

Guidelines for sampling wines and musts intended for analysis

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GUIDELINES FOR SAMPLING WINES AND MUSTS PRIOR TO ANALYSIS

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1 Scope

These guidelines provide a methodology for the rational implementation and control of sampling procedures for wines and musts prior to organoleptic, physical, microbiological, or chemical analysis. Sampling may be performed at any time in the life of the wine from production to consumption as a prerequisite to the analyses performed to ensure compliance with technical, regulatory, commercial or food safety requirements. The steps in the sampling process are:

- precise identification of the object(s) to be sampled;
- definition of the needs generated by the envisaged inspection, as a function of the specified requirements (sampling quantity, precautions to take to avoid modifying the element(s) under investigation);
- selection of a sampling plan, possibly involving the use of statistical tools;
- selection of the sampling technique to be applied and consideration of the associated critical points.

These guidelines apply without prejudice to the applicable regulations in the fields in question.

2 Normative references

CAC/GL 50-2004: General guidelines on sampling (Codex Alimentarius)

ISO 2859-1: 1999: Sampling procedures for inspection by attributes

ISO 3951: 2005: Sampling procedures for inspection by variables

ISO 3170: 2004: Petroleum liquids – Manual sampling

ISO 7002: 1986: Agricultural food products – Layout for a standard method of sampling from a lot

3 Definition of the main terms used in these guidelines

Characteristic

A property which helps to identify, or differentiate between, items within a given lot. The differentiation may be quantitative (based on measured values) or qualitative (based on attributes).

Conformity and nonconformity

Satisfaction of the specified requirements, and the failure to satisfy the specified requirements respectively.

Inspection

Actions such as measuring, examining, testing or straining performed to investigate one or more characteristics of a product and to allow the results obtained to be compared with the specific requirements in order to determine the compliance status of each characteristic.

Inspection by attributes

A form of inspection whereby the item is classified simply as conforming or nonconforming (pass/fail) compared with one or more specified requirements.

Inspection by variables for percent nonconforming

A sampling plan based on inspection by variables for percent nonconforming provides a method for estimating the quality of a lot and involves measuring, in each sampled item, the value of a variable that characterises the inspected commodity.

Inspection of an average value

An inspection that investigates whether the mean content of the inspected characteristic of a lot is at least equal to a specified content (e.g.: regulation, labelling, etc.).

Ullage

The volume left vacant above the surface of the liquid in a fixed-volume sample container.

Sampled

A set composed of one or more items sampled from a lot and intended to provide information about this lot.

Representative sample

A sample is representative if its physical and chemical characteristics are identical to the average characteristics of the total volume of product from which the sample was taken.

Spot sample

A sample taken from a specified location in a container or from a liquid flowing through a pipe at a specified time.

Composite sample

A sample produced by mixing several spot samples in defined proportions to create a representative sample of the entire product.

Low sample

A spot sample taken from a point about $5/6^{\text{th}}$ of the way down through the depth of the liquid from the surface.

Bottom sample

A spot sample of the product taken from or near to the bottom of the container.

Surface sample

A spot sample taken from the surface of the liquid.

Middle or average sample

A spot sample taken halfway down through the depth of the liquid.

High sample

A spot sample taken from a point about $1/6^{\text{th}}$ of the way down through the depth of the liquid from the surface.

All-level sample

A sample taken using equipment which fills in one direction only when travelling vertically through the entire volume of liquid.

Zone sample

A sample corresponding to a column of liquid trapped inside the sampling device used at the instant when it is closed at a specific location in a container, after immersing this device down through the liquid during its positioning.

Tap or valve sample

A spot sample taken via a tap located on the side of the container.

Dynamic sampling

A method in which the liquid is sampled while passing through a pipe or tube.

Single random sampling

This method produces a sample using a technique which ensures that every sample that could be obtained has the same probability of being selected.

Static sampling

A method in which samples are taken from a liquid while it is stored in a given container.

Stratified sampling

Stratified sampling involves taking a single random sample from each defined stratum in a lot.

Lot size

Number of individual items in a lot.

Sample size

Number of individual items that make up a sample.

Lot homogeneity

A lot is homogeneous relative to a given characteristic if the characteristic is uniformly distributed throughout the lot in accordance with a given probability law (uniform law, normal law, etc.).

NOTE: A lot being homogeneous for a given characteristic does not mean that the value of the characteristic is the same throughout the lot (other than for a distribution that respects the uniform law).

Lot heterogeneity

A lot of must or wine is heterogeneous relative to a given characteristic if the characteristic is not uniformly distributed.

Individual item

That which may be described and considered individually (e.g.: a tank, a bottle, a barrel, etc.).

Integrity of the sample

The status of a complete and unimpaired sample, i.e. whose characteristics are the same as at the time when it was sampled.

Lot

A defined quantity of a specified product manufactured or produced under conditions which are presumed to be uniform.

Consignment

A quantity of some commodity delivered at one time. It may consist in either a portion of a lot, or a set of several lots, however, in the case of a statistical inspection, the consignment shall be considered as a new lot for the interpretation of the results.

- If the consignment is a portion of a lot, each portion is considered as a lot for the inspection;
- If the consignment is a set of several lots, before any inspection, care shall be given to the homogeneity of the consignment. If not homogeneous, a stratified sampling may be used.

Handling the sample

Any operation that involves packaging, transferring, splitting or transporting the sample.

Inspection level

There are three inspection levels of varying discrimination: normal, reduced and tightened. The inspection level should be selected as a function of the acceptable sampling error and to accommodate technical and economic contingencies.

Acceptable quality level (AQL)

The acceptable quality level (AQL) is an indexing criterion applied to a continuous series of lots which corresponds to a maximum rate of acceptable defective items in lots (or the maximum number of defective items per hundred items).

The users of the sampling plan must agree on the AQL for the plan applied to the quality control of the lots at the earliest possible stage.

Sampling plan

A planned procedure which lets the user select or draw separate samples from a lot, in order to obtain the information needed, such as a decision on the compliance status of the lot.

More precisely, a sampling plan defines the number of items that constitutes the sample and the number of nonconforming items required to allow evaluation of the compliance status of the lot.

Direct and indirect sampling

Direct sampling is performed in the final container with no decanting involved. Indirect sampling is when the liquid is transferred or decanted from the lot to the final container for the sample, via sampling equipment.

Sampling procedure

A definition of the means to be implemented to perform a sampling that satisfies the requirements specified as a function of the nature of the objects from which the samples are to be taken.

Sampled item

A quantity of matter sampled in one go from a greater quantity of matter.

Quality

The ability of a set of intrinsic characteristics to satisfy the requirements.

Sample container

The container that holds a sample.

Sample-taking container

The container used to collect a sample.

Stratum

When a consignment or lot is considered as non-homogeneous, it is divided into homogeneous sub-groups of known size called "strata" whose characteristics differ from the other sub-groups.

Uniformity

A lot of must or wine is uniform relative to a characteristic if the distribution of this characteristic is the same throughout the lot.

4 Symbols and abbreviations

α : significance level

Ac: acceptance criterion

AQL: Acceptable quality level (as a percentage of defective items or as a number of defects per hundred items)

Re: rejection criterion

i : sample number

k : acceptance constant

L : lower limit of a characteristic

n : sample size

t_α : the value of the Student's t distribution with $n-1$ degrees of freedom and a significance level of α

U : upper limit of a characteristic

U_α : standardised normal distribution corresponding to the significance level α

Z_U : Z-score corresponding to an upper limit

Z_L : Z-score corresponding to a lower limit

σ : standard deviation

s : estimator of the standard deviation

x_i : the value of a characteristic determined from a sampled item

\bar{x} : the mean of the values measured in a sample

5 Implementing a sampling procedure

Sampling is a process implemented to collect information about a given consignment. Since it is most often impossible to measure the characteristic under consideration in all the individual items or for the entire volume of a consignment, a sample must be collected consisting of a restricted number of items, or a smaller volume.

Because there is a cost associated with inspection or measurement activities, clearly the size of the sample should be limited and optimised.

Since it consists in only a restricted number of individual items or a small volume taken from the consignment, the sample can only ever provide incomplete information about the consignment

under consideration. However, the operator's objective is to create a representative sample, i.e. one that differs as little as possible from the average characteristics of the consignment.

The sampling procedure is a methodological approach, detailed below in 4 steps that the operator can follow for all sampling situations, including those that appear to be the most basic.

5.1 Identifying the consignment and specifying the lots to be sampled

The total quantity of product or the total number of individual items from which the sample is taken is conventionally called the consignment. The consignment may consist of one or more lots.

The first step in the sampling procedure is thus to identify and demarcate the consignment, and then to identify and demarcate the consignment's component lots.

The following information in particular is collected at this stage:

- Nature (must, wine, etc.),
- Process status (e.g.: "fermenting", "ready for packaging", "packaged", etc.),
- Designation (Lot number, Appellation of Origin, Name of the Domain, Name of the producer, Name of the packager for packaged products, etc.),
- Nature, number and designation (if applicable) of the containers in which the lots are contained,
- Quantities present: consignment – lots.

