AOAC EUROPE SECTION

INTERNATIONAL WORKSHOP: "FOODS TO DYE FOR - CONTAMINANTS - SAMPLING, ANALYSIS, LEGAL LIMITS"

SAMPLING AND MEASUREMENT UNCERTAINTY - SHOULD WE BE MORE CERTAIN OF THEIR IMPORTANCE IN FOOD CONTROL?

Roger Wood, Food Standards Agency,
c/o Institute of Food Research,
Norwich Research Park, Colney, Norwich NR4 7UA

Will cover:

- Measurement Uncertainty - The Relationship Between the Analytical Result and the Specification in Legislation

- Sampling Uncertainty

“COMPLIANCE”

The relationship between the final analytical result and the sampling, the measurement uncertainty and the recovery factor used to obtain that result.

Discussions in both EU and Codex

EU REPORT:

THE RELATIONSHIP BETWEEN THE FINAL ANALYTICAL RESULT AND THE SAMPLING, THE MEASUREMENT UNCERTAINTY AND THE RECOVERY FACTOR USED TO OBTAIN THAT RESULT

These factors affect the relationship between the final analytical result and the provisions in legislation

Decisions taken by those responsible for the enforcement of legislation directly affect decisions as to whether a lot is in compliance with that legislation.

SCIENTIFIC CO-OPERATION TASK 9.1

"PREPARATION OF A WORKING DOCUMENT IN SUPPORT OF THE UNIFORM INTERPRETATION OF LEGISLATIVE STANDARDS AND THE LABORATORY QUALITY STANDARDS PRESCRIBED UNDER DIRECTIVE 93/99/EEC"

was initiated to identify differences amongst Member States.

14 participated. Final Report is now published.

