RECENT QUALITY ASSURANCE INITIATIVES FOR THE ANALYSIS LABORATORY – ARE WE ON THE RIGHT PATH?

THEOBALD LECTURE

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Theobald was an interesting character – apparently he marked students' notes of his lectures.

Went from IC to consultancy and then set up this lectureship, to recognise a successful career.

The “quality assurance aspects” of the Food Analysis Laboratory are now well defined as the result of Codex

REQUIREMENTS

3. The following criteria shall be adopted by laboratories involved in the import and export control of foods:

- Compliance with the general criteria for testing laboratories laid down in ISO/IEC Guide 25:1990 “General requirements for the competence of calibration and testing laboratories”; [i.e. effectively accreditation],
- Participation in appropriate proficiency testing schemes for food analysis which conform to the requirements laid down in “The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories”, Pure and Applied Chemistry 65 (1993) 2132-2144; [already adopted for Codex purposes by the CAC at its 21st Session in July 1995]

Whenever available, use methods of analysis which have been validated according to the principles laid availabl05l2

Method Criteria in Codex

- accuracy
- applicability (matrix, concentration range and preference given to 'general' methods)
- detection limit
- determination limit
- precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories), but generated from collaborative trial data rather than measurement uncertainty considerations
- recovery
- selectivity
- sensitivity
- linearity

These requirements target towards accreditation, proficiency testing and method validation.

REGULATION (EC) No 882/2004 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 29 April 2004

on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules.

Article 11

(b) in the absence of the above, with other methods fit for the intended purpose or developed in accordance with scientific protocols

2. Where paragraph 1 does not apply, validation of methods of analysis may take place within a single laboratory according to an internationally accepted protocol.

IUPAC harmonised guidelines for single-laboratory validation of methods of analysis (Michael Thompson, Stephen L R Ellison and Roger Wood, Pure Appl. Chem., 2002, 74(5), 835-355) now accepted in EU and Codex.

3. Wherever possible methods of analysis shall be characterised by the appropriate criteria set out in Annex III.

4. The following implementing measures may

(b) performance criteria, analysis parameters, measurement uncertainty and procedures for the validation of the methods referred to in (a); and

(c) rules on the interpretation of results

6. In particular, they shall ensure that feed and

Article 12

Official laboratories

1. The competent authority shall designate