5.2 Specifying the nature of the characteristic of the lot(s) to be investigated

The characteristic is a property which helps to identify, or differentiate between, items within a given lot. The differentiation may be quantitative (based on measurement) or qualitative (based on attributes).

Examples:

- Product defect (colour, labelling, foreign substances, organoleptic anomaly, etc.),
- Characteristics related to the composition. They may be distributed normally (e.g. alcoholic strength by volume) or abnormally,
- Properties relating to the state of health (microbiological degradation, sporadic chemical contamination, etc.).

The characteristic to be investigated must be specified before any sample is taken. The impact of the nature of the characteristic on the selection of the sampling plan and sampling techniques must be determined.

5.3 Studying the apparent or predicted distribution of the characteristic within the lot(s) considered

Before taking any samples the operator must, wherever possible, define how the characteristic under investigation is distributed within the consignment's component lot(s).

The lots considered shall be confirmed as being lots for sampling purposes once their homogeneity has been established for the characteristic studied. A lot is homogeneous for a given characteristic if its characteristic is distributed through the lot in accordance with a given probability distribution:

- Uniform distribution

- Normal distribution
- Rare events (Poisson's law)
- etc.

If the homogeneity cannot be established, the operator must divide the lot into sub-lots (such that the characteristic is distributed homogeneously within each sub-lot) and then perform a "stratified" sampling from these sub-lots.

As a last resort, if it is not possible to establish the distribution of the characteristic, samples must be collected randomly from the entire lot. Depending on the observations made, it may then be possible to identify which lots are homogeneous for the characteristic(s) investigated.

In all cases, statistical sampling can only be performed on a lot that is homogeneous for the characteristic under consideration.

5.4 Drawing up the sampling plan

The sampling plan is a planned procedure for selecting, drawing, and constituting samples from a lot in order to obtain the information needed about one or more investigated characteristics, such as to make a decision on the compliance status of this lot. The sampling plan defines the number of items that constitutes the sample and the decision rules used to evaluate the compliance status of the lot.

The sampling plan may be either:

- Random statistical, if it is established that the distribution of the characteristic is homogeneous within the lot;
- Stratified statistical, if it is established that the lot is heterogeneous, but can be divided into homogeneous sub-lots for the characteristic studied;
- Targeted, if it is established that the characteristic is heterogeneous. In this case the sampling plan concentrates on a portion of the lot. The nature of this type of sampling plan is pragmatic and empirical;
- Random, empirical, distributed over the entire lot, if the distribution type of the characteristic cannot be established with sufficient certainty.

6 Simplified statistical sampling plans for oenological applications

6.1 General principles

Sampling plans are statistical tools used to determine the size of a sample and to establish the acceptability or rejection conditions for a lot based on inspection and analysis of the sample. The objective of a sampling plan is therefore to allow a decision to be made regarding the compliance status of a lot, in a context of cost control and error management.

A necessary prerequisite to the sampling plan is thus the definition of precise conformity criteria, in addition to defining an acceptable quality level, corresponding to the maximum number of nonconforming items that the lot can contain yet still be considered as acceptable (see §6.2.1).

A sampling plan can be implemented once it is established that the characteristic investigated is distributed according to a given probability distribution (i.e. the lot is homogeneous), notable examples of which are:

- Uniform distribution: this is the simplest case wherein the characteristic is identical for all the items. When the distribution is uniform, the statistics for the sampling plan become very simple, since just one sample needs to be taken (e.g.: alcoholic strength by volume for a finished wine, contained in a small or medium-sized container).
- Normal distribution: the distribution of the characteristic is Gaussian, with a mean and a standard deviation.
- Poisson's law: used for rare events (e.g.: bottles that have a mouldy taint, etc).

If the characteristic's distribution is heterogeneous through the consignment (it does not match any probability distribution), it is then appropriate, if possible, to split up the consignment into sub-lots within which the characteristic can be considered to be distributed homogeneously. In this case, "stratified" sampling is performed, with as many single sampling plans implemented as there are sub-lots created by the stratification.

If the initial observations or measurements of individual items show that the items within a lot considered as homogeneous are not in fact distributed homogeneously, then the heterogeneous lot should be split into as many sub-lots as are necessary to ensure that each sub-lot is homogeneous.

Depending on the nature of the characteristic, different types of sampling plan must be defined:

- If the characteristic under consideration is qualitative, with the only possible results of measurements being: "conforming" and "nonconforming", the sampling plan is an **inspection by attribute**.
- If the characteristic under consideration varies continuously and measurably, and its distribution is normal, or can be treated as though it is, two types of inspection are possible:
 - Inspection **by variables for percent nonconforming**
 - Inspection of the **mean content**.

In these two cases, the characteristic shall be defined by measuring each individual item in the sample.

This type of "by variables" inspection only applies to the lots within which the characteristic is distributed normally, or can be treated as though it is.

"By attribute" plans offer a number of advantages:

- no conditions on the mathematical law describing the distribution of the characteristic;
- straightforward processing of the results obtained from the sample.

"By variables" plans offer other advantages:

- greater inspection efficacy compared with a by attributes plan performed under the same sampling conditions,
- less expensive than a by attributes plan since, for the same efficacy, the sample size is smaller.

The pros and cons of each type of inspection must be considered by the person responsible for the sampling when selecting the type of inspection.

6.2 Definitions common to the various sampling plans

The methodologies of the various sampling plans described in these guidelines share certain features, as described below.

6.2.1 Acceptable Quality Level (AQL)

The acceptable quality level (AQL) is a criterion which corresponds to the maximum percentage of defective items admissible in the lot. It is expressed as a percentage. The AQL must be defined before drawing up a sampling plan since this parameter is a prerequisite to its composition.

The process leading to the definition of the AQL can vary. Generally, it is based on:

- normative or regulatory data;
- agreements between the parties;
- standard professional practices;
- etc.

The AQL must be specified at the start of the sampling procedure.

6.2.2 Inspection levels

There are three inspection levels of varying discrimination: normal, reduced and tightened. The tighter the inspection level, the lower the sampling error. The inspection level should be selected as a function of the acceptable sampling error and to accommodate technical and economic contingencies. The inspection level initially selected may prove to be unsuitable, in which case it should be tightened or reduced.

By default, if no specific requirement is stated, it is standard practice to begin with a normal inspection level.

The sampling data corresponding to each inspection level varies, as indicated in the tables in the sections below.

6.2.3 Taking samples

Traditionally, the individual items collected to make up the sample must be taken from the lot by **single random sampling**. This sampling method ensures that each sample has the same probability of being selected.

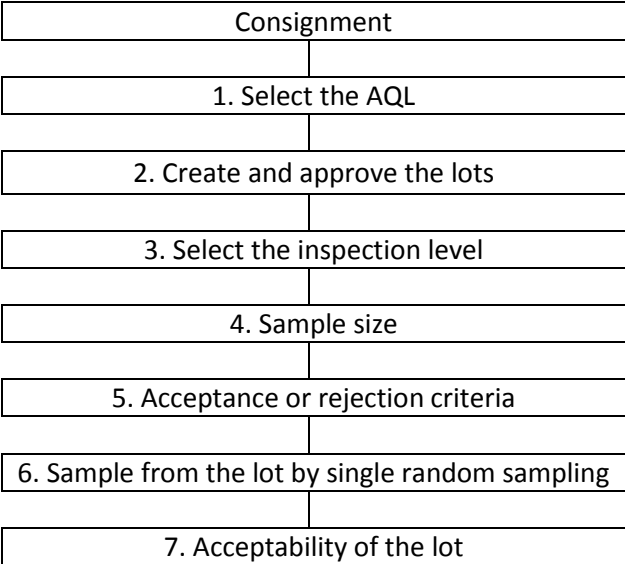
However, some information is often known in advance about the consignment studied. Consequently, it is possible to determine, in advance, whether it should be divided into sub-lots of known size: strata (or zones) which have different properties (different production batches, variation in the ingredients used, etc.). In this case, each stratum or zone should be inspected separately, so that the general mean can be determined based on the mean of the strata. If the strata are homogeneous and the means are different, this method is more accurate than single random sampling. **Stratified sampling** thus involves taking a single random sample from each stratum.

6.3 Inspection by attributes of percent nonconforming

A **sampling plan by attributes** provides a method for estimating the quality of a lot and involves determining the “conforming” or “nonconforming” status of the characteristic or attribute of each component item of the sample, depending on whether or not it meets the specification.

In this case this characteristic is qualitative (*e.g.: leaking bottles, analytical limit value exceeded*). The individual items that have the defective attribute are then counted. If the number of defective items is not greater than the acceptance criterion stated in the sampling plan then the lot is accepted. Otherwise, it is rejected.

The diagram below summarises the steps in the methodology for the implementation of a sampling procedure for a consignment up until the point when the results of the test by attributes are known. Note that this diagram only covers the sampling of homogeneous lots.



6.3.1 Selecting the Acceptable Quality Level (AQL)

The first step is to set the AQL.

