

SAMPLING AND ANALYSIS FOR VERIFYING SAFETY OF FOOD

SAMPLING IN CODEX STANDARDS – HOW IT SHOULD BE TREATED

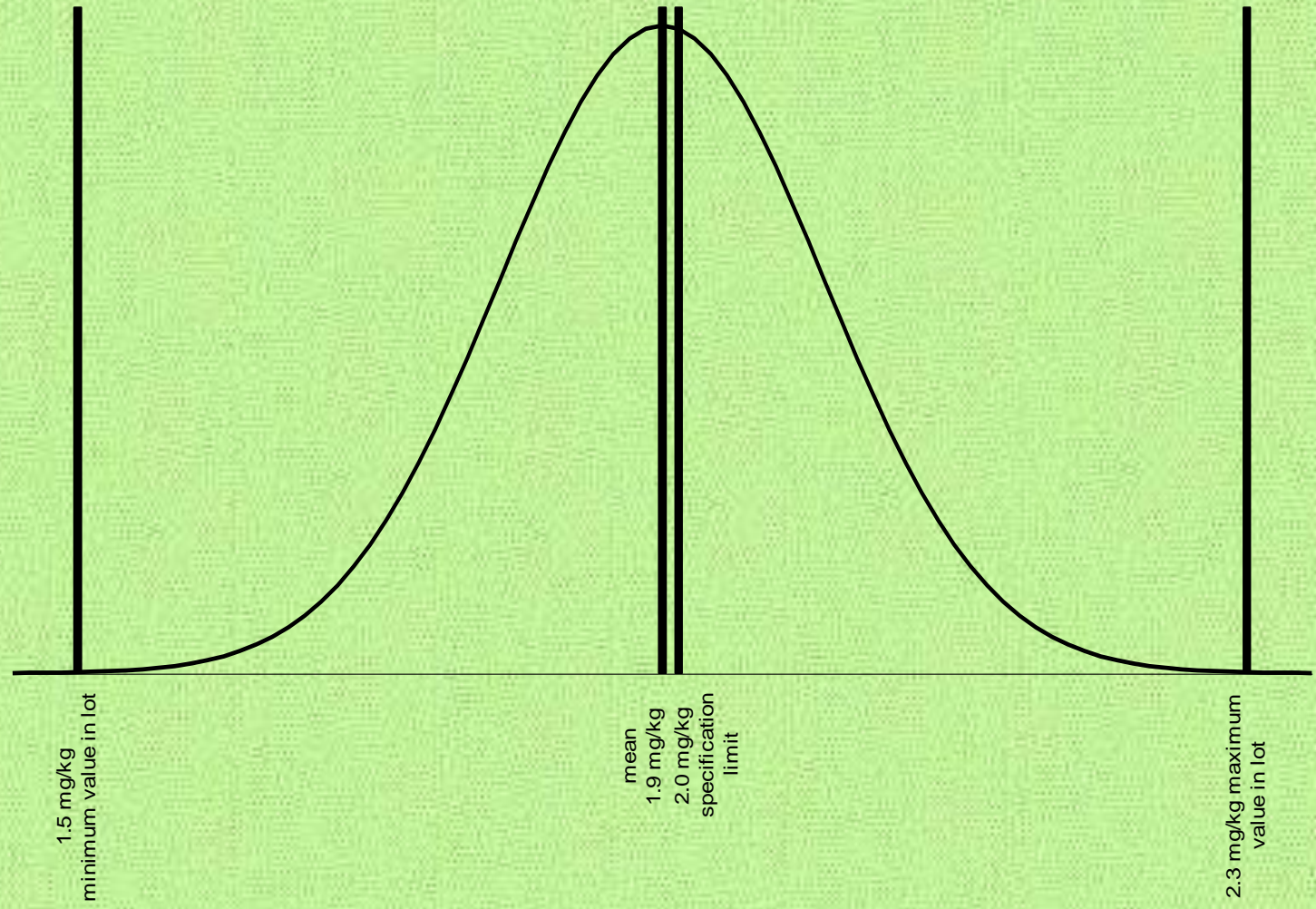
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Frequency



1.5 mg/kg
minimum value in lot

mean
1.9 mg/kg
2.0 mg/kg
specification
limit

2.3 mg/kg maximum
value in lot

Two countries may have different national rules for the interpretation of results from lots.