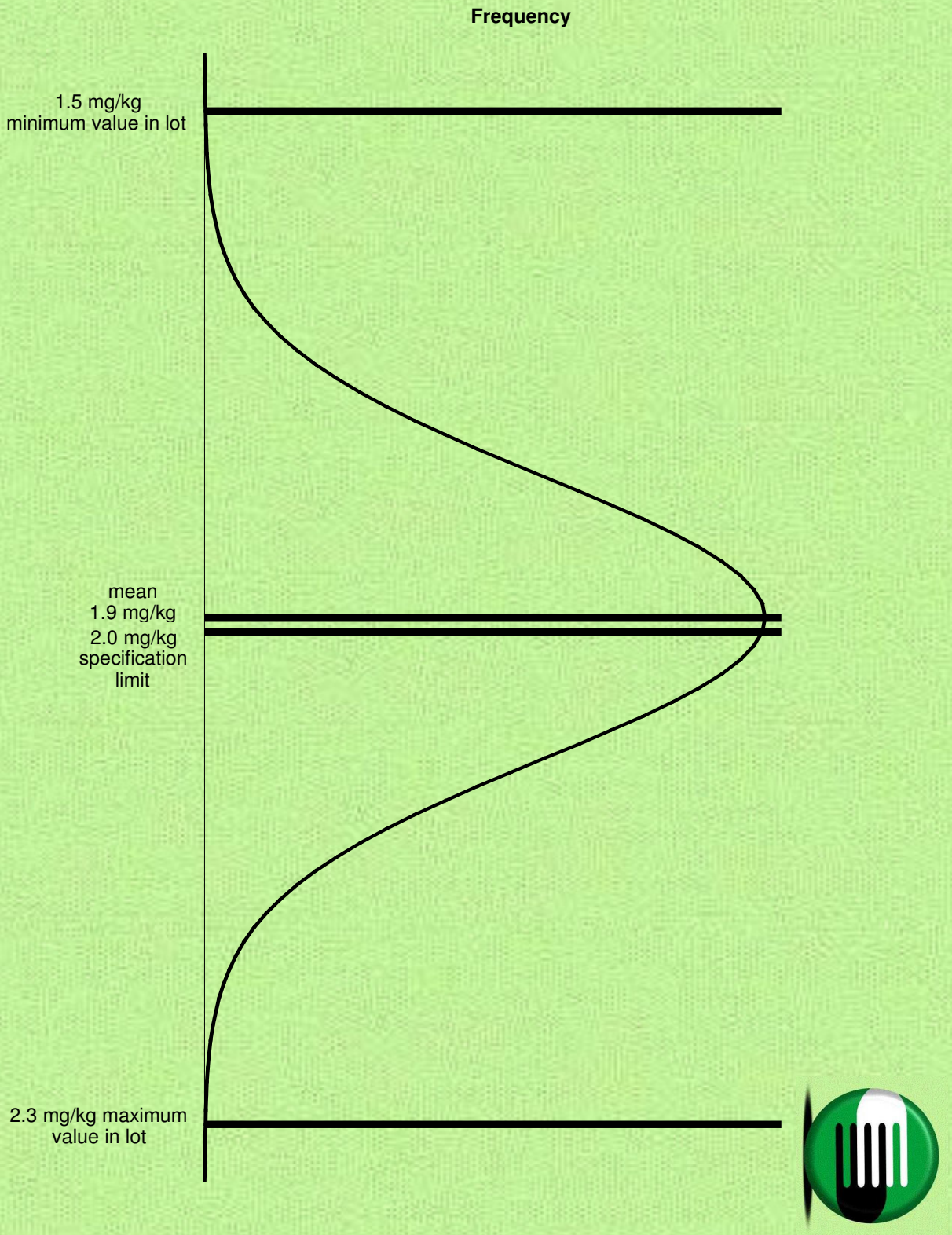
MAJOR ISSUES IDENTIFIED

The basic principles of the sampling procedures used by The Member States, the treatment of analytical variability (normally known as the measurement uncertainty) in the interpretation of an EU specification, and the use of recovery corrections when calculating and reporting analytical results were found to be different,

The effect of different countries taking different approaches for each of the issues identified are described. It must be appreciated that there may be other enforcement issues which have a similar effect.

There was no common interpretation of analytical results across the EU in the food sector so significantly different decisions may be taken after analysis of the "same sample". Material for which there is a statutory limit of, say, 4µg/kg for a contaminant (e.g. total aflatoxins) may be interpreted as containing 3µg/kg on analysis in one country but 8 µg/kg in another. This is because some countries correct analytical results for recovery, others do not; some countries use an "every-item-must-comply" sampling regime, others may use an "average of a lot" regime, some make an allowance for measurement uncertainty, others do not.

It is essential that interpretation of analytical results is similar if there is to be equivalence across the EU; without it there is no uniform interpretation of legislation.


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.

REPORTING OF RESULTS WITH RESPECT TO THEIR MEASUREMENT UNCERTAINTY

All analytical results should be reported in the form "a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "a-U" to "a+U" is the range within which the true value is estimated, with a given probability, to fall. The value of "U", the (expanded) "measurement uncertainty", may be estimated by the analyst in a number of different ways.

REPORTING OF RESULTS BY FOOD CONTROL ANALYSTS

The procedure adopted by some food control analysts is to report samples as containing “not less than “a” – “U”” in situations where the statutory limit is a maximum permissible concentration. Thus, in any enforcement situation the maximum benefit is given to the food producer. This is consistent with the requirement to prove ***beyond reasonable doubt*** that a limit has been exceeded, if the case should come to Court. This does mean that the effective enforcement limit is, in such countries, not identical to the numerical value given in legislation.

Other food analysts may report the value “a” without taking into account any measurement uncertainty considerations when assessing compliance with a specification.

This was found to be the case in the SCOOP Task

Similar considerations identified in Codex Alimentations Commission

Here a section on:

“The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards”

has been approved to be included in Procedural Manual

ISSUES INVOLVED

There are a number of analytical and sampling considerations which prevent the uniform implementation of legislative standards. In particular, different approaches may be taken regarding sampling procedures, the use of measurement uncertainty and recovery corrections.

At present there is no official guidance on how to interpret analytical results across the Codex Community. Significantly different decisions may be taken after analysis of the "same sample". For example some countries use an "every-item-must-comply" sampling regime, others use an "average of a lot" regime, some deduct the measurement uncertainty associated with the result, others do not, some countries correct analytical results for recovery, others do not. This interpretation may also be affected by the number of significant figures included in any commodity specification.

It is essential analytical results are interpreted in the same way if there is to be equivalence across the Codex Community.

It is stressed that this is not an analysis or sampling problem as such but an administrative problem which has been highlighted as the result of recent activities in the analytical sector, most notably the development of International Guidelines on the Use of Recovery Factors when Reporting Analytical Results and various Guides prepared dealing with Measurement Uncertainty.