6.3.2 Creating and approving the lots

Creating the lots

The consignment must be segmented into identifiable lots or sub-lots, or segmented in any other way that can be specified. Each lot must, wherever possible, be created from a single type and a single class, size and composition. For packaged products, the packaging must have been produced under the same conditions and essentially over the same period of time.

Approving the lots

The creation of the lots, their size, and how each lot must be represented and identified shall be specified or approved by the person responsible for the sampling.

6.3.3 Selecting the inspection level

The inspection level is specified at the start of the procedure.

6.3.4 Sample size

The determination of the sample size is a two-stage process based on the following tables.

6.3.4.1 Determining the code letter as a function of lot size

Table 1 indicates the code letter to be attributed as a function of lot size.

Lot size	Code letter
2 to 8	A
9 to 15	B
16 to 25	C
26 to 50	D
51 to 90	E
91 to 150	F
151 to 280	G
281 to 500	H
501 to 1,200	J
1,201 to 3,200	K
3,201 to 10,000	L
10,001 to 35,000	M
35,001 to 150,000	N
150,001 to 500,000	P
500,001 or more	Q

Table 1. Code letter as a function of sample size

Example: A lot containing 290 individual items would be attributed the code letter H.

6.3.4.2 Determining the sample size

The sample size is determined based on tables 2, 3 and 4 as a function of the selected inspection level:

- normal (table 2);
- reduced (table 3);
- tightened (table 4).

and based on the following parameters:

- AQL;
- Sample size code letter.

6.3.5 Acceptance and rejection criteria

The acceptance criterion (Ac) is the maximum number of defective sampled items admissible for lot acceptance. The rejection criterion (Re) is the minimum number of defective sampled items which results in the rejection of the lot. These values are given in tables 2, 3 and 4.

Table 2: Single sampling plan by attribute for a **normal** inspection level

Sample size code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot																	
		0.01	0.015	0.025	0.04	0.065	0.1	0.15	0.25	0.4	0.65	1	1.5	2.5	4	6.5	10	15	25
		Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re
A	2															0 1	0 1	1 2	1 2
B	3														0 1	0 1	1 2	1 2	2 3
C	5													0 1	0 1	1 2	1 2	2 3	3 4
D	8												0 1	0 1	1 2	1 2	2 3	3 4	5 6
E	13										0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	
F	20									0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	
G	32								0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	
H	50							0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22	
J	80						0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22		
K	125					0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22			
L	200					0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22			
M	315				0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22				
N	500			0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22					
P	800		0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22						
Q	1250	0 1	0 1	1 2	1 2	2 3	3 4	5 6	7 8	10 11	14 15	21 22							

Ac Acceptance criterion
 Re Rejection criterion

If the box is empty, take the figure from the nearest filled box in the same column

Example: For a lot of 2,000 items, the code letter is K, and 125 samples should be taken. For an AQL = 1%, the acceptance criterion is a maximum of 3 defective items and the rejection criterion is 4 or more defective items.

Table 3: Single sampling plan with inspection by attribute and a **tightened** inspection level

Sample size code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot																	
		0.01	0.015	0.025	0.04	0.065	0.1	0.15	0.25	0.4	0.65	1	1.5	2.5	4	6.5	10	15	25
		Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re
A	2																0 1	1 2	1 2
B	3															0 1	1 2	1 2	1 2
C	5														0 1	1 2	1 2	1 2	2 3
D	8													0 1	1 2	1 2	1 2	2 3	3 4
E	13												0 1	1 2	1 2	1 2	2 3	3 4	5 6
F	20											0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9
G	32										0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13
H	50								0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19	
J	80							0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19		
K	125						0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19			
L	200					0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19				
M	315					0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19				
N	500				0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19					
P	800			0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19						
Q	1250	0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	8 9	12 13	18 19							

Ac Acceptance criterion
 Re Rejection criterion

If the box is empty, take the figure from the nearest filled box in the same column

Example: For a lot of 2,000 items, the code letter is K, and 125 samples should be taken. For an AQL = 1%, the acceptance criterion is a maximum of 2 defective items and the rejection criterion is 3 or more defective items.

Table 4: Single sampling plan with inspection by attribute and a **reduced** inspection level

Sample size code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot																	
		0.01	0.015	0.025	0.04	0.065	0.1	0.15	0.25	0.4	0.65	1	1.5	2.5	4	6.5	10	15	25
		Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re	Ac Re
A	2															0 1	1 2	1 2	1 2
B	2														0 1	0 1	1 2	1 2	1 2
C	2													0 1	0 1	1 2	1 2	1 2	2 3
D	3												0 1	0 1	1 2	1 2	1 2	2 3	3 4
E	5											0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6
F	8									0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	
G	13								0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	
H	20							0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11	
J	32							0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11	
K	50						0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11		
L	80					0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11			
M	125				0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11				
N	200			0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11					
P	315		0 1	0 1	1 2	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11						
Q	500	0 1	0 1	0 1	1 2	1 2	2 3	3 4	5 6	6 7	8 9	10 11							

Ac Acceptance criterion
 Re Rejection criterion

If the box is empty, take the figure from the nearest filled box in the same column

Example: For a lot of 2,000 items, the code letter is K, and 50 samples should be taken. For an AQL = 1%, the acceptance criterion is a maximum of 2 defective items and the rejection criterion is 3 or more defective items.

6.3.6 Taking samples

The individual items selected for the sample must be taken from the lot by **single random sampling**.

6.3.7 Determining the acceptability of the lot

The defective (nonconforming) individual items must be counted. If the number of nonconforming individual items in the sample is less than or equal to the acceptance criterion then the lot must be considered as acceptable. Conversely, if the number of nonconforming individual items in the sample is greater than or equal to the rejection criterion, then the lot must be considered as unacceptable.

6.4 Inspection by variables for percent nonconforming

6.4.1 Methodology

The purpose of an inspection by variables of defective items is to check that the mean of the values of a characteristic of a lot satisfies the determined limits or intervals.

The method involves collecting a sample, measuring the characteristic under consideration in all the sampled items, and then comparing a Z-score with a coefficient k , known as the acceptance constant, which is a function of the AQL, the inspection level and the sample size.

The conditions under which this type of inspection are applied are as follows:

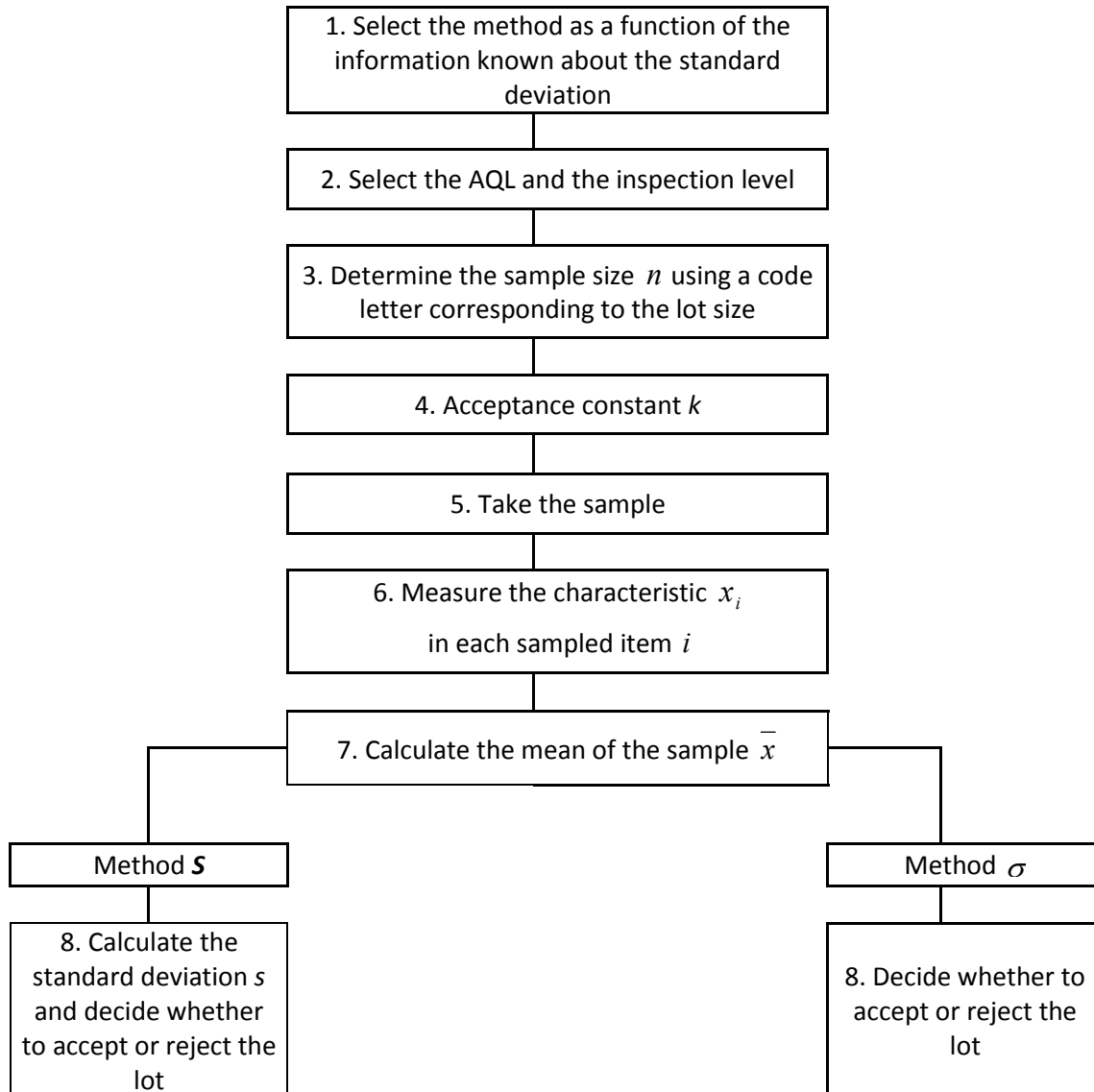
- The characteristic under consideration must be a continuous and measurable variable,
- The lot is homogeneous for the characteristic investigated, AND the distribution of the characteristic is normal, or can be treated as though it is, and shall be characterised by a mean and a standard deviation.