Country A requires that each and every item in the lot meets the specification. In this example it means that all 1,000 units, if analysed separately, would have to be less than 2.0 mg/kg. Here a significant number of units are greater than 2.0 mg/kg so the lot would be deemed to be in non-compliance with the legal specification and so would be rejected.

Country B requires that the mean value of the specification in the lot is to be less than the legal specification. In this case the mean value is 1.9 mg/kg so the lot would be deemed to be in compliance with the legal specification.

Consequence: the two countries A and B will make different judgements as to compliance with a legal specification on essentially the same lot. This is unacceptable and can only be avoided if the sampling procedures are elaborated at the same time as the commodity standard is elaborated. In addition it should also be noted that the number of units to be analysed also influences the decision on compliance.

To recognise that different sampling plans when applied to the same lot may result in different assessments of the lot with respect to a Codex specification. In that way sampling is similar in effect as Type I, empirical, method of analysis, i.e. if a sampling plan is not specified then the application of different sampling plans by different operators to the same lot may result in different decisions with respect to compliance of the lot with the specification. In addition, the application of the same sampling plan by different operators to the same lot may also result in different decisions with respect to compliance.

To recognise that sampling is complex and inherently variable when considering lots. As a result many Codex Committees do not specify a defined sampling plan in many (most?) of their Standards.

Acceptable Quality Level (AQL) (from GL 50)

The **Acceptable Quality Level (AQL)** is used as 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). This is a quality goal fixed by the profession. This does not mean that all the lots having a rate of defective items greater than the AQL will be rejected at the control, but this means that the higher the rate of defective items exceeds the AQL, the greater is the probability of rejection of a lot. For any given sample size, the lower the AQL, the greater the protection for the consumer against accepting lots with high defective rates, and the greater the requirement for the producer to conform with sufficiently high quality requirements. Any value for AQL should be realistic in practice and be economically viable. If necessary, the value of AQL should take into account safety aspects.

It should be recognised that the selection of a value for the AQL depends on the specific characteristic considered and of its relevance (economic or other) for the standard in its whole. A risk analysis may be undertaken to assess the possibility and severity of negative impacts on public health caused, for example, by the presence in food products of additives, contaminants, residues, toxins or pathogenic micro-organisms.

The characteristics which may be linked to critical defects (for example to sanitary risks) shall be associated with a low AQL (i.e. 0,1 % to 0,65 %) whereas the compositional characteristics such as the fat or water content, etc may be associated with a higher AQL (e.g., 2,5 % or 6,5 % are values often used for milk products). The AQL is used as an indexing device in the tables of the Standards ISO 2859-1, ISO 3951 and in some tables of ISO 8422 and ISO 8423.

Sampling has been discussed in Codex for the past 40 years!

Originally driven by the AQL 6.5% attributes plan for visible defects.

Acceptance sampling became the norm.

Led to the “PRINCIPLES FOR THE ESTABLISHMENT OR SELECTION OF CODEX SAMPLING PROCEDURES”

Which are:

Purpose of Codex Methods of Sampling

Codex Methods of Sampling are designed to ensure that fair and valid sampling procedures are used when food is being tested for compliance with a particular Codex commodity standard. The sampling methods are intended for use as international methods designed to avoid or remove difficulties which may be created by diverging legal, administrative and technical approaches to sampling and by diverging interpretation of results of analysis in relation to lots or consignments of foods, in the light of the relevant provision(s) of the applicable Codex standard.

Important – applicable to lots.