RECOMMENDATIONS

It is recommended that when a Codex Commodity Committee discusses and agrees on a commodity specification and the analytical methods concerned, it states the following information in the Codex Standard:

1. Sampling Plans

The appropriate sampling plan to control conformity of products with the specification. This should state:

- whether the specification applies to every item in a lot, to the average in a lot or the proportion nonconforming;
- the appropriate acceptable quality level to be used;
- the acceptance conditions of a lot controlled, in relation to the qualitative/quantitative characteristic determined on the sample.

2. Measurement Uncertainty

That an allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification. This requirement may not apply in situations when a direct health hazard is concerned, such as for food pathogens.

3. Recovery

Where relevant and appropriate the analytical results are to be reported on a recovery corrected basis and that the recovery should be quoted in any analytical report. Analytical results are to be expressed on a recovery corrected basis where appropriate and relevant, **and when corrected it has to be so stated.**

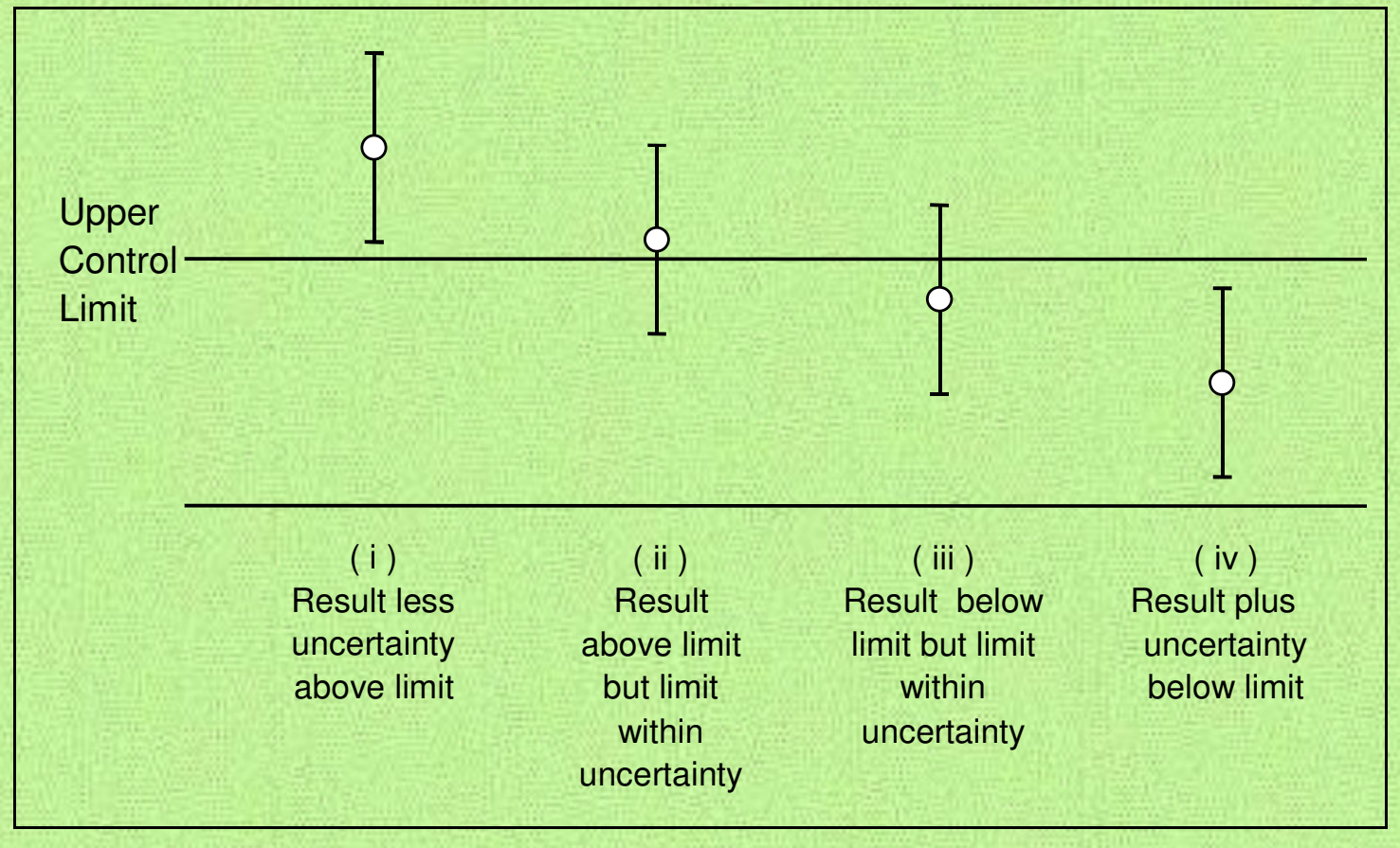
In all cases it has to be stated when the result is corrected for recovery.