There are two types of situation, which the two inspection methods cover:

- either the standard deviation of the population is not known, in which case it is estimated.
- or the standard deviation σ is known (e.g. for the continuous production of packaged products, the production history provides information about the standard deviation for a new production batch).

For both these methods, specific sampling plans and sample sizes are implemented. However, the rules for accepting or rejecting the lots are similar for the two methods.

Procedure: The diagram below shows the steps in the methodology for the implementation of a sampling procedure for a given consignment, up until the point when the results of the test are known.



6.4.2 Type of method (s or σ) and choice of inspection level

If the standard deviation is not known, then “Method s ” is used. On the contrary, if the standard deviation is known, then “Method σ ” is used.

As for inspection by attributes, there are three inspection levels: normal, reduced and tightened. The inspection level is specified at the start of the procedure.

6.4.3 Selecting the AQL

The AQL should be set at the start of the procedure. The AQL is used to define the acceptance and rejection criteria for the lot.

6.4.4 Determining the sample size n

The first stage in determining the sample size is to find the code letter corresponding to the lot size, from table 5.

Lot size	Code letter
2 to 8	B
9 to 15	B
16 to 25	C
26 to 50	D
51 to 90	E
91 to 150	F
151 to 280	G
281 to 500	H
501 to 1,200	J
1,201 to 3,200	K
3,201 to 10,000	L
10,001 to 35,000	M
35,001 to 150,000	N
150,001 to 500,000	P
500,001 or more	Q

Table 5: Determination of the code letter as a function of lot size

The second step is to determine, as a function of the code letter obtained and as a function of the method used (s or σ), the corresponding sample size from table 6.

Code letter	Sample size Method s		Sample size Method σ	
	Normal or tightened inspection	Reduced inspection	Normal or tightened inspection	Reduced inspection
B	3	3	2	2
C	4	3	3	2
D	6	3	4	2
E	9	4	6	3
F	13	6	8	4
G	18	9	10	6
H	25	13	12	8
J	35	18	15	10
K	50	25	18	12
L	70	35	21	15
M	95	50	25	18
N	125	70	32	21
P	160	95	40	25
Q	200	125	50	32

Table 6: Determination of the sample size as a function of code letter

Note that method σ is less expensive to conduct than method s since fewer samples are collected.

6.4.5 Determining the acceptance constant k

Tables 7 to 9 (method \bar{s}) and 10 to 12 (method σ) are used to determine an acceptance constant k which is considered when determining the acceptability of the lot. The value of k depends on the AQL, the sample size n and the inspection level.

Table 7: Single sampling plan with **normal** inspection by variables for percent nonconforming – Determination of the acceptance constant *k* – Method **s**

Code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot															
		0.01 k	0.015 k	0.025 k	0.04 k	0.065 k	0.1 k	0.15 k	0.25 k	0.4 k	0.65 k	1 k	1.5 k	2.5 k	4 k	6.5 k	10 k
B	3														0.954	0.818	0.526
C	4													1.163	1.046	0.853	0.580
D	6												1.395	1.275	1.108	0.902	0.587
E	9											1.615	1.494	1.338	1.159	0.907	0.597
F	13										1.830	1.712	1.565	1.405	1.189	0.938	0.614
G	18									2.025	1.910	1.770	1.622	1.429	1.212	0.944	0.718
H	25								2.215	2.102	1.969	1.829	1.652	1.457	1.225	1.035	0.809
J	35							2.399	2.289	2.160	2.028	1.862	1.684	1.476	1.311	1.118	0.912
K	50						2.569	2.461	2.336	2.209	2.052	1.885	1.693	1.543	1.372	1.193	0.947
L	70					2.736	2.631	2.510	2.389	2.239	2.082	1.904	1.766	1.611	1.451	1.238	
M	95				2.889	2.787	2.670	2.553	2.410	2.261	2.093	1.965	1.822	1.676	1.484		
N	125			3.037	2.937	2.824	2.711	2.574	2.432	2.274	2.154	2.021	1.886	1.710			
P	160		3.179	3.082	2.973	2.865	2.733	2.597	2.447	2.334	2.209	2.083	1.921				
Q	200	3.310	3.215	3.109	3.004	2.877	2.747	2.603	2.495	2.377	2.258	2.106					
R	250	3.350	3.247	3.146	3.023	2.898	2.760	2.657	2.545	2.432	2.289						

If the box is empty, take the figure from the nearest filled box in the same column

Table 8: Single sampling plan with **tightened** inspection by variables for percent nonconforming – Determination of the acceptance constant k – Method **s**

Code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot															
		0.01	0.015	0.025	0.04	0.065	0.1	0.15	0.25	0.4	0.65	1	1.5	2.5	4	6.5	10
		k	k	k	k	k	k	k	k	k	k	k	k	k	k	k	k
B	3															0.954	0.818
C	4														1.163	1.046	0.853
D	6													1.395	1.275	1.108	0.902
E	9												1.615	1.494	1.338	1.159	0.907
F	13											1.830	1.712	1.565	1.405	1.189	0.938
G	18										2.025	1.910	1.770	1.622	1.429	1.212	0.944
H	25									2.215	2.102	1.969	1.829	1.652	1.457	1.225	0.995
J	35								2.399	2.289	2.160	2.028	1.862	1.684	1.476	1.242	1.010
K	50							2.569	2.461	2.336	2.209	2.052	1.885	1.693	1.481	1.277	1.044
L	70						2.736	2.631	2.510	2.389	2.239	2.082	1.904	1.710	1.526	1.322	
M	95					2.889	2.787	2.670	2.553	2.410	2.261	2.093	1.913	1.745	1.559		
N	125				3.037	2.937	2.824	2.711	2.574	2.432	2.274	2.105	1.949	1.779			
P	160			3.179	3.082	2.973	2.865	2.733	2.597	2.447	2.288	2.141	1.984				
Q	200		3.310	3.215	3.109	3.004	2.877	2.747	2.603	2.452	2.313	2.165					
R	250	3.442	3.350	3.247	3.146	3.023	2.898	2.760	2.616	2.485	2.345						

If the box is empty, take the figure from the nearest filled box in the same column

Table 9: Single sampling plan with **reduced** inspection by variables for percent nonconforming – Determination of the acceptance constant *k* – Method *s*

Code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot															
		0.01	0.015	0.025	0.04	0.065	0.1	0.15	0.25	0.4	0.65	1	1.5	2.5	4	6.5	10
		k	k	k	k	k	k	k	k	k	k	k	k	k	k	k	k
B-D	3												0.954	0.907	0.818	0.526	0.023
E	9											1.163	1.119	1.046	0.853	0.580	0.099
F	13										1.395	1.348	1.275	1.108	0.902	0.587	0.161
G	18								1.615	1.566	1.494	1.338	1.159	0.907	0.597	0.368	
H	25							1.830	1.782	1.712	1.565	1.405	1.189	0.938	0.763	0.461	
J	35						2.025	1.978	1.910	1.770	1.622	1.429	1.212	1.065	0.823	0.619	
K	50					2.215	2.168	2.102	1.969	1.829	1.652	1.457	1.329	1.123	0.995	0.809	
L	70					2.399	2.353	2.289	2.160	2.028	1.862	1.684	1.569	1.387	1.242	1.118	
M	95				2.569	2.524	2.461	2.336	2.209	2.052	1.885	1.778	1.612	1.481	1.372		
N	125			2.736	2.692	2.631	2.510	2.389	2.239	2.082	1.982	1.829	1.710	1.611			
P	160		2.889	2.846	2.787	2.670	2.553	2.410	2.261	2.167	2.023	1.913	1.822				
Q	200	0.037	2.995	1.937	2.824	2.711	2.574	2.432	2.344	2.208	2.105	2.021					
R	250	3.139	3.082	2.973	2.865	2.733	2.597	2.513	2.385	2.288	2.209						

If the box is empty, take the figure from the nearest filled box in the same column

Table 10: Single sampling plan with **normal** inspection by variables for percent nonconforming – Determination of the acceptance constant k – Method σ