Types of Sampling Plans and Procedures given in the Procedural Manual

Sampling Plans for Commodity Defects:

Such plans are normally applied to visual defects (e.g. loss of colour, misgrading for size, etc.) and extraneous matter. They are normally attributes plans, and plans such as those included in Section 3.1 and 4.2 of the *General Guidelines on Sampling (CAC/GL 50-2004)* (hereinafter referred to as "General Guidelines") may be applied.

Sampling Plans for Net Contents:

Such plans are those which apply to pre-packaged foods generally and are intended to serve to check compliance of lots or consignments with provisions for net contents. Plans such as those included in Section 3.3 and 4.4 of the General Guidelines may be applied.

Sampling Plans for Compositional Criteria:

Such plans are normally applied to analytically determined compositional criteria (e.g., loss on drying in white sugar, etc.). They are predominantly based on variable procedures with unknown standard deviation. Plans such as those included in Section 4.3 of the General Guidelines may be applied.

Specific Sampling Plans for Health-related Properties:

Such plans are normally applied to heterogeneous conditions, e.g. in the assessment of microbiological spoilage, microbial by-products or sporadically occurring chemical contaminants.

General Instructions for the Selection of Methods of Sampling

- (a) Sampling methods described in the General Guidelines or official methods of sampling elaborated by international organizations occupying themselves with a food or a group of foods are preferred. Such official methods may be written using the General Guidelines when attracted to Codex standards.

- (b) When selecting appropriate sampling plans, Table 1 in the General Guidelines may be utilized.

- (c) The appropriate Codex Commodity Committee should indicate, before it elaborates any sampling plan, or before any plan is endorsed by the Codex Committee on Methods of Analysis and Sampling, the following:

the basis on which the criteria in the Codex Commodity standards have been drawn up (e.g. whether on the basis that every item in a lot, or a specified high proportion, shall comply with the provision in the standard or whether the average of a set of samples extracted from a lot must comply and, if so, whether a minimum or maximum tolerance, as appropriate, is to be given);

whether there is to be any differentiation in the relative importance of the criteria in the standards and, if so, what is the appropriate statistical parameter each criterion should attract, and hence, the basis for judgement when a lot is in conformity with a standard.

SAMPLING IN CODEX

1986: The principles for the establishment or selection of codex sampling procedures were first adopted by the Commission.

1988: Instructions on Codex sampling procedures.

These covered:

Aspects of sampling and acceptance procedures

Types of sampling plans

Procedure to be followed by Codex Commodity Committee when developing a sampling plan

Diagrammatic representation of possible Codex sampling plans

Description of and formulae to be used in acceptance sampling plans adopted by Codex

Net contents

Selection of values of mathematical parameters for the operation of Codex sampling plans

2004: General Guidelines on Sampling (published as CAC/GL 50-2004).

These 1988 Guidelines extended and made much more detailed. Off-putting for Codex Committees?

Diagrammatic representation of types of acceptance plans shown:

DIAGRAMMATIC REPRESENTATION OF POSSIBLE CODEX SAMPLING PLANS

The various possible types of Codex sampling plans are explained diagrammatically below.