If a result has been corrected for recovery, the method by which the recovery was taken into account should be stated. The recovery rate is to be quoted **wherever** possible.

When laying down provisions for standards, it will be necessary to state whether the result obtained by a method used for analysis within conformity checks shall be expressed on an recovery-corrected basis or not.

4. Significant Figures

The units in which the results are to be expressed and the number of significant figures to be included in the reported result.


Upper
Control
Limit
(i)
Result less
uncertainty
above limit
(ii)
Result
above limit
but limit
within
uncertainty
(iii)
Result below
limit but limit
within
uncertainty
(iv)
Result plus
uncertainty
below limit

This means that the legal specification and enforcement limit are different.

This should be appreciated when specification is being set.

EURACHEM General Paper

USE OF UNCERTAINTY INFORMATION IN COMPLIANCE ASSESSMENT

Out for “public comment”. Agrees with the previous approaches discussed, but the terminology is different - guard band etc.

UNCERTAINTY OF SAMPLING

Discussed in the Draft EURACHEM Guide and in Codex

The Eurachem/EUROLAB/CITAC/Nordtest Working Group on Uncertainty from Sampling was formed in September 2003. This Working Group includes representatives from a wide range of disciplines, including those from the food sector. The Eurachem Working Group is currently preparing guidance for the evaluation of uncertainties in measurement arising from the process of sampling.

This guidance will be applicable to all chemical measurements that require the taking of a sample. It will provide guidance on the assessment of the uncertainty of the measurement that is caused by the process of sampling, and any physical preparation of the sample prior to analysis, and how this can be combined with estimates of uncertainty arising from the analytical process.

Once an estimate of uncertainty has been made, we are presented with the question as to whether the estimate is fit for purpose - we are in essence asking ‘is it good enough?’ It may be the case that a reduction in measurement uncertainty is desirable. A reduction can be reached by simply increasing sample mass, increasing the number of analyses and so on. However, it is unrealistic to expect an unrestricted financial budget or time-scale for completion of analysis. Given these external considerations, a decision needs to be made on how much uncertainty can be tolerated.

This document looks firstly at the methods of estimating uncertainty and uses real case studies to exemplify each. The role of measurement uncertainty in the decision making process is also addressed, as is the assessment of fitness for purpose. The second part of this document examines whether it is a good idea to set global fitness for purpose criteria for sampling uncertainty. This document is focussed on measurement processes that result in quantitative data

METHODS FOR ESTIMATING SAMPLING UNCERTAINTY

Sampling theorists consider that sampling error can be minimised by using a correct sampling protocol. Theory of sampling relies on a great deal of prior knowledge of both the sampling target and characteristics are required, e.g. particle-size distribution factors, density, shape factors (Gy, 1979). This methodology was initially applied to bulk particulate materials, and wider application requires a correct interpretation of the theory. Without a good grasp of the principles, adaptation of the theory across the range of commodities covered by Codex will be difficult.

THE DUPLICATE METHOD – GENERAL PRINCIPLES

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.

All test portions are anonymised (so it is unclear which are duplicates) and subsequently analysed in a randomised order.

An analysis of variance is applied to the resultant data to separate out between-target variances, sampling (or within-target) variances and analytical variances.

The inclusion of certified reference materials (CRM) and /or spike samples within the analytical run will allow the systematic effects of analysis to be quantified. This is generally routine in most laboratories. As described, the duplicate method does not permit the estimation of systematic effects from the sampling process. When the duplicate method of uncertainty estimation is utilised, the costs will increase by 10% for sampling and 30% for analysis.

UNCERTAINTY OF SAMPLING

Example 1 – Nitrate concentration in glasshouse lettuce

Mean = 4408 mg kg^{-1}

Analytical uncertainty: 168 mg kg^{-1}

Sampling uncertainty: 319 mg kg^{-1}

Measurement uncertainty: 361 mg kg^{-1}

Example 2 – Infant wet meals (retail survey) (Cadmium)

