard deviation ( $s_d$ ) based on approximately 30 degrees of freedom.

7.2.1 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.

7.3 *Procedure When Basic Variances are Unknown*:

7.3.1 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:

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

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.

7.3.2 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:

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

where  $T_{S_{\bar{x}}^2}$  is the target value of an estimate of the variance of  $\bar{X}$ . The target value  $T_{S_{\bar{x}}^2}$  will depend on the width of the desired confidence interval. If it is hoped to have a 0.95 confidence interval of width  $2\Delta$ , then for  $n_2 > 30$ ,  $T_{S_{\bar{x}}^2}$  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.



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

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

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

7.4.1 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  $\mu$  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.

7.4.2 If the basic variances are unknown and the variance of  $\bar{X}$  is estimated as in 6.3 ( $n_1$  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  $\mu$  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.

## 8. Acceptance Sampling for a Lot Mean—Basic Variances Unknown

NOTE 6—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.

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

corporating both a buyer's and seller's risk. The following procedures are based on this concept.

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

8.2.1 *Procedure:*

8.2.1.1 *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.

8.2.1.2 *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.

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

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

$$s_1 = \sqrt{\sum_{i=1}^{n_1} (X_i - \bar{X})^2/(n_1 - 1)} \quad (11)$$

$$\text{Set } \hat{\sigma}_1 = s_1 \quad (12)$$

8.2.1.4 *Step 4*—Note the value of  $\Delta$  agreed to in Step 2. Compute  $\lambda_1 = \Delta/\hat{\sigma}_1$  and find from Table 1 the value of  $n$  that comes closest to that given by the computed value of  $\lambda_1$ . Call this  $n_2$ .

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

$$\bar{X}_2 = \sum_{i=1}^{n_2} X_i/n_2, \text{ and} \quad (13)$$

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

8.2.1.6 *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 1 and find the value of  $n$  corresponding to  $\lambda_2$ . 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.

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

$$[(L - \bar{X})/(s/\sqrt{n})] \leq t_{0.05} \quad (15)$$

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.

#### 8.2.2 Example:

8.2.2.1 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:

$$\begin{aligned} L &= 98.0 \\ L - \Delta &= 97.0 \\ \Delta &= 1.0 \end{aligned}$$

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

$$\hat{\sigma}_1 = s_1 = 0.8$$

The values for  $\bar{X}$  and  $\lambda_1$  were also calculated:

$$\begin{aligned} \bar{X} &= 97.5 \% \\ \lambda_1 &= \Delta/\hat{\sigma}_1 = 1.0/0.8 = 1.25 \end{aligned}$$

8.2.2.3 Entering Table 1, the sample size  $n$  for  $\lambda_1 = 1.25$  is found to be 7. Accordingly, no further sampling is required.

8.2.2.4 Substituting the above values in Eq 15:

$$\begin{aligned} (L - \bar{X})/(s/\sqrt{n}) &= (98.0 - 97.5)/(0.8/\sqrt{10}) \\ &= (0.5 \times \sqrt{10})/0.8 = 1.97 \end{aligned}$$

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.

8.3 *Single Upper Specification Limit (U); Simple Random Sampling from a Large Lot*—The procedures of 8.2 will apply here except that  $U$  will replace  $L$  and  $U + \Delta$  will replace  $L - \Delta$ . The criterion for acceptance will be:

$$(\bar{X} - U)/(s/\sqrt{n}) \leq t_{0.05} \quad (16)$$

8.4 *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 8.2.1. Accept the lot if

$$(L - \bar{X})/(s/\sqrt{n}) \leq t_{0.05}, \text{ and} \quad (17)$$

$$(\bar{X} - U)/(s/\sqrt{n}) \leq t_{0.05} \quad (18)$$

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

#### 8.5 General Remarks:

8.5.1 If  $\Delta$  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.

8.5.2 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).

8.5.3 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.

8.5.4 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.

8.5.5 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.

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

9.1 *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.

9.1.1 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 8 of this practice; the isolated lot with unknown standard deviation.

9.1.2 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.

9.1.3 The recommended procedures of 9.2 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.

9.1.4 *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 9 are thoroughly understood.

## 9.2 Acceptance Tests Based on Current Sam-

ples:

9.2.1 *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:

9.2.2 *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 7). 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 9.2.4). 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 7—A fraction of a batch should be treated as a whole batch in determining  $n_1$ .

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

$$\sigma_{\bar{x}}^2 = \frac{\hat{\sigma}_b^2}{n_1} + \frac{\hat{\sigma}_w^2}{n_1 n_2} + \frac{\hat{\sigma}_r^2}{2} + \frac{\hat{\sigma}_t^2}{4} \quad \dots (1)$$

where:

$\hat{\sigma}_b$  = 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.

9.2.4 *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:

9.2.4.1 *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.

9.2.4.2 *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.