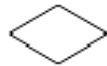
Symbols



Individual items within a lot



Analysis of item or blended bulk sample



Decision on whether concentration of item meets specification



Mix samples into a homogeneous blended bulk sample

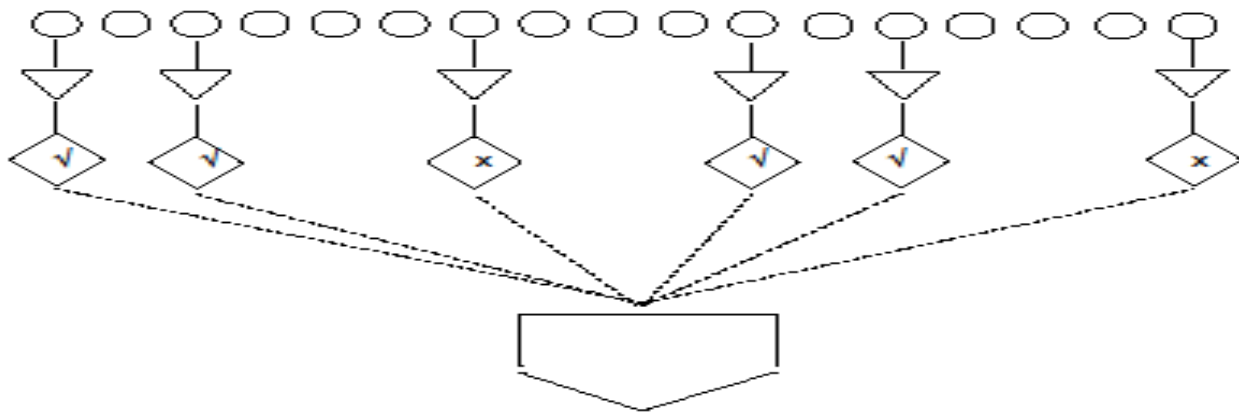


Prepare estimate of distribution curve of concentration within units from analytical measurements.







Decision on whether analysis of sample indicates lot meet specifications for characteristic in Codex Standard

ATTRIBUTE PLANS FOR PROPORTION DEFECTIVE



Procedure

1. Set AQL.
2. Sample prescribed number of discrete items  from lot 
3. Analyse each item individually  satisfactory
 unsatisfactory
4. Let x = number of defective items in the sample; then if $x < c$, accept the lot: if $x > c$, reject the lot.
5. If $c = 0$, then akin to an each-and-every-item-must-comply system.

Sampling by attributes is sampling whereby either the item or the product is classified as defective or non-defective with respect to a given requirement or set of requirements.

“Item” and “defective” are defined.

The number of defective items, c , permitted in the samples. For different AQL levels, and probabilities, is given in Table 2. The lot is accepted when the number of defective items equals or is less than c .

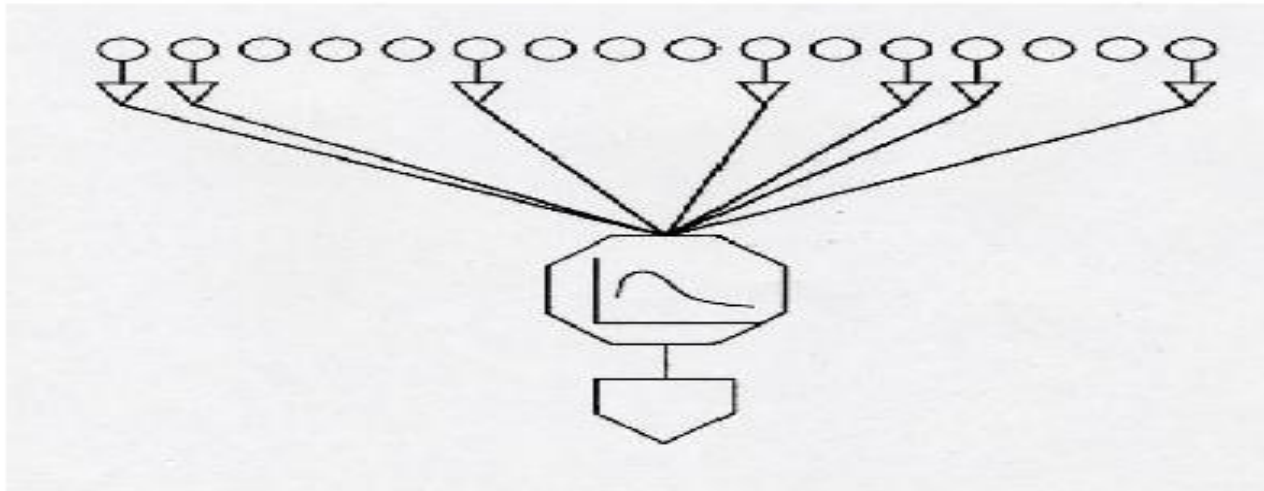
1. Sample Size

The number of items to be inspected from lots of different sizes as five different levels of inspection is given in Tables.

