



# Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count<sup>1</sup>

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## INTRODUCTION

This test method may be used to determine the volume fraction of constituents in an opaque specimen using a polished, planar cross section by the manual point count procedure.

### 1. Scope

1.1 This test method describes a systematic manual point counting procedure for statistically estimating the volume fraction of an identifiable constituent or phase from sections through the microstructure by means of a point grid.

1.2 The use of automatic image analysis to determine the volume fraction of constituents is described in Practice E 1245.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*

E 3 Guide for Preparation of Metallographic Specimens<sup>2</sup>

E 7 Terminology Relating to Metallography<sup>2</sup>

E 407 Practice for Microetching Metals and Alloys<sup>2</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>3</sup>

E 1245 Practice for Determining the Inclusion or Second Phase Constituent Content of Metals by Automatic Image Analysis<sup>2</sup>

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, see Terminology E 7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *point count*—the total number of points in a test grid that fall within the microstructural feature of interest, or on the feature boundary; for the latter, each test point on the boundary is one half a point.

3.2.2 *point fraction*—the ratio, usually expressed as a percentage, of the point count of the phase or constituent of interest on the two-dimensional image of an opaque specimen to the number of grid points, which is averaged over  $n$  fields to produce an unbiased estimate of the volume fraction of the phase or constituent.

3.2.3 *stereology*—the methods developed to obtain information about the three-dimensional characteristics of microstructures based upon measurements made on two-dimensional sections through a solid material or their projection on a surface.

3.2.4 *test grid*—a transparent sheet or eyepiece reticle with a regular pattern of lines or crosses that is superimposed over the microstructural image for counting microstructural features of interest.

3.2.5 *volume fraction*—the total volume of a phase or constituent per unit volume of specimen, generally expressed as a percentage.

3.3 *Symbols:*

$P_T$  = total number of points in the test grid.

$P_i$  = point count on the  $i^{\text{th}}$  field.

$P_p(i)$  =  $\frac{P_i}{P_T} \times 100$  = percentage of grid points, in the constituent observed on the  $i^{\text{th}}$  field.

$n$  = number of fields counted.

$\bar{P}_p$  =  $\frac{1}{n} \sum_{i=1}^n P_p(i)$  = arithmetic average of  $P_p(i)$ .

$s$  = estimate of the standard deviation ( $\sigma$ ) (see (Eq 3) in Section 10).

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

- 95 % CI = 95 % confidence interval  
 =  $\pm ts / \sqrt{n}$  (see Note 1).
- $t$  = a multiplier related to the number of fields examined and used in conjunction with the standard deviation of the measurements to determine the 95% CI.
- $V_V$  = volume fraction of the constituent or phase expressed as a percentage (see (Eq 5) in Section 10).
- % RA = % relative accuracy, a measure of the statistical precision =  $(95 \% CI / \bar{P}_p) \times 100$ .

NOTE 1— Table 1 gives the appropriate multiplying factors ( $t$ ) for any number of fields measured.

#### 4. Summary of Test Method

4.1 A clear plastic test grid or eyepiece reticle with a regular array of test points is superimposed over the image, or a projection of the image, produced by a light microscope, scanning electron microscope, or micrograph, and the number of test points falling within the phase or constituent of interest are counted and divided by the total number of grid points yielding a point fraction, usually expressed as a percentage, for that field. The average point fraction for  $n$  measured fields gives an estimate of the volume fraction of the constituent. This method is applicable only to bulk opaque planar sections viewed with reflected light or electrons.

#### 5. Significance and Use

5.1 This test method is based upon the stereological principle that a grid with a number of regularly arrayed points, when systematically placed over an image of a two-dimensional section through the microstructure, can provide, after a representative number of placements on different fields, an unbiased statistical estimation of the volume fraction of an identifiable constituent or phase (1, 2, 3).<sup>4</sup>

5.2 This test method has been described (4) as being superior to other manual methods with regard to effort, bias, and simplicity.

5.3 Any number of clearly distinguishable constituents or phases within a microstructure (or macrostructure) can be counted using the method. Thus, the method can be applied to any type of solid material from which adequate two-dimensional sections can be prepared and observed.