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

$$n_2 = \frac{\hat{\sigma}_w^2}{n_1[(\Delta^2/8.5673) - (\hat{\sigma}_b^2/n_1) - (\hat{\sigma}_r^2/2) - (\hat{\sigma}_t^2/4)]} \quad \dots(2)$$

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.

9.2.5 *Determination of the Value of  $n_2$  with a Single Upper Specification (U)*—The procedure is the same as that of 9.2.4 except that  $U$  replaces  $L$  and  $U + \Delta$  replaces  $L - \Delta$ . The formula for  $n_2$  is the same.

9.2.6 *Determination of the Value of  $n_2$  with Both a Lower and Upper Specification Limit*—The procedure is exactly the same as that of 9.2.4 and the formula for  $n_2$  is the same. It is assumed that the spread between specification limits is at least  $3 \sigma_{\bar{x}}$ .

9.2.7 *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  $\hat{\sigma}_r^2$ , 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

$$0 \text{ and } 3.686 \left( \frac{\hat{\sigma}_b^2}{n_1} + \frac{2\hat{\sigma}_w^2}{n_1 n_2} + \hat{\sigma}_r^2 + \frac{\hat{\sigma}_t^2}{2} \right)^{1/2}$$

and the central line on which shall be

$$1.128 \left( \frac{\hat{\sigma}_b^2}{n_1} + \frac{2\hat{\sigma}_w^2}{n_1 n_2} + \hat{\sigma}_r^2 + \frac{\hat{\sigma}_t^2}{2} \right)^{1/2}$$

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 8—If a point falls above the upper limit, this means that the purchaser's testing variance is probably greater than  $\hat{\sigma}_r^2$ . An estimate of the former based on

addition data would consequently have to be made. The acceptance procedure could thus continue with the purchaser's test variance in place of the original  $\hat{\sigma}_r^2$ . This new estimate should be based on at least 20 degrees of freedom.

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

9.2.8.1 *Step 1*—Compute

$$\bar{X}_{La} = L - 1.645 \left( \hat{\sigma}_b^2/n_1 + \hat{\sigma}_w^2/n_1 n_2 + \hat{\sigma}_r^2/2 + \hat{\sigma}_t^2/4 \right)^{1/2} \quad \dots(3)$$

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

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

9.2.9.1 *Step 1*—Compute

$$\bar{X}_{Ua} = U + 1.645 \left( \hat{\sigma}_b^2/n_1 + \hat{\sigma}_w^2/n_1 n_2 + \hat{\sigma}_r^2/2 + \hat{\sigma}_t^2/4 \right)^{1/2} \quad \dots(4)$$

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

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

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

$$3(\hat{\sigma}_b^2/n_1 + \hat{\sigma}_w^2/n_1 n_2 + \hat{\sigma}_r^2/2 + \hat{\sigma}_t^2/4)^{1/2}$$

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

9.2.10.2 *Step 2*—Compute  $\bar{X}_{La}$  and  $\bar{X}_{Ua}$  as in 9.2.8 and 9.2.9.

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

## SIMPLE LIQUIDS

### 10. Scope

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

NOTE 9—This procedure is based on Method D 270.

### 11. Summary

11.1 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 sam-



pling procedure that is to be used. A summary of the sampling procedures and their application is presented in Table 2. 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.

## 12. Definitions

12.1 *simple liquid*—a single-phase liquid having a vapor pressure of less than 16 psi Reid vapor pressure at 100°F (830 mm Hg at 37.8°C) and a Saybolt viscosity of less than 10 000 s (2160 cSt) at 25°C.

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

12.3 *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 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 10—The tube sampling procedure, 20.3, may be used to obtain an all-levels sample from a drum.

12.4 *upper sample*—one obtained from the middle of the upper third of the tank contents (Fig. 1).

NOTE 11—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.

12.5 *middle sample*—one obtained from the middle of the tank contents (Fig. 1) (Note 10).

12.6 *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 3.

12.7 *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.

12.8 *top sample*—one normally obtained 6 in. (152 mm) below the top surface of the tank contents (Fig. 1).

12.9 *outlet sample*—one normally obtained at the level of the tank outlet (either fixed or a swing line outlet) (Fig. 1).

12.10 *continuous sample*—one 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.

12.11 *jar sample*—one 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.

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

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

12.14 *drain sample*—one 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.

12.15 *bottom sample*—one 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.).

## 13. Sampling Equipment

13.1 *General Requirements*—all sampling apparatus and closures shall be clean, dry, free of contaminants, and constructed to 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.

13.2 *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, chromic acid cleaning solution, 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. Close containers as soon as they are dry.

**13.3 Screw-Neck and Press-Cover Cans—**Cans of tin plate with seams soldered on the outside should be used. The neck should be shaped to receive a screw cap or pressed cover. Care should be taken 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.