2. Operating Characteristics

The percentage of defective quality items in submitted lots having 95%, 50% and 10% chance of being accepted by the Sampling Plan are given in Tables.

VARIABLES PLAN FOR PROPORTION DEFECTIVE: UNKNOWN STANDARD DEVIATION



Procedure

1. Set AQL
2. Sample prescribed number of discrete items from lot
3. Analyse each item individually.
4. Calculate mean and standard deviation (s). Whether that standard deviation includes the sampling as well as analysis component should be clearly defined.
5. Calculate to see if proportion defective is exceeded from given formula ($\text{mean} \leq U - ks$ or $\text{mean} \geq L + ks$) where U is the upper specification limited and L is the lower specification limit.

6. Accept/reject lot if proportion defective criterion is satisfied/exceeded.

Sampling by variables is sampling whereby the values of a specified criterion for a set of items forming the sample are measured on a continuous scale and the values used to determine the acceptability or otherwise of the lot from which the items are taken.

The lot is accepted when:

$$\bar{x} \leq U - ks$$

or

$$\bar{x} \geq L + ks$$

Where

\bar{x} is the mean value of the characteristic under consideration in the sample as is

U is the upper specification limit

L is the lower specifications limit

k is the constant multiplier associated with the scheme

s is the sample estimate of the criterion standard deviation

1. Sample Size

The number of items to be inspected from lots of different sizes at five different levels of inspection are given in Tables.

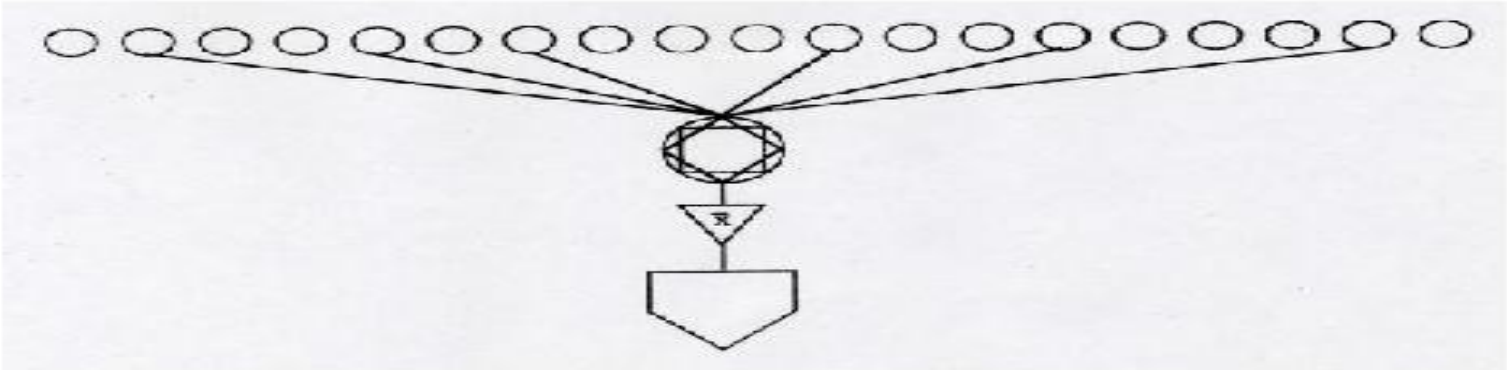
2. Operating Characteristics

The percentage of defective quality items in submitted lots having 95%, 50% and 10% chance of being accepted by the Sampling Plan is given in Tables.

n	AQL	95%	50%	10%
3	6.5	3.5	25	57
4	6.5	4.1	23	50
5	6.5	4.3	21	45
7	6.5	4.6	18	43
10	6.5	4.6	18	37
15	6.5	5.3	14	26
20	6.5	5.7	13	23
25	6.5	6.0	13	21
35	6.5	6.2	12	19
50	6.5	6.5	11	17
75	6.5	6.8	11	15
100	6.5	7.0	10	14
150	6.5	7.1	9.9	13
200	6.5	7.4	9.9	12

n = sample size and
Percentage of Defectives in a lot which may be accepted
95% , 50% and 10% of the time

**VARIABLES PLAN FOR PROPORTION
DEFECTIVE: KNOWN STANDARD DEVIATION**



Acceptance Sampling

Strengths:-

Easy to implement, once the correct protocol has been devised.