5.4 A condensed step-by-step guide for using the method is given in Annex A1.

#### 6. Apparatus

6.1 *Test Grid*, consisting of a specified number of equally spaced points formed by the intersection of very thin lines. Two common types of grids (circular or square array) are shown in Fig. 1.

6.1.1 The test grid can be in the form of a transparent sheet that is superimposed upon the viewing screen for the measurement.

**TABLE 1 95 % Confidence Interval Multipliers**

No. of Fields $n$	$t$	No. of Fields $n$	$t$
5	2.776	19	2.101
6	2.571	20	2.093
7	2.447	21	2.086
8	2.365	22	2.080
9	2.306	23	2.074
10	2.262	24	2.069
11	2.228	25	2.064
12	2.201	26	2.060
13	2.179	27	2.056
14	2.160	28	2.052
15	2.145	29	2.048
16	2.131	30	2.045
17	2.120	40	2.020
18	2.110	60	2.000
		$\infty$	1.960

6.1.2 *Eyepiece Reticle*, may be used to superimpose a test grid upon the image.

6.2 *Light Microscope*, or other suitable device with a viewing screen at least 100 mm  $\times$  125 mm, preferably with graduated  $x$  and  $y$  stage translation controls, should be used to image the microstructure.

6.3 *Scanning Electron Microscope*, may also be used to image the microstructure; however, relief due to polishing or heavy etching must be minimized or bias will be introduced as a result of deviation from a true two-dimensional section through the microstructure.

6.4 *Micrographs*, of properly prepared opaque specimens, taken with any suitable imaging device, may be used provided the fields are selected without bias and in sufficient quantity to properly sample the microstructure.

6.4.1 The applicable point counting grid shall only be applied once to each micrograph. Point counting measurements should be completed on different fields of view and, therefore, different micrographs. Repeated point count measurements on an individual micrograph is not allowed.

6.4.2 The magnification of the micrograph should be as high as needed to adequately resolve the microstructure without resulting in adjacent grid points overlaying a single constituent feature.

#### 7. Sample Selection

7.1 Samples selected for measurement of the phase or constituent should be representative of the general microstructure, or of the microstructure at a specified location within a lot, heat, or part.

7.2 A description of the sample locations should be included as a part of the results.

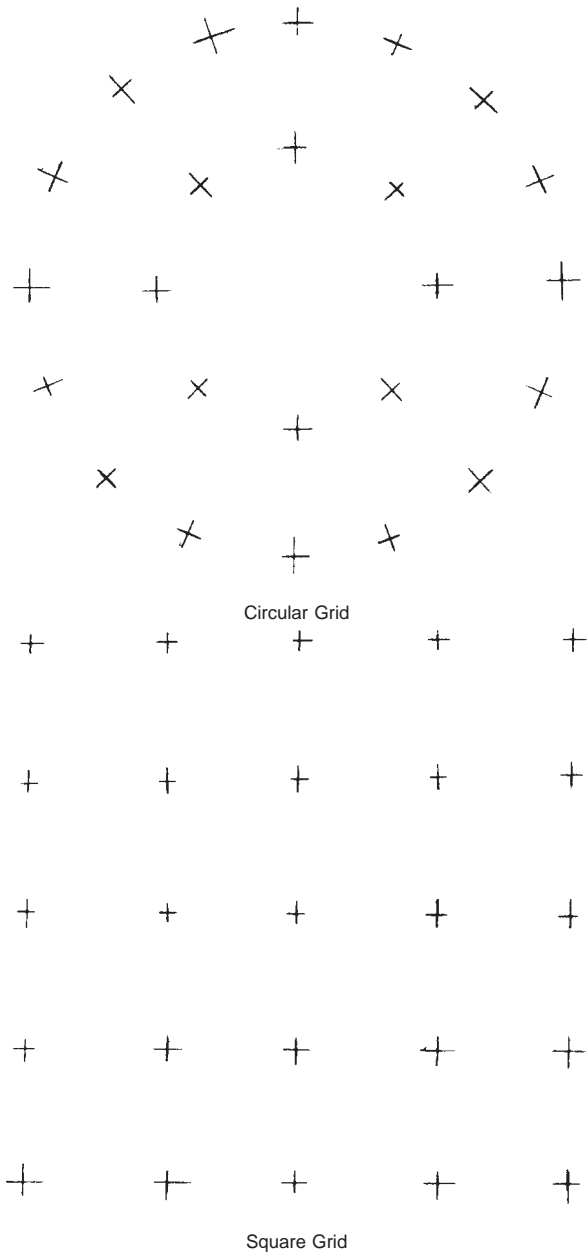
7.3 Any orientation of the prepared section (that is, whether longitudinal or transverse) can be used. However, it should be recorded since it may have an effect upon the precision obtained.