Mean = 7.575 µg kg^{-1}

Analytical uncertainty: 1.100 µg kg^{-1}

Sampling uncertainty: 1.235 µg kg^{-1}

Measurement uncertainty: 1.654 µg kg^{-1}

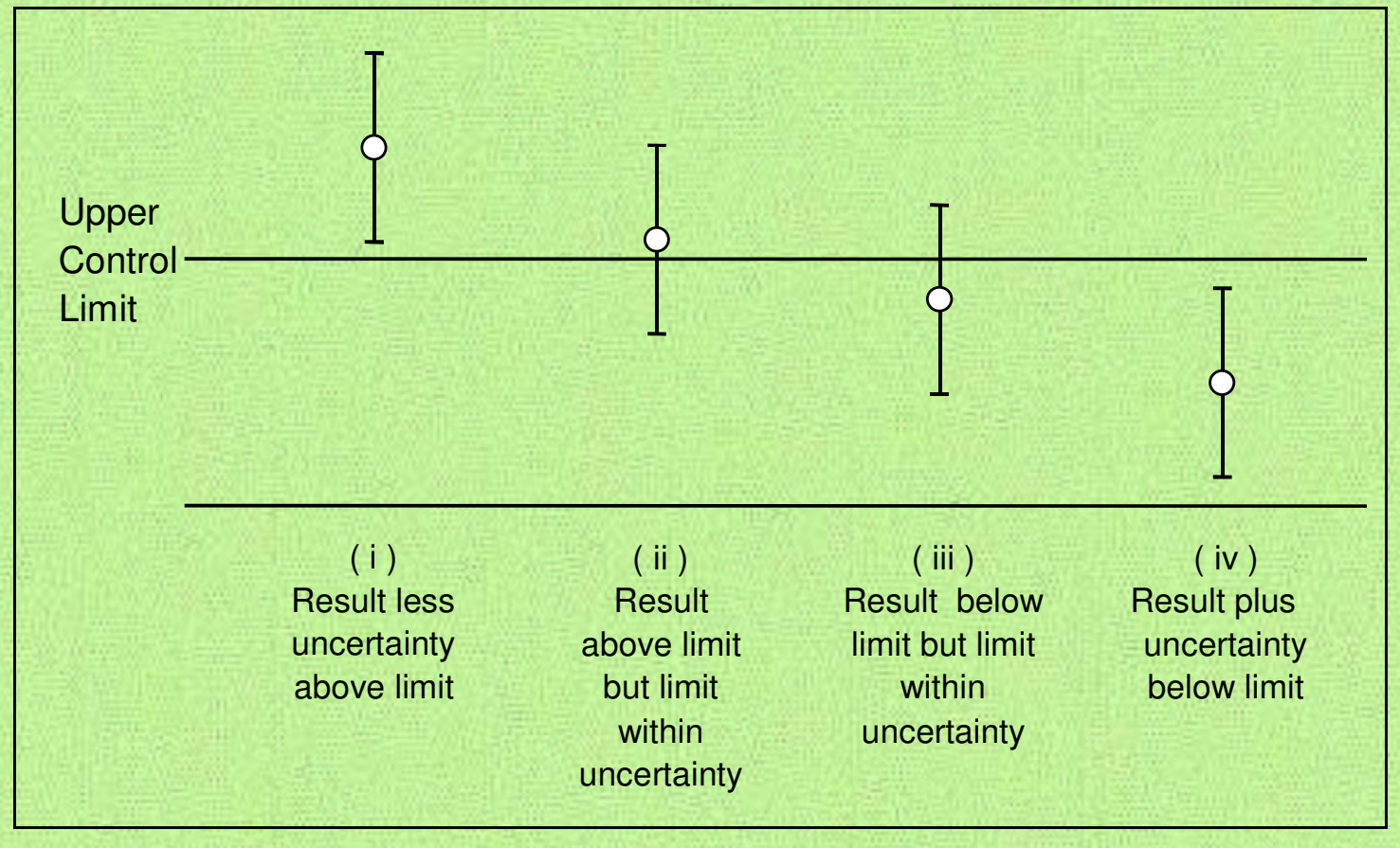
Example 3 – Moisture in wholesale butter

Mean = 15.754 % (m/m)