Code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot																
		0.01 k	0.015 k	0.025 k	0.04 k	0.065 k	0.1 k	0.15 k	0.25 k	0.4 k	0.65 k	1 k	1.5 k	2.5 k	4 k	6.5 k	10 k	
B	2															0.620	0.478	0.273
C	3													0.991	0.841	0.643	0.412	
D	4												1.296	1.148	1.964	0.760	0.478	
E	6											1.578	1.432	1.256	1.068	0.818	0.528	
F	8										1.821	1.682	1.517	1.344	1.121	0.872	0.564	
G	10								2.030	1.897	1.742	1.581	1.378	1.157	0.893	0.675		
H	12							2.223	2.096	1.949	1.800	1.613	1.412	1.179	0.991	0.771		
J	15							2.410	2.289	2.150	2.009	1.835	1.650	1.439	1.273	1.082	0.879	
K	18						2.576	2.459	2.327	2.193	2.029	1.857	1.662	1.511	1.340	1.162	0.919	
L	21					2.738	2.627	2.500	2.374	2.218	2.057	1.876	1.737	1.582	1.422	1.210		
M	25				2.890	2.783	2.661	2.540	2.393	2.240	2.070	1.941	1.797	1.650	1.459			
N	32			3.041	2.937	2.820	2.704	2.563	2.419	2.258	2.136	2.001	1.866	1.690				
P	40		3.186	3.086	2.974	2.862	2.727	2.589	2.436	2.321	2.194	2.068	1.905					
Q	50	3.319	3.222	3.113	3.005	2.875	2.742	2.596	2.487	2.367	2.247	2.094						
R	65	3.359	3.254	3.150	3.025	2.897	2.758	2.653	2.539	2.426	2.281							

If the box is empty, take the figure from the nearest filled box in the same column

Table 11: Single sampling plan with **tightened** inspection by variables for percent nonconforming – Determination of the acceptance constant k – Method σ

Code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot																
		0.01	0.015	0.025	0.04	0.065	0.1	0.15	0.25	0.4	0.65	1	1.5	2.5	4	6.5	10	
		k	k	k	k	k	k	k	k	k	k	k	k	k	k	k	k	
B	2															0.620	0.478	
C	3															0.991	0.841	0.643
D	4													1.296	1.148	0.964	0.760	
E	6												1.578	1.432	1.256	1.068	0.818	
F	8											1.821	1.682	1.517	1.344	1.121	0.872	
G	10									2.030	1.897	1.742	1.581	1.378	1.157	0.893		
H	12								2.223	2.096	1.949	1.800	1.613	1.412	1.179	0.913		
J	15							2.410	2.289	2.150	2.009	1.835	1.650	1.439	1.204	0.976		
K	18						2.576	2.459	2.327	2.193	2.029	1.857	1.662	1.449	1.245	1.015		
L	21					2.738	2.627	2.500	2.374	2.218	2.057	1.876	1.681	1.497	1.293			
M	25				2.890	2.783	2.661	2.540	2.393	2.240	2.070	1.888	1.719	1.534				
N	32			3.041	2.937	2.820	2.704	2.563	2.419	2.258	2.087	1.929	1.758					
P	40		3.186	3.086	2.974	2.862	2.727	2.589	2.436	2.274	2.127	1.968						
Q	50	3.319	3.222	3.113	3.005	2.875	2.742	2.596	2.443	2.303	2.154							
R	65	3.454	3.359	3.254	3.150	3.025	2.897	2.758	2.611	2.478	2.337							

If the box is empty, take the figure from the nearest filled box in the same column

Table 12: Single sampling plan with **reduced** inspection by variables for percent nonconforming – Determination of the acceptance constant k – Method σ

Code letter	Sample size	AQL: percentage of nonconforming items accepted in a lot															
		0.01	0.015	0.025	0.04	0.065	0.1	0.15	0.25	0.4	0.65	1	1.5	2.5	4	6.5	10
		k	k	k	k	k	k	k	k	k	k	k	k	k	k	k	k
B-D	2												0.620	0.565	0.478	0.273	0.011
E	6											0.991	0.931	0.841	0.643	0.412	0.067
F	8										1.296	1.236	1.148	0.964	0.760	0.478	0.129
G	10								1.578	1.518	1.432	1.256	1.068	0.818	0.528	0.323	
H	12							1.821	1.764	1.682	1.517	1.344	1.121	0.872	0.705	0.422	
J	15						2.030	1.975	1.897	1.742	1.581	1.378	1.157	1.012	0.776	0.581	
K	18					2.223	2.170	2.096	1.949	1.800	1.613	1.412	1.283	1.078	0.913	0.771	
L	21				2.410	2.360	2.289	2.150	2.009	1.835	1.650	1.533	1.349	1.204	1.082		
M	25			2.576	2.527	2.459	2.327	2.193	2.029	1.857	1.748	1.580	1.449	1.340			
N	32		2.738	2.691	2.627	2.500	2.374	2.218	2.057	1.956	1.801	1.681	1.582				
P	40		2.890	2.845	2.783	2.661	2.540	2.393	2.240	2.145	1.999	1.888	1.797				
Q	50	3.041	2.998	2.937	2.820	2.704	2.563	2.419	2.328	2.191	2.087	2.001					
R	65	3.144	3.086	2.974	2.862	2.727	2.589	2.503	2.373	2.274	2.194						

If the box is empty, take the figure from the nearest filled box in the same column

6.4.6 Taking the sample

The items sampled must be taken from the lot by **single random sampling**.

6.4.7 Measuring the characteristic

For each sampled item i , the value x_i is measured of the characteristic under consideration.

6.4.8 Calculating the mean

Let \bar{x} be the mean of the values measured: $\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$

Where:

- n is the sample size,
- x_i is the value measured of the characteristic under consideration for n sampled items.

6.4.9 Decision rules

The approach consists in comparing a Z-score (or standard error calculated from the limit values U and L , from the mean of the measurements and from the standard deviation of the distribution) with the constant k determined previously.

Method S

The estimated **standard deviation** s is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{(n-1)}}$$

where n is the sample size, x_i the value measured for n sampled items, and \bar{x} the mean of the various measured values x_i .

Calculating the Z score:

The Z-score expresses the value of an observation derived from a known population in terms of distance from a limit.

$$\text{For an upper limit } U, Z_U = \frac{(U - \bar{x})}{s}$$

$$\text{For a lower limit } L, Z_L = \frac{(\bar{x} - L)}{s}$$

Method σ

The known **standard deviation** is σ .

Calculating the Z score:

$$\text{For an upper limit } U, Z_U = \frac{(U - \bar{x})}{\sigma}$$

$$\text{For a lower limit } L, Z_L = \frac{(\bar{x} - L)}{\sigma}$$

The decision to accept or reject the lot is determined based on comparing Z_U or Z_L with the acceptance constant k .

The decision rules are common to the two methods and are given in table 13.

	Characteristic to exceed a minimum value L , $x \geq L$	Characteristic to not exceed a maximum value U , $x \leq U$	Characteristic to fall within an interval $L \leq x \leq U$
Lot accepted	$Z_L \geq k$	$Z_U \geq k$	$Z_L \geq k$ and $Z_U \geq k$
Lot rejected	$Z_L < k$	$Z_U < k$	$Z_L < k$ or $Z_U < k$

Table 13: Decision rules – Inspection by variables for percent nonconforming

6.5 Inspection of the mean of a characteristic

This sampling plan is used when inspecting the mean of a non-discrete and measurable characteristic of a lot, compared with the specifications for this mean.