7.4 If the sample microstructure contains gradients or inhomogeneities (for example, banding) then the section should contain or show the gradient or inhomogeneity.

#### 8. Sample Preparation

8.1 The two-dimensional sections should be prepared using standard metallographic, ceramographic, or other polishing procedures, such as described in Methods E 3.

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.



NOTE 1—The entire 24 points can be used, or the outer 16, or the inner 8 points.

FIG. 1 Examples of Possible Grid Configurations That Can Be Utilized

8.2 Smearing or other distortions of the phases or constituents during preparation of the section or sections should be minimized because they tend to introduce an unknown bias into the statistical volume fraction estimate.

8.3 Etching of the sections, as described in Test Methods E 407, should be as shallow (that is, light) as possible because deviations from a planar two-dimensional section will cause a bias toward over estimation of the volume fraction.

8.4 Stain- or coloring-type etchants are preferable to those that cause attack of one or more of the constituents or phases.

8.5 Description of the etchant and etching procedure should be included in the report.

TABLE 2 Guidelines for Grid Size Selection<sup>A</sup>

NOTE 1—A grid size selection which gives a significant number of fields having no grid points on the constituent of interest should be avoided.

Visual Area Fraction Estimate Expressed as a Percentage	Grid Size (Number of Points, $P_T$ )
2 to 5 %	100
5 to 10 %	49
10 to 20 %	25
>20 %	16

<sup>A</sup> These guidelines represent an optimum for efficiency for the time spent counting and for the statistical information obtained per grid placement.

8.6 If etching is used to provide contrast or distinguishability of constituents then the volume fraction estimates should be obtained as a function of etching time to check the significance of any bias introduced.

9. Procedure

9.1 Principle:

9.1.1 An array of points formed by a grid of lines or curves is superimposed upon a magnified image (that is, a field of view) of a metallographic specimen.

9.1.2 The number of points falling within the microstructural constituent of interest is counted and averaged for a selected number of fields.

9.1.3 This average number of points expressed as a percentage of the total number of points in the array ( $P_T$ ) is an unbiased statistical estimation of the volume percent of the microstructural constituent of interest.

9.1.4 A condensed step-by-step description of the procedure is provided in Annex A1.

9.2 Grid Selection:

9.2.1 The grid should consist of equally spaced points formed by the intersection of fine lines. Diagrams of two possible grids, one with a circular pattern and one with a square pattern, which are recommended for use, are shown in Fig. 1.

9.2.2 Determine the number of points (that is, the grid size,  $P_T$ ) from a visual estimate of the area fraction occupied by the constituent of interest. Table 2 provides guidelines for this selection. The values in Table 2 do not correspond to theoretical constraints; but, by using these values, empirical observations have shown that the method is optimized for a given precision.

9.2.2.1 The user may choose to employ a 100 point grid over the entire range of volume fractions. The use of 100-point grid facilitates easy volume percent calculations. the use of only one overlay or eyepiece reticle for all volume percent determinations may save both time and money.

9.2.2.2 For constituents present in amount of less than 2%, a 400-point grid may be used.

9.2.3 Superimpose the grid, in the form of a transparency, upon a ground glass screen on which the section image is projected.

9.2.4 A grid in the form of an eyepiece reticle may also be used.

9.2.5 If the constituent areas form a regular or periodic pattern on the section image, avoid the use of a grid having a similar pattern.

