



EURACHEM SYMPOSIA: SAMPLING UNCERTAINTY AND UNCERTAINTY FOR COMPLIANCE ASSESSMENT

Uncertainty from sampling – discussions within the Codex Alimentarius Commission

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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 covered

Purpose of Codex guidelines on sampling
Main notions of sampling
The selection of sampling plans for single or isolated lots moving in international trade
The selection of sampling plans for a continuous series of lots from a single source
The selection of sampling plans for the inspection by variables of bulk materials: known standard deviation



PRINCIPLES FOR THE ESTABLISHMENT OR SELECTION OF CODEX SAMPLING PROCEDURES

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.



Types of Sampling Plans and Procedures

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 GUIDELINES ON SAMPLING CAC/GL 50-2004



SECTION I. PURPOSE OF CODEX GUIDELINES ON SAMPLING

1.1 PURPOSE

Sampling plans are required which ensure that fair and valid procedures are used when food is being controlled for compliance with a particular Codex commodity standard.

Since numerous, yet often complex, sampling plans are available it is the purpose of these guidelines to help those responsible for sampling to select sampling plans that are appropriate for statistical inspections under specifications laid down by Codex standards. No sampling plan can ensure that every item in a lot conforms.

These sampling plans are nevertheless useful for guaranteeing an acceptable quality level.



The guidelines contain the elementary principles of statistical control at reception, which complete the basic recommendations laid down in the Preamble.



1.3 USERS OF SAMPLING PLANS RECOMMENDED BY THE GUIDELINES

The sampling plans described in these Guidelines may be implemented either by Governmental food control authorities, or by professionals themselves (self-inspection performed by producers and/or traders). In the latter case, these Guidelines enable the governmental authorities to check the appropriateness of the sampling plans implemented by the professionals.

It is recommended that the different parties concerned with sampling come to an agreement on the implementation of the same sampling plan for the respective controls.



1.4 SCOPE OF THE GUIDELINES

The following sampling situations are covered for the control of only homogeneous goods:

- control of percentage of defective items by attributes or by variables, for goods in bulk or in individual items,
- control of a mean content.



These Guidelines do not cover the control of:

- non-homogeneous goods;
- for homogeneous goods, the cases where measurement error is not negligible compared to sampling error (see 2.4), as well as the control of a qualitative characteristic in a bulk material and;
- they do not deal with double, multiple and sequential sampling plans, deemed too complex in the frame of these Guidelines.



CODEX GUIDELINES ON MEASUREMENT UNCERTAINTY (CAC/GL 54-2004)

Introduction

It is important and required by ISO/IEC 17025:1999 that analysts are aware of the uncertainty associated with each analytical result and estimates that uncertainty. The measurement uncertainty may be derived by a number of procedures. Food analysis laboratories are required, for Codex purposes, to be in control, use collaboratively tested or validated methods when available, and verify their application before taking them into routine use. Such Laboratories therefore have available to them a range of analytical data which can be used to estimate their measurement uncertainty.



These guidelines only apply to quantitative analysis.

Most quantitative analytical results take the form of
“ $a \pm 2u$ or $a \pm U$ ”

where “ a ” is the best estimate of the true value of the concentration of the measurand (the analytical result) and “ u ” is the standard uncertainty and “ U ” (equal to $2u$) is the expanded uncertainty. The range “ $a \pm 2u$ ” represents a 95% level of confidence where the true value would be found. The value of “ U ” or “ $2u$ ” is the value which is normally used and reported by analysts and is hereafter referred to as “measurement uncertainty” and may be estimated in a number of different ways.



Terminology

The international definition for Measurement Uncertainty is:
"Parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand"



CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

29th Session, Budapest, Hungary, 10-14 March 2008

GUIDANCE ON MEASUREMENT UNCERTAINTY AND UNCERTAINTY OF SAMPLING

GUIDANCE ON UNCERTAINTY FROM SAMPLING

(Paper CX/MAS 29/9- Add 1.)



INTRODUCTION

PUBLICATION OF SAMPLING UNCERTAINTY

GUIDES SINCE PREVIOUS SESSION of CCMAS

1. EURACHEM/EUROLAB/CITAC/Nordtest *Guide on the Estimation of Measurement Uncertainty Arising from Sampling*
2. Nordtest handbook for sampling planners on sampling quality assurance and uncertainty estimation *Uncertainty from sampling*



SAMPLING IN CODEX

BACKGROUND

METHODS FOR ESTIMATING SAMPLING UNCERTAINTY

The duplicate method



A sampling protocol (detailing, how many samples, how to sample, sample mass etc.) is a prerequisite for all food surveys, assessments etc. The duplicate method requires a second (duplicate) sample to be taken for 10% (or a minimum of 8) of the total number of sampling targets. This second 'duplicate' sample should be taken to represent the ambiguity in interpreting the protocol, what this means is perhaps better explained using the examples.



The duplicate samples are then each subject to independent physical preparation (i.e. they are not combined). Two analytical test portions are drawn from each of the duplicate 'prepared' samples.

The procedures given in the Nordtest Guide are reproduced as Annex. A range of sampling exercises that span from grower level to retail sampling using one of the procedures given in Annex I were described in the CCMAS paper.



RECOMMENDATIONS

It is recommended that the Committee:

- Notes the publication of the EURACHEM/EUROLAB/CITAC/Nordtest Guide on the “Estimation of Measurement Uncertainty Arising from Sampling” and the Nordtest handbook.



- discusses the issue of uncertainty and sampling and decides whether it should develop recommendations in the area in the same way that it already has for [Analytical] Measurement Uncertainty. In particular, it should discuss what is the likely magnitude of sampling uncertainties likely to be encountered in routine compliance assessments.



- discusses whether sampling uncertainty should be taken into account when a lot is assessed for compliance with a Codex specification.
- whether it should prepare Guidance for Codex Committee Committees on sampling uncertainty.



OUTCOME

The Committee recognized that at this stage it was premature to undertake new work but that this question should be kept under consideration and therefore agreed that the Delegation of the United Kingdom, with the assistance of an electronic working group, would revise the discussion paper for consideration by the next session.

Countries now addressing the issue internally.



THREE EXAMPLES FROM THE FOOD SECTOR USING DOUBLE SPLIT DESIGN AND RANGE STATISTICS

Three examples of where the range procedure given in the Nordtest Guide 604 have been applied are given below.

These indicate the problems that may arise when sampling uncertainty is identified.



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 $\text{g } 100\text{g}^{-1}$

Mean: 15.75

Standard deviation of analysis : 0.041

Standard deviation of sampling : 0.219