6.5.1 Methodology

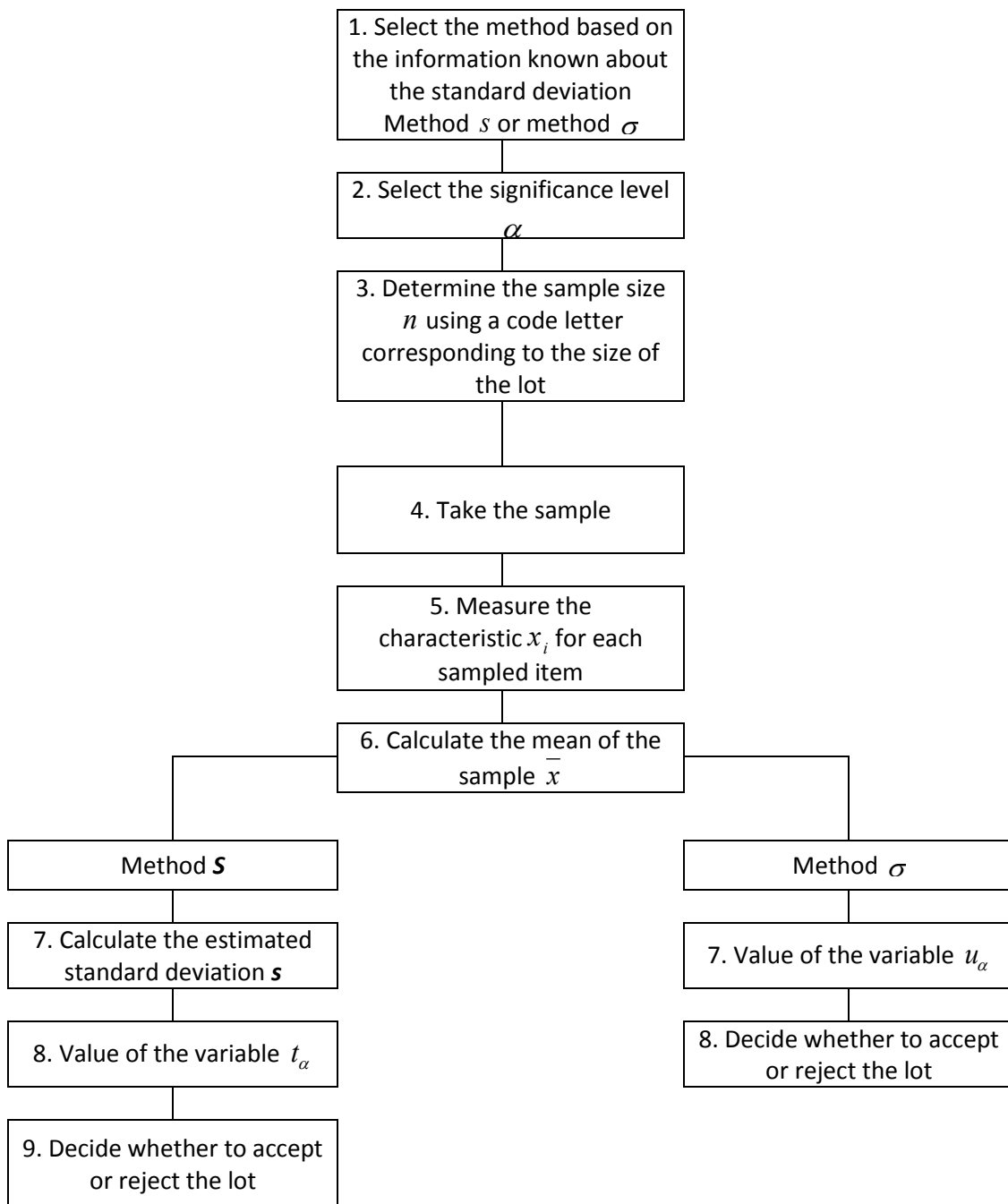
There are two types of situation:

- the standard deviation of the population of the lot is not known – in this case it is estimated (and denoted S) from the items in the sample.
- the standard deviation σ is known in advance (e.g. from previous results for the continuous production of packaged products).

For each of these two situations, specific sampling plans are implemented, using different sample sizes. However, the principles on which the acceptance criteria are based are very similar in both cases.

For this type of inspection, the concept of AQL does not apply. Another criterion is used: the significance level α , whose most common values are 0.5 and 5%.

Procedure: The diagram below shows the steps in the methodology for implementing a sampling procedure for a given consignment up until the point when the results of the test are known.



6.5.2 Selecting the significance level α

α is the significance level for the test of the mean, i.e. the probability of wrongly concluding that the mean of the characteristic inspected falls outside the estimated confidence interval. For a severe test the value chosen is $\alpha = 0.5\%$, for a more relaxed test the value chosen is $\alpha = 5\%$

6.5.3 Determining the sample size n

The first stage in determining the sample size is to find the code letter corresponding to the lot size, from table 14.

Lot size	Code letter
2 to 8	B
9 to 15	B
16 to 25	C
26 to 50	D
51 to 90	E
91 to 150	F
151 to 280	G
281 to 500	H
501 to 1,200	J
1,201 to 1,320	K
1,321 to 10,000	L
10,001 to 35,000	M
35,001 to 150,000	N
150,001 to 500,000	P
500,001 or more	Q

Table 14: Determination of the code letter as a function of lot size

The second stage is to determine, using the code letter obtained and as a function of the method used (s or σ), the corresponding sample size, from table 15.

Code letter	Sample size Method S		Sample size Method σ	
	Normal or tightened inspection	Reduced inspection	Normal or tightened inspection	Reduced inspection
B	3	3	2	2
C	4	3	3	2
D	6	3	4	2
E	9	4	6	3
F	13	6	8	4
G	18	9	10	6
H	25	13	12	8
J	35	18	15	10
K	50	25	18	12
L	70	35	21	15
M	95	50	25	18
N	125	70	32	21
P	160	95	40	25
Q	200	125	50	32

Table 15: Determination of the sample size as a function of code letter

Note that method σ is less expensive to conduct than method s since fewer samples are collected.

6.5.4 Taking the sample

The individual items selected for the sample must be taken from the lot by single random sampling. The measured value of the characteristic in item i is denoted as x_i .

6.5.5 Calculating the mean of the sample

n is the sample size,

\bar{x} is the mean of the characteristic inspected in the sample:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$$

6.5.6 Acceptance criteria and decision rules

6.5.6.1 Methods

Estimated standard deviation s

This value is calculated using the equation:

$$s = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{(n-1)}}$$

Variable t_α

t_α is the value of the Student's t distribution, with $n-1$ degrees of freedom and a significance level of α

Table 16 provides the values of the variable t_α for sample sizes n with a significance level of α . These values are used when calculating the decision rules.

Selected t_α values of the Student's distribution with $n-1$ degrees of freedom				
	$\alpha = 5\%$		$\alpha = 0.5\%$	
n	t_α	$t_{\alpha/2}$	t_α	$t_{\alpha/2}$
5	2.13	2.78	4.6	5.60
10	1.83	2.62	3.25	3.69
15	1.76	2.15	2.98	3.33
20	1.73	2.09	2.86	3.17
25	1.71	2.06	2.8	3.09
30	1.7	2.05	2.76	3.04
40	1.68	2.02	2.71	2.97
50	1.68	2.01	2.68	2.94
100	1.66	1.98	2.63	2.87
∞	1.65	1.96	2.58	2.81

Table 16: A few selected t values of the Student's distribution with $n-1$ degrees of freedom

Decision rules

M is the target value for the mean of the lot.

There are three possible cases:

1/ M is considered as a minimum value for the mean

The lot is accepted if, and only if: $\bar{x} \geq M - \frac{t_{\alpha} * s}{\sqrt{n}}$

2/ M is considered as a maximum value for the mean

The lot is accepted if, and only if: $\bar{x} \leq M + \frac{t_{\alpha} * s}{\sqrt{n}}$

3/ M is considered neither as a maximum value for the mean nor as a minimum value for the mean

The lot is accepted if, and only if: $M - \frac{t_{\alpha/2} * s}{\sqrt{n}} \leq \bar{x} \leq M + \frac{t_{\alpha/2} * s}{\sqrt{n}}$

6.5.6.2 Method σ

Variable U_{α}

U_{α} is the value of the standardised normal distribution corresponding to the significance level α , as indicated in table 17. These values are used to calculate the decision rules.

Selected U_{α} values of the standardised normal distribution corresponding to the significance level α			
$\alpha = 5\%$		$\alpha = 0.5\%$	
U_{α}	$U_{\alpha/2}$	U_{α}	$U_{\alpha/2}$
1.65	1.96	2.58	2.81

Table 17: A few selected values of U for a standardised normal distribution

Decision rules

M is the target value for the mean of the lot.

There are three possible cases:

1/ M is considered as a minimum value for the mean

The lot is accepted if, and only if: $\bar{x} \geq M - \frac{U_{\alpha} \times \sigma}{\sqrt{n}}$

2/ M is considered as a maximum value for the mean

The lot is accepted if, and only if: $\bar{x} \leq M + \frac{U_{\alpha} \times \sigma}{\sqrt{n}}$

3/ M is considered neither as a maximum value for the mean nor as a minimum value for the mean

The lot is accepted if, and only if: $M - \frac{U_{\alpha/2} \times \sigma}{\sqrt{n}} \leq \bar{x} \leq M + \frac{U_{\alpha/2} \times \sigma}{\sqrt{n}}$

6.6 Sampling error

Intrinsically, statistical sampling can only estimate the characteristics of the lot. An error is therefore associated with the result of the sampling. There will always be a difference between the value measured in the sample and the value measured for the entire lot.

The objective of the sampling plans is to manage this error and to ensure that, for a given cost, it is as small as possible.

The sampling errors may be determined experimentally, by multiplying different samplings for the same lot, or mathematically. This document does not cover the estimation of sampling errors, since this requires the use of specific statistical methods that the user may find in the normative texts listed herein as reference documents.

7 Techniques applied to the sampling of wines and musts

The sampling plans define how many items should be taken. At the sampling stage, environmental factors and the technique used may degrade the degree to which the sample is representative of the lot. These factors must be identified as critical points. Accordingly, appropriate sampling techniques must be selected and implemented to control these critical points and ensure the quality of the sample.

7.1 Packaged wines

Packaged wines are sampled by taking individual samples in accordance with a sampling plan. The sampling unit is one packaged unit (bottle, bag-in-box, etc.). There are no critical points associated with packaged wines, unless the wine or must is transferred into a new container.