**TABLE 3 Prediction of the Number of Fields ( $n$ ) to be Observed as a Function of the Desired Relative Accuracy and of the Estimated Magnitude of the Volume Fraction of the Constituent**

Amount of volume fraction, $V_v$ in percent	33 % Relative Accuracy				20 % Relative Accuracy				10 % Relative Accuracy			
	Number of fields $n$ for a grid of $P_T =$				Number of fields $n$ for a grid of $P_T =$				Number of fields $n$ for a grid of $P_T =$			
	16 points	25 points	49 points	100 points	16 points	25 points	49 points	100 points	16 points	25 points	49 points	100 points
2	110	75	35	20	310	200	105	50	1,250	800	410	200
5	50	30	15	8	125	80	40	20	500	320	165	80
10	25	15	10	4	65	40	20	10	250	160	85	40
20	15	10	5	4	30	20	10	5	125	80	40	20

NOTE 1—The given values in the table above are based on the formula:

$$n \approx \frac{4}{E^2} \cdot \frac{100 - V_v}{V_v}$$

where:

$E = 0.01 \times \% \text{ RA}$ , and

$V_v =$  is expressed in %.

### 9.3 Magnification Selection:

9.3.1 Select the magnification so that it is as high as needed to clearly resolve the microstructure without causing adjacent grid points to fall over the same constituent feature.

9.3.2 As a guideline, choose a magnification that gives an average constituent size that is approximately one half of the grid spacing.

9.3.3 As the magnification is increased, the field area decreases, and the field-to-field variability increases, thus requiring a greater number of fields to obtain the same degree of measurement precision.

### 9.4 Counting:

9.4.1 Count and record for each field the number of points falling on the constituent of interest.

9.4.2 Count any points falling on the constituent boundary as one half.

9.4.3 In order to minimize bias, any point that is doubtful as to whether it is inside or outside of the constituent boundary should be counted as one half.

9.4.4

$$P_{P(i)} = \frac{P_i \times 100}{P_T} \quad (1)$$

9.4.5 The values of  $P_{P(i)}$  are used to calculate  $\bar{P}_p$  and standard deviation,  $s$ .

### 9.5 Selection of the Number of Fields:

9.5.1 The number of fields or images to measure depends on the desired degree of precision for the measurement. Table 3 gives a guide to the number of fields or images to be counted as a function of  $P_T$ , the selected relative accuracy (statistical precision), and the magnitude of the volume fraction.

### 9.6 Selection of the Array of Fields:

9.6.1 Use a uniformly spaced array of fields to obtain the estimated value,  $P_p$ , and the estimated standard deviation,  $s$ .

9.6.2 If gradients or inhomogeneities are present, then a uniform spacing of fields may introduce a bias into the estimate. If another method of field selection is used, for example, random, then describe it in the report.

9.6.3 When the microstructure shows a certain periodicity of distribution of the constituent or phase being measured, any coincidence of the points of the grid and the structure must be

avoided. This can be achieved by using either a circular grid or a square grid placed at an angle to the microstructural periodicity.

9.7 *Grid Positioning Over Fields*—Make grid positioning of each field without viewing the microstructure to eliminate any possibility of operator bias. This can be accomplished by moving the  $x$  and  $y$  stage mechanism a fixed amount while shifting to the next field without looking at the microstructure.

9.8 *Improving Measurement Precision*—It is recommended that the user attempt to sample more of the microstructure either by multiple specimens or by completely repeating the metallographic preparation on the same sample when the precision for a single set of data is not acceptable (see Section 11).

## 10. Calculation of the Volume Percentage Estimate and % Relative Accuracy

10.1 The average percentage of grid points on the features of interest provides an unbiased statistical estimator for the volume percentage within the three dimensional microstructure. The value of the multiplier,  $t$ , can be found in Table 1. Thus, the average,  $\bar{P}_p$ , the standard deviation estimator,  $s$ , and the 95 % confidence interval, 95 % CI, should be calculated and recorded for each set of fields. The equations for calculating these values are as follows:

$$\bar{P}_p = \frac{1}{n} \sum_{i=1}^n P_p(i) \quad (2)$$

$$s = \left[ \frac{1}{n-1} \sum_{i=1}^n [P_p(i) - \bar{P}_p]^2 \right]^{1/2} \quad (3)$$

$$95 \% \text{ CI} = t \times \frac{s}{\sqrt{n}} \quad (4)$$

10.2 The volume percentage estimate is given as:

$$V_v = \bar{P}_p \pm 95 \% \text{ CI} \quad (5)$$

10.3 An estimate of the % relative accuracy associated with the estimate can be obtained as:

$$\% \text{ RA} = \frac{95 \% \text{ CI}}{\bar{P}_p} \times 100 \quad (6)$$



10.3.1 Estimates for the number of fields required to obtain a % relative accuracy of 10, 20, or 33 % with different volume percentages and grid sizes are provided in Table 3. These values were calculated under the assumption that the features have a random distribution upon the metallographic section.