Recognises that producers and consumers both have risks of incorrect decisions and that they need to be balanced.

Makes empirical estimates of variability arising from sampling, sample preparation and chemical analysis, and uses them to adjust the effective threshold (e.g. as AQL).

Weaknesses:-

Underestimates the overall uncertainty of the measurement (excludes contribution from sampling), which will affect the reliability of decisions on batch acceptance/rejection.

Does not give the information on sampling variability (and hence larger measurement uncertainty) to the decision maker.

No way of checking on the quality of the actual implementation of the sampling protocol in routine operation.

Hard to devise correct protocol for heterogeneous material sampled *in situ* (e.g. un-mixed nuts in a container).

Does not include potential financial losses that may arise from decision errors (caused by uncertainty) in calculation of final sampling protocol.

Weaknesses:-

Gives rise to a high probability of accepting defective items, especially for small batches, that is not appreciated by some regulators.

Multicity effect of probability if characteristics measured are independent.

Research work carried out by UK Ministry confirmed that.

An Alternative Way Forward

Auto-Control

Auto-control is a system based on the official use of results of self-monitoring obtained by a production facility. Provided that the validity of these factory results can be verified they could replace the official control laboratory results to decide if the product meets quality specifications.

Problems with present system

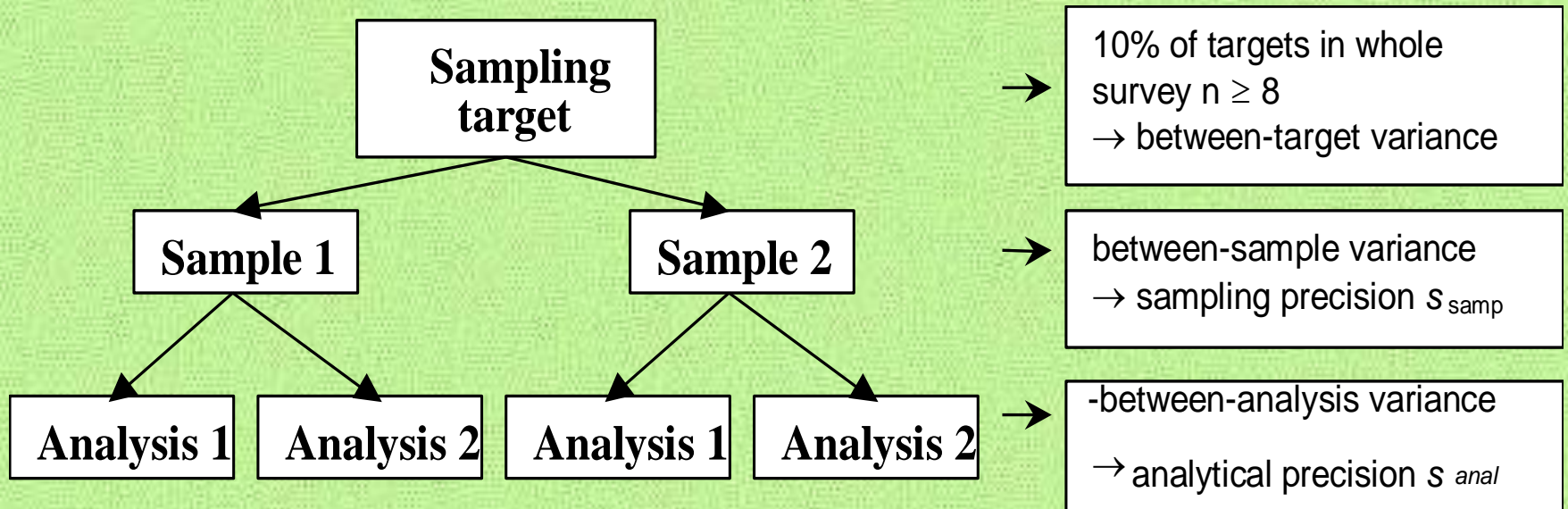
Difficult to improve present system on cost basis – but are we losing much available data. Some sectors have looked the possibility of using manufacturer's continuous control data.