7.2 Wines and musts in bulk

7.2.1 Critical points

7.2.1.1 Interface effects

Contact with a gas or solid that might release certain substances can change one or more of the characteristics investigated in the must or wine located near to the contact interface. Taking a sample from this zone could degrade the degree to which the sample represents the entire volume.

Examples:

- *contact with ambient air at opening points on the container (doors, chimneys, bungs, etc.);*
- *contact with contaminated container walls (polyester can release styrene, excessive tartar can contain micro-organisms, etc.);*
- *contact with solids immersed in the wine or must (pieces of oak, etc.);*
- *contact with sampling systems fitted to the container (metal taps, valves, etc.).*

Procedure to follow:

- if the interface effect is **local** and does not affect, under any circumstances, the entire volume, then the sample should be taken from a location away from the zone that is locally affected.

Examples:

- o *effect of oxygen on the surface of the wine under a tank's chimney;*
 - o *contamination of the wine due to its contact with the metal components of the tank's taps and associated pipes.*
- for the particular case of contamination of the wine in the tank's taps, valves and associated piping, the wine in these locations should be flushed through so that this liquid cannot be included in the sample, even partially.
 - if the interface effect could **affect the entire volume** of the lot, the volume from which the sample is to be taken should be mixed before taking the sample.

Example: effect of pieces of oak on the organoleptic or chemical characteristics of the wine

7.2.1.2 Gravity effects

Gravity can change a uniform medium into one in which there is a concentration gradient of the characteristic(s) susceptible to this effect.

Example: Gravity affects the distribution of solid particles in a liquid medium. This may be the case, for wines and musts, for various possible precipitations (tartar crystals, various cassia, etc.) and micro-organisms.

Procedures to follow:

- If the sample is designed to provide a mean value of the lot for the characteristic under consideration: the volume from which the sample is to be taken should be mixed before taking the sample.
- If the sample is designed to provide information about a specific risk, e.g. the presence of micro-organisms: the sample may be taken from a specified zone.

7.2.1.3 Homogeneity of the additives and oenological inputs

When a soluble product is added to the wine or must it is not always distributed instantaneously and uniformly through the entire volume.

Examples:

- *homogeneity of the SO₂ in a tank immediately after sulphiting;*
- *problematic mixing of products for enrichment (concentrated must, grape sugar or rectified concentrated must, sucrose);*

- *homogenisation of metatartaric acid, etc.*

Procedure to follow:

- allow a sufficient period of time after adding an additive to ensure the homogeneity of the soluble product (some products require, in all cases, dynamic mixing/solubilisation);
- mixing of the volume before taking the sample.

7.2.1.4 Unstable and changing media

The must or wine may be going through a period of significant change. The sample taken may then not be sufficiently representative, since the change experienced by the sample may not necessarily be the same as that experienced by the product in its original container.

Examples:

- *Musts and wines during alcoholic or malolactic fermentation;*
- *Samples of musts likely to precipitate tartar and/or not follow a normal fermentation process, etc.*

Procedure to follow:

- limit the time period between sampling and measuring
- stabilise the sample to “freeze” the characteristics investigated at the instant when the sample was taken (see § 7.2.7)

7.2.1.5 Effects of the sampling equipment on the sample

Care should be taken to ensure that the sampling equipment and the sample container do not change the composition of the sample taken.

7.2.1.5.1 Sampling devices

All sampling equipment must always be inspected carefully to ensure that it is clean and dry; for microbiological sampling it must also be sterile.

The same requirement applies to sampling systems fitted to the original container (pipes, taps and valves). Allowing the sample to flow via a tap or valve, or to come into direct contact with the sampling system may contaminate the sample chemically (primarily from the metals from which the taps or valves are made) or microbiologically (micro-organisms frequently grow in these interface zones).

7.2.1.5.2 Container holding the samples

Preparing the containers

Before use, and in order to preserve the integrity of the sample, the sample container should be inspected to ensure that it is clean and dry. It may be beneficial to rinse the sample containers with some of the liquid from which the sample is subsequently taken. The container must be sterile for some types of analysis, e.g. microbiological analyses.

Container materials

The sample containers must be made from a material that can preserve the sample until its analysis without modifying the characteristic(s) under investigation. The same requirement applies to the stoppering system used.

Examples:

- *glass or plastic bottles are suitable for most analyses.*
- *plastics offer advantages in terms of light weight and strength, but may impair the integrity of the sample by adsorbing certain compounds (e.g. haloanisoles) and/or by releasing chemical compounds into the sample (phthalates, etc.).*
- *plastics are porous to oxygen and thus unsuitable for the long-term storage of samples.*

Volume of the containers

The volume of the container must be selected based on the quantity of sample required for analysis (and/or storage). This quantity is defined by the laboratory as a function of the analysis required.

Ullage

Sufficient vacant space should be left in the container to accommodate expansion of the liquid. This ullage must be small to minimise the effect of the oxygen which it contains on the wine and must. Some methods allow this ullage to be filled with an inert gas.

Stoppering the containers

The stopper used to close the sample container must not modify the characteristic(s) under investigation. In most cases, the stopper must be impermeable to the sample as a liquid and to any gases evolved. For the particular case of fermenting wines and musts, a stopper may be selected that allows carbon dioxide to escape.

7.2.1.6 Effects of handling the sample

Transferring or decanting between containers and/or the transport of the sample from the time and place of its sampling to those of its analysis must not modify the characteristic(s) under investigation.

7.2.1.7 Effect of sample stabilisation

See § 7.2.3

7.2.1.8 Effects of sample storage

See § 7.2.4

7.2.2 Sampling techniques for wines and musts in bulk

A distinction is made between direct sampling systems, wherein the sample-taking container constitutes the final sample container, and indirect sampling systems, wherein the product is transferred or decanted from the sample-taking container to the final sample container.

7.2.2.1 Direct sampling systems

7.2.2.1.1 Direct sampling using an immersion system

This method relies on the tank being fitted with a chimney, a door at the top of the tank or a large bung. Immersion systems involve immersing one or more sample containers into the liquid to be sampled. These systems can be fitted with operator-controlled stoppers or valves. This technique can be used to take “zone” or “all level” samples. When the immersion technique is possible it offers the best solution for wines and musts in tanks, since it ensures that the sample is properly representative.

7.2.2.1.2 Direct sampling via a tap or valve

Samples may be taken via the taps or valve fitted to the container of wines or musts specifically for sampling purposes. The sample container(s) can be filled directly. The pipes and valves should systematically be flushed through with some liquid before taking the sample.

7.2.2.1.3 Direct sampling using a siphon

For tanks fitted with a bung only, and for casks or barrels, sampling via a siphon, with a flexible tube, offers the same advantages as immersion systems. The flexible tube must be made from a material which does not react with the wine or must, and must be cleaned and flushed through before use

7.2.2.2 Indirect sampling systems

7.2.2.2.1 Indirect sampling using an immersion system

With this system, the immersed sample-taking container is not the sample container, and the wine or must is transferred after taking the sample. The sample must be transferred immediately after being collected.

Examples:

- *“Sharp-tipped” tube for taking a sample from above the surface, and for piercing the cap, if necessary.*
- *Sampling pipettes for barrels or small containers made from various materials (glass, plastic, etc.).*
- *Immersion system with a fixed sample-taking container.*

Sample-taking containers must be clean and rinsed through with the product to be sampled.

7.2.2.2.2 Indirect sampling via a tap or valve

The principle and limitations are the same as for direct sampling via a tap or valve, except that the sample-taking container is not the sample container (the contents of the sample-taking container are transferred to a sample container. Sample-taking containers must be clean and rinsed through beforehand with the product to be sampled.

7.2.3 **Stabilising the samples**

In order to stabilise and extend the shelf life of the characteristic(s) under investigation the sample may be stabilised. Although the selected stabilisation technical must not modify the characteristic(s) under investigation, it may have effects on other characteristics. For this reason, when sample stabilisation is performed it must be indicated on the sample's label.

There are various stabilisation methods:

- Physical stabilisation of the sample by refrigeration, freezing or high-temperature pasteurisation.
- Chemical stabilisation: sulphur dioxide, salicylic acid, natamycin, etc.

7.2.4 Storing the samples

The shelf life of a sample depends on the nature of the wine or must, on the characteristics investigated, on the nature of the container and on how well the container is stoppered, particularly its impermeability to gases.

The storage conditions must also be controlled to ensure that the characteristic(s) investigated are preserved.

7.3 Sample identification

The samples taken must be identified as samples, with a reference indicating the lot from which they were collected. When the sampling plan requires several samples to be collected then these samples must be identified individually (numbered, etc.).

The label must adhere firmly to the sample's container.

It is advisable to include the following information on the label:

1. The words "Sample for analysis"
2. Owner of the lot
3. Nature and quantity of the consignment
4. Identification and quantity of the lot sampled
5. Sampling date
6. Name of the sampling operator
7. Sample number (if multiple samples are taken)
8. Sample quantity
9. Stabilisation techniques and additives, if applicable
10. Shelf life of the sample.