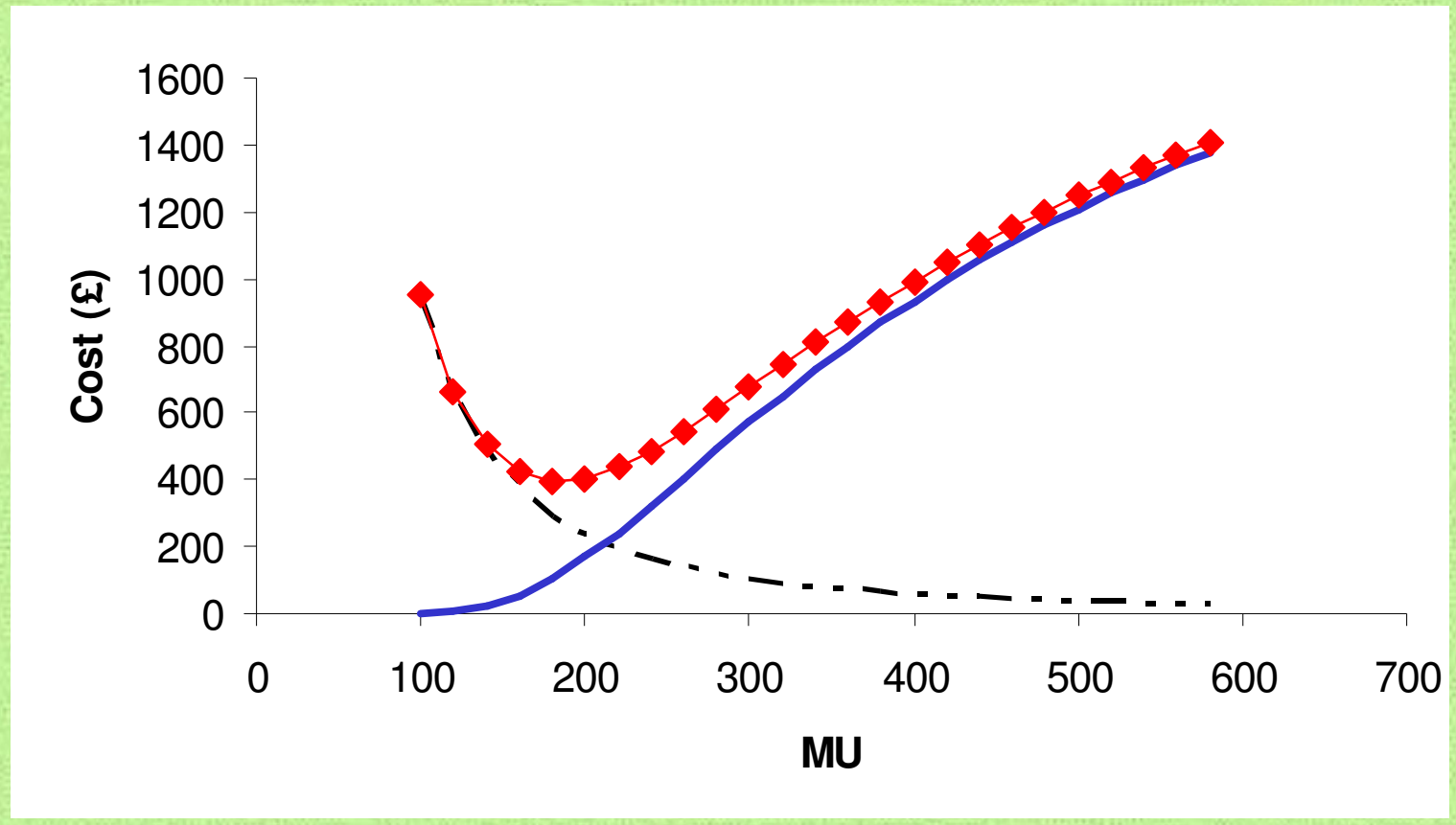
Analytical uncertainty: 0.0421 % (m/m)

Sampling uncertainty: 0.1947 % (m/m)

Measurement uncertainty: 0.1992 % (m/m)


Upper
Control
Limit
(i)
Result less
uncertainty
above limit
(ii)
Result
above limit
but limit
within
uncertainty
(iii)
Result below
limit but limit
within
uncertainty
(iv)
Result plus
uncertainty
below limit

Can also assess the economic consequences of sampling and analytical uncertainties


1600
1400
1200
1000
800
600
400
200
0
Cost (£)
0
100
200
300
400
500
600
700
MU

LOOKING FORWARD

Community Reference Laboratories

National Reference laboratories

Should they be concerned?

SAMPLING PROFICIENCY TEST PROJECT

Analogous to an Analytical Proficiency Test.

But having problems setting up - industry is “afraid” of the situation?

CONCLUSIONS

Needs to understand the use of measurement uncertainty estimations

Uncertainty of sampling will have to be addressed - but at what consequences?