Within a single country that is possible – but for cross border issues it becomes much more difficult.

MEASUREMENT UNCERTAINTY DERIVED FROM BOTH SAMPLING AND ANALYSIS

Does Measurement Uncertainty apply to both Sampling and Analysis?

Does the Measurement Uncertainty including from sampling have to be Estimated in Codex?



**THREE EXAMPLES FROM THE FOOD SECTOR
FOR TOTAL MEASUREMENT UNCERTAINTY
ESTIMATES**

Example 1 – Nitrate concentration in glasshouse lettuce

All values given in mg kg⁻¹

Mean: 4346

Standard deviation of analysis : 167.2

Standard deviation of sampling : 448.0

Example 2 – infant wet meals (retail survey)

All values given in $\mu\text{g kg}^{-1}$

Mean: 7.7

Standard deviation of analysis : 1.754

Standard deviation of sampling : 0.689

Example 3 – Moisture in wholesale butter (offered for EU subsidy)

All values given in g 100g⁻¹

Mean: 15.75

Standard deviation of analysis : 0.041

Standard deviation of sampling : 0.219

Upper
Control
Limit



(i)
Result less
analytical and/or
total uncertainties
above limit

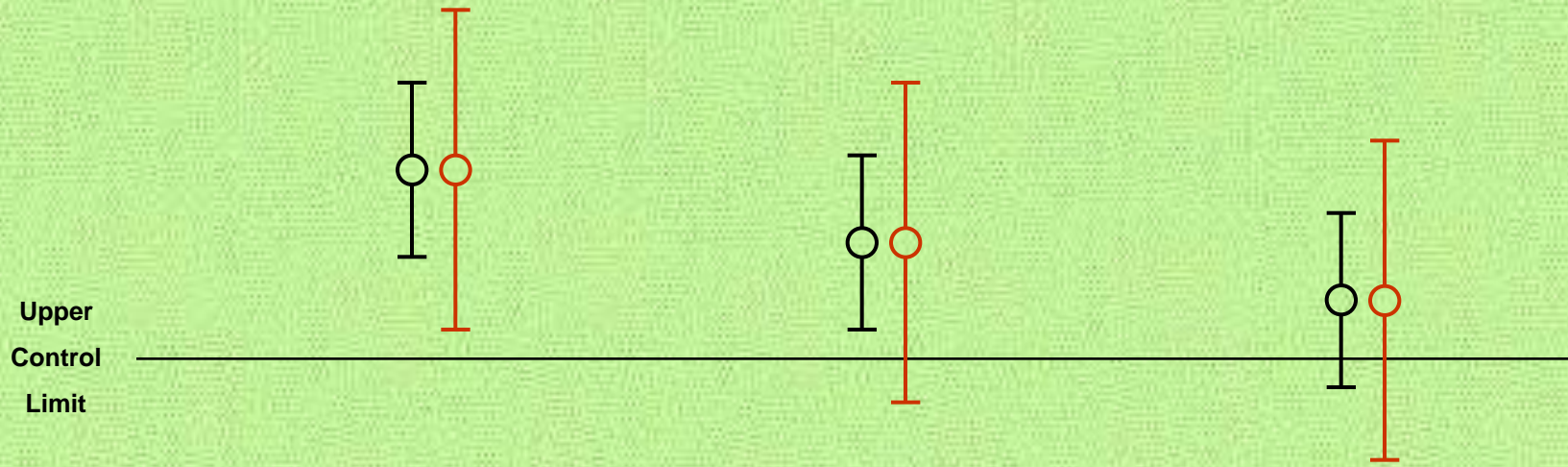
(ii)
Result less analytical
uncertainty is above
limit, but limit
is within total
uncertainty