10.4 The % relative accuracy reported should **always** be calculated from the sample data and should not be taken from Table 3.

## 11. Improving the Volume Fraction Estimate

11.1 If additional fields are measured to reduce the % relative accuracy, then the following rule gives an excellent guideline: To reduce the % RA by 50 %, then a total of four times the original number of fields should be measured.

11.2 When additional fields are selected on the same section, they should not overlap the initial set but may fit between fields of the initial set, and should also form a systematic sampling array.

11.3 As an example, if a 6 by 5 array of fields was used to obtain the initial set, then by halving the spacing and measuring the intermediate field positions, a total of four times the number of fields can be measured. Hence, 120 total fields would be measured by halving the spacing (in both  $x$  and  $y$  directions) and measuring the intermediate positions to form a 12 by 10 array. This additional effort should reduce the confidence interval, and thus the % RA, by approximately 50 %.

11.4 Where additional fields are measured on the same section, the average,  $\bar{P}_p$ , the standard deviation estimate,  $s$ , the 95 % confidence interval, 95 % CI, and the % relative accuracy, % RA, should be calculated using the increased total number of fields as a single data set.

11.5 If additional sections are prepared from the same sample by completely repeating the sample preparation, or if additional samples are prepared, then the same procedure should be used for each section, and the data recorded and reported separately. A grand average can be calculated by taking the average of the set means in this case. If no sample heterogeneity is indicated (that is, the confidence intervals about the mean of each set overlap), then the 95 % CI can be calculated from the standard deviation obtained using the data from all of the sets (that is, pooling the data and calculating a mean, standard deviation, and 95 % CI).

11.6 Where the 95 % CI do not overlap for the different sets, then a statistically significant difference between samples or sections may be present. In this case, more rigorous statistical significance tests should be considered.

## 12. Report

12.1 Report the following information:

12.1.1 Raw data,

12.1.2 Estimated volume % ( $\bar{P}_p$ )  $\pm$  95 % CI,

12.1.3 % relative accuracy (calculated value, not one estimated from Table 2),

12.1.4 Number of fields per metallographic section,

12.1.5 Number of sections,

12.1.6 Sample description and preparation, including etchant, if used,

12.1.7 Section orientation,

12.1.8 Magnification,

12.1.9 Grid description,

12.1.10 Field array description and spacing, and

12.1.11 List of volume % estimates for each metallographic section  $\pm$  95 % CI.

## 13. Effort Required

13.1 A reasonable estimate for the time required to perform the manual point count on 30 fields for a single type of microstructural feature is 30 min. This time estimate can probably be decreased to 15 min after some experience and familiarity with the point counting procedure and the microstructure analyzed are obtained.

## 14. Precision and Bias <sup>5</sup>

14.1 The systematic point count technique is the most efficient manual technique for development of an unbiased estimate of the volume fraction of an identifiable constituent or phase.

14.2 The presence of periodicity, structural gradients or inhomogeneities in the section can influence the precision and accuracy of the volume fraction estimate. Guidelines are given in 7.4, 9.2.5, 9.6.2, 9.6.3, 11.5 and 11.6.

14.3 The quality of the sample preparation can influence precision and accuracy of the volume fraction estimate. Guidelines are given in Section 8.

14.4 The point density of the grid used to make the volume fraction estimate can influence the efficiency, precision and relative accuracy of the volume fraction estimate. Guidelines are given in 9.2.

14.5 The magnification employed in the point count can influence precision and relative accuracy. Guidelines are given in 9.3.

14.6 The counting of grid points at a constituent boundary, particularly when doubt exists as to their exact location, presents an opportunity for bias in the estimate of the volume fraction. Guidelines are given in 9.4.2, and 9.4.3.

14.7 The number of fields measured, the method of field selection and their spacing will influence the precision and relative accuracy of the volume fraction estimate. Guidelines are given in 9.5, and 9.6.

14.8 The precision of a given measurement of the volume fraction is determined by calculation of the standard deviation, 95 % confidence interval, and % relative accuracy as described in Section 10.