#### 14. Time and Place of Sampling

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

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

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

#### 15. Number and Location of Samples

**15.1 Bulk Containers (Tanks, Tank Cars etc.)—**Simple liquids in bulk containers are frequently found to be homogeneous and only limited sampling is usually required. Upper, middle, and lower samples (21.3) or top and outlet samples (21.5) can be individually tested to confirm this, by means of simple physical tests such as refractive index, specific gravity, viscosity, etc. Complete testing can then be performed on a composite prepared as described in 21.4.

**15.2 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 5 and 6. The specific containers to be sampled for individual testing should be chosen by means of a table of random numbers.

#### 16. Sampling Operations

**16.1** Procedures for sampling cannot be made explicit enough to cover all cases. Extreme care and good judgment are necessary to ensure samples 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, contaminating samples.

**16.2** 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 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.

**16.3** When sampling nonvolatile 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, the sample container shall be rinsed with some of the product to be sampled and drained before it is filled with the actual sample.

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

**16.4.1** The sample container is damaged or defective.

**16.4.2** 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.

**16.4.3** There is evidence of an unexpected lack of uniformity; for example, a separate layer



or suspended matter.

16.4.4 Obvious and unusual variations are apparent in the sample.

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

16.4.6 Evidence that the closure or liner has been attacked.

## 17. Size of Sample

17.1 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.

## 18. Precautions

18.1 *Volatile Samples (2 to 16 psi Reid Vapor Pressure at 100°F (105 to 830 mm Hg at 37.8°C))*—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.

18.2 *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.

18.3 *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.

18.4 *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.

## 19. Shipping Precautions

19.1 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 should be lined with material inert to the sample. The screw

caps should be secured by use of adhesive tape or similar material.

NOTE 12—Shipping of any chemical must comply with current federal, state, and local regulations for the specific material being shipped.

## 20. Labeling Sample Containers

20.1 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:

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

20.1.2 Name of sampler,

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

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

20.1.5 Reference symbol and necessary identification number.

## 21. Bottle Sampling

21.1 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 3/4-in. (19-mm). Stopper and label bottles immediately after taking them and deliver them to the laboratory in the original sampling bottle.

NOTE 13—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, extreme care should be exercised 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.

21.2 *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.

**21.3 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.

**21.4 Composite Sample—**Prepare a composite sample in the laboratory (not in the field) by mixing portions of all-levels samples as specified in 12.7 or by mixing portions of the upper, middle, and lower samples as specified in 12.6.

**21.5 Top and Outlet Samples—**Obtain these samples (Fig. 1) in the same manner as specified in 12.13, but at the following depths:

|               |                                                              |
|---------------|--------------------------------------------------------------|
| Top sample    | 6 in. (152 mm) below the top surface of the tank contents    |
| Outlet sample | opposite the tank outlet (either fixed or swing line outlet) |

## 22. Thief Sampling

**22.1** The thief sampling procedure is applicable for obtaining bottom samples (Fig. 1), of liquids of 2 psi Reid vapor pressure at 100°F (105 mm Hg at 37.8°C) or less, in tank cars and storage tanks.

**22.2 Thief—**The thief shall be designed so that a sample can be obtained with ½ in. (13 mm) of the bottom of the car or tank. Two types of thieves 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.

**22.3 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 content to the sample container. Close and label the container immediately, and deliver it to the laboratory.

## 23. Tap Sampling

**23.1** 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.

**23.2 Tank Taps—**The tank should be equipped with at least three sampling taps placed equidistant throughout the tank height and extending at least 3 ft (9 m) inside the tank shell. A standard ¼-in. (6-mm) pipe with suitable valve is satisfactory.

**23.3 Tube—**A delivery tube which will not contaminate the product being sampled and long enough to reach to the bottom of the sample container is required to allow submerged filling.

**23.4 Procedure—**Before a sample is drawn, flush the tap (or gage glass drain cock) and line until they are purged completely. Connect the clean delivery tube to the tap. Draw upper, middle, or lower samples directly from the respective taps after the flushing operation. Stopper and label the sample container immediately after filling, and deliver it to the laboratory.

## 24. Continuous Sampling

**24.1** The continuous sampling procedure is applicable for sampling simple liquids in pipe lines, filling lines, and transfer lines. The continuous sampling may be done manually or by using automatic devices.

**24.1.1 Precaution—**The sample line should be purged three times before the sample is taken and special precautions should be taken to minimize exposure to the chemical being sampled.

**24.2 Sampling Probe—**The function of the sampling probe is to withdraw from the flow stream portion that will be representative of the entire stream. The apparatus assembly for continuous sampling is shown in Fig. 5. Probe designs that are commonly used are as follows:

**24.2.1** A tube extending to the center of the line and beveled at a 45° angle facing upstream.

**24.2.2** A long-radius elbow or bend extending to the center line of the pipe and facing upstream. The end of the probe should be reamed to give a sharp entrance edge.

**24.2.3** A tube extending across the pipeline