8 Sampling examples

The examples given in this section correspond to situations frequently encountered in practice. The solutions described are provided for information only and may be implemented differently by the person responsible for the sampling process to suit the specific conditions encountered.

8.1 Sampling from wine in a tank

8.1.1 Measuring a chemical parameter in wine in a tank: Alcoholic Strength by Volume (ASV)

This example relates to a straightforward situation, with an undivided lot in which the characteristic under investigation is distributed uniformly. The sampling plan simply requires the collection of a single sample. The sampling techniques must ensure that the sample is representative.

1. Consignment

Wine in a tank

2. Lots

1 undivided lot = tank.

3. Characteristics

ASV

4. Distribution

The lot is considered to be homogeneous for this characteristic for a standard size of tank.

5. Sampling plan

The ASV is uniformly distributed in the wine in the tank: taking a single sample is sufficient so long as the precautions applicable to sampling from a tank are applied. No statistical sampling plan is necessary.

6. Sampling method

When sampling from a tank the following key critical points must be considered:

- surface effects;
- interface effects at the tap or valve used for sampling.

Direct sampling using an immersion system is the most suitable solution.

Other sampling methods are possible (the list below is not exhaustive):

- Indirect sampling using an immersion system;
- Sampling via a tap or valve (after flushing through).

7. Transporting and storing the sample

If a stopper is fitted that provides impermeability to gases, and so long as there are no excessive variations in temperature, the sample collected has a shelf life of several days.

8.1.2 Measuring the sulphur dioxide content of wine in a tank

This example is similar in some respects to the previous example. However, the characteristic under investigation, sulphur dioxide, is not necessarily distributed uniformly through a tank, notably if sulphiting has been carried out recently.

1. Consignment

Finished wine in a tank

2. Lots

1 undivided lot = 1 tank.

3. Characteristics

Free and total sulphur dioxide.

4. Distribution

Homogeneity not achieved initially.

5. Sampling plan

If prior mixing of the wine in the tank is not carried out, a targeted sampling plan is implemented, with two samples taken: one from the top and the other from the bottom of the tank.

If it can be assumed that this characteristic is uniformly distributed through the wine, either because it has not been sulphited recently, or because the tank contents have been mixed, then the sampling plan can be reduced to taking a single sample.

6. Sampling method

When sampling from a tank the following key critical points must be considered:

- surface effects;
- interface effects at the tap or valve.

Direct sampling using an immersion system is the most suitable solution.

Other sampling methods are possible (the list below is not exhaustive):

- Indirect sampling using an immersion system
- Sampling via a tap or valve (after flushing through).

7. Transporting and storing the sample

The free and total sulphur dioxide content can change fairly rapidly in a sample container. The sample cannot be stored for more than 3 days, and the storage temperature must be less than 18 – 20°C.

8.1.3 Microbiological measurements on wine in a tank: search for yeasts

The characteristic under investigation is not distributed uniformly through the tank, due to a gravity effect. Since the aim of the sampling is to investigate the presence of a microbiological population, mixing prior to sampling is not necessarily an appropriate solution. Instead, the sample may be taken from the zone that is most likely to contain the characteristic under investigation, in this case the bottom of the tank.

The sampling method must consider the restrictions associated with taking a sample for microbiological analysis.

1. Consignment

Finished wine in a tank

2. Lots

1 undivided lot = 1 tank.

3. Characteristics

Presence of a yeast population

4. Distribution

The characteristic is not uniformly distributed through the lot due to a gravity-induced gradient, which concentrates the populations at the bottom of the tank. Two sub-lots may thus be defined, within which the characteristic is presumed to be homogeneous and uniformly distributed:

- Top of the tank
- Bottom of the tank

5. Sampling plan

The sampling plan is a targeted sampling which concentrates on the “bottom of the tank” sub-lot for a maximum estimation of the populations.

Taking a single sample may be sufficient.

6. Sampling method

The sample-taking container must be sterile.

Direct-type sampling is very strongly recommended to avoid any external contamination when the sample is taken.

The sample is targeted at the bottom of the tank, and collected either:

- Via a tap or valve: flush the piping extensively and sterilise the tap by flaming with alcohol;
- Via an immersion system: which requires a controllable system for opening/sealing the immersed container.

7. Transporting and storing the sample

The sterility of the sample must be preserved by ensuring the stopper's integrity and ability to seal. For a microbiological investigation, the shelf life of the sample is short. The temperature at which the sample should be stored until it arrives at the place of analysis must be between 10 and 25°C

8.2 Sampling wine in a barrel

8.2.1 Measuring the ASV of wine in a drum

There are strong similarities between this example and § 8.1.1. In this case the lot is divided into several containers.

There are two possible hypotheses:

- *the ASV is uniform from one drum to another;*
- *factors may have influenced the ASV differently from one barrel to another.*

The sampling plan must accommodate these two hypotheses.

1. Consignment

Finished wine in barrels

2. Lots

1 lot divided into 30 barrels.

3. Characteristics

ASV

4. Distribution

The ASV is uniform from one barrel to another. This hypothesis must, however, be confirmed.

5. Sampling plan

If the characteristic is distributed uniformly a single sample taken from one barrel may be sufficient. To confirm this hypothesis, several samples should, however, be taken. For this example, one option would be to take three samples and to check that the results for the three samples are similar. If there is a difference in the results a statistical sampling plan of the type proposed in § 8.2.1 should then be implemented.

6. Sampling method

The key critical points relating to taking samples from barrels are:

- surface effects;
- effects induced by the sampling equipment.

Direct sampling via a siphon may be used, or indirect sampling via a pipette, taking the usual precautions.

7. Transporting and storing the sample

If a stopper is fitted that provides impermeability to gases, and so long as there are no excessive variations in temperature, the sample collected has a shelf life of several days.

8.2.2 Measuring the sulphur dioxide content in wine in a barrel

This example considers a divided lot, in which the characteristic is not distributed uniformly. A statistical sampling plan is then implemented for an inspection by attribute.

1. Consignment

Finished wine in barrels

2. Lots

1 lot divided into 30 barrels.

3. Characteristics

Content of free sulphur dioxide correctly adjusted for maturing in barrels.

The conformance criterion selected here, for inspection by attribute, is a free sulphur dioxide content of more than 25 mg/L.

4. Distribution

It is assumed that the free sulphur dioxide content varies from one barrel to another. The distribution is likely to be normal, or may be treated as normal.

5. Sampling plan

A statistical sampling plan is implemented: inspection by attribute (see § 6.3)

- Sample size

lot size = 30

code letter: D

normal inspection ⇔ 8 individual items

- AQL: 0%

- Decision-making criteria:

Acceptance criterion = 0

Rejection criterion = 1

6. Sampling method

The key critical points relating to taking samples from barrels are:

- surface effects;
- effect induced by the sampling equipment.

Direct sampling via a siphon may be used, or indirect sampling via a pipette, taking the usual precautions.

7. Transporting and storing the sample

The free and total sulphur dioxide content can change fairly rapidly in a sample container. The sample cannot be stored for more than 3 days, and the storage temperature must be less than 18 – 20°C.

8.3 Sampling bottled wine

8.3.1 Investigating the conformance with the analytical criteria stated in the requirements specification

The lot is divided into bottles. In the absence of variation of the characteristic(s) investigated the sampling plan is straightforward. This is the case considered in this example.

1. Consignment

Lot of 12,000 bottles, at the outfeed from the packaging line.

2. Lots

1 lot of wine taken from the same tirage tank, packaged without a break over a short period of time, under the same technical conditions into equal-volume containers sealed with the same stopper. The lot number is constant.

3. Characteristics

Analytical criteria as defined in the requirements specification

4. Distribution

Characteristic presumed to be homogeneous and stable.

5. Sampling plan

When the distribution is uniform, one sample is sufficient, taken ideally at the mid-point of the tirage process.

6. Sampling method

Sample the bottle(s) randomly.

7. Transporting and storing the sample

The bottled wine is considered to be stable. The sample may be stored for a long period of time.

8.3.2 Searching for a random defect: contamination with Trichloroanisole (TCA) introduced by contaminated corks

The lot is divided into bottles. The “mouldy” taint resulting from contamination by TCA varies randomly from one bottle to another. A statistical sampling plan is thus selected.

1. Consignment

Lot of 12,000 bottles.

2. Lots

1 lot, a single bottling process, same lot number, same lot of corks.

3. Characteristics

“Musty-earthy” taste item does not conform if this characteristic is present.

4. Distribution

The lot is homogeneous for this characteristic, whose distribution type is as a “rare event” modelled by Poisson’s law.

5. Sampling plan

A statistical sampling plan is implemented: inspection by attribute (see § 6.3)

- Sample size:

lot size = 12,000

code letter: M

A reduced inspection is selected for cost reasons.

↳ the sampling process collects 125 bottles.

- AQL:

AQL of 4%

- Decision-making criteria:

Acceptance criterion = 10

Rejection criterion = 11

6. Sampling method

Sample the bottles randomly from the entire lot.

7. Transporting and storing the sample

Bottled wine is stable. The characteristic under investigation evolves fairly slowly. The sample may be stored for several weeks.