14.9 If a greater degree of precision and relative accuracy is required, follow the guidelines in Section 11.

14.10 Results from a round-robin interlaboratory program (5), where three micrographs with different constituent volume fractions were point counted using two different grids (25 and 100 points) by 33 different operators, were analyzed<sup>5</sup> in accordance with Practice E 691 to develop repeatability and reproducibility standard deviations and 95 % confidence limits (see Table 4). For the same number of random grid placements (10) on each micrograph, the repeatability and reproducibility standard deviations and 95 % confidence intervals increased

<sup>5</sup> Support data are available from ASTM Headquarters. Request RR:E04-1003.

**TABLE 4 Results of Interlaboratory Point Counting Round-Robin<sup>5</sup>**

Micrograph	$\bar{P}_p$ (%)	Repeatability Std. Dev. (%)	Reproducibility Std. Dev. (%)	Repeatability 95 % CI (%)	Reproducibility 95 % CI (%)	Repeatability % RA	Reproducibility % RA
25 Point Test Grid							
A	9.9	5.3	5.3	14.8	14.8	149.5	149.5
B	17.8	6.6	6.9	18.6	19.4	104.5	109.0
C	27.0	8.8	9.4	24.7	26.2	91.5	97.0
100 Point Test Grid							
A	9.3	3.9	3.9	11.0	11.0	118.3	118.3
B	15.9	3.4	4.0	9.4	11.2	59.1	70.4
C	25.1	3.9	4.3	10.9	12.1	43.4	48.2

with increasing  $\bar{P}_p$  for measurements with the 25 point test grid but were essentially constant for the 100 point test grid. Note that the interlaboratory % relative accuracies (which are much poorer than those for the individual operators) improve as  $\bar{P}_p$

increases and as the grid point density ( $P_T$ ) increases. The 100 point grid, with four times the number of grid points, decreased the relative accuracies by about 21 to 51 % as  $\bar{P}_p$  increased (Micrographs A to C).

## ANNEX

### (Mandatory Information)

#### A1. PROCEDURE FOR SYSTEMATIC MANUAL POINT COUNT

A1.1 Visually estimate area percent of constituent or feature of interest on metallographic section.

A1.2 Using Table 3, select grid size,  $P_T$ .

A1.3 Superimpose the grid upon the microscope viewing screen and select magnification such that the size of the features of interest are approximately one half of the spacing between grid points.

A1.4 Select a statistical precision, (% RA) for example, 10, 20, or 33 %, desired for the measurement. Note that the % RA is defined as follows:

$$\% \text{ RA} = \frac{95 \% \text{ CI}}{\bar{P}_p} \times 100$$

A1.5 Using Table 3, obtain an estimate of the number of fields,  $n$ , required to obtain the desired degree of precision.

NOTE A1.1—A minimum of 30 fields must be measured in order to calculate the 95 % confidence interval using the equation given in A1.12.

A1.6 Determine the spacing between fields that will form a systematic (equally spaced) array covering a majority of the sample area without overlap.

A1.6.1 For example, on a 10 mm × 15 mm specimen area where 40 fields are indicated from Table 3, a 5 by 8 array of fields at 1.5 mm intervals might be used.

A1.7 Determine the number of turns required on the stage translation knobs to move the stage from one field position to the next. Do not observe the image while translating to a new field to avoid bias in positioning the grid.

A1.8 Count and record the number of grid points,  $P_i$ , falling within the features of interest.

NOTE A1.2—Any point that falls on the boundary should be counted as one half. To avoid bias, questionable points should be counted as one half.

A1.9 Calculate the average % of points per field,  $\bar{P}_p$ , and its standard deviation,  $s$ .

NOTE A1.3—A hand calculator with a  $\Sigma$  + key can be used to calculate these quantities.

A1.10 The average percentage of points is:

$$\bar{P}_p = \frac{1}{n} \sum_{i=1}^n P_p(i) = \frac{1}{n} \sum_{i=1}^n P_i/P_T$$

A1.11 The standard deviation estimate is:

$$s = \left[ \frac{1}{n-1} \sum_{i=1}^n [P_p(i) - \bar{P}_p]^2 \right]^{1/2}$$

A1.12 The 95 % confidence interval for  $\bar{P}_p$  is:

$$95 \% \text{ CI} = \frac{ts}{\sqrt{n}}$$

## REFERENCES

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