(iii)
Result above limit
but limit within both
analytical and total
uncertainties

(iv)
Result below limit
but limit
within both
analytical and total
uncertainties

(v)
Result below limit but
limit plus analytical
uncertainty
still below limit but within
total uncertainty

(vi)
Result plus
analytical or total
uncertainties below
limit

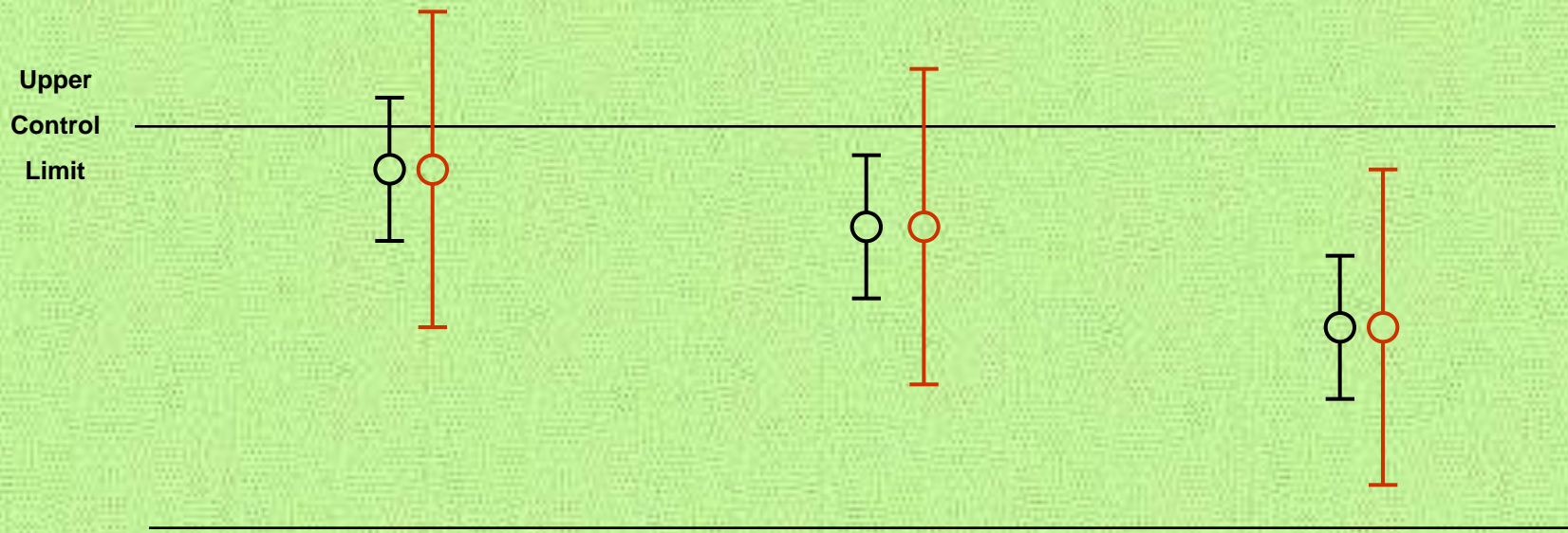


Upper
Control
Limit

(i)
Result less analytical and/or total
uncertainties above limit

(ii)
Result less analytical
uncertainty is above limit, but limit
is within total uncertainty

(iii)
Result above limit but
limit within both
analytical and total
uncertainties



(iv)

Result below limit but limit within both analytical and total uncertainties

(v)

Result below limit but limit plus analytical uncertainty still below limit but within total uncertainty

(vi)

Result plus analytical or total uncertainties below limit

Conclusions

Measurement uncertainty has to be estimated.

Important that we do not do extra work – method verification will help here.

Need colleagues to understand significance of MU.

MU vital when assessing compliance.

Where do we go with sampling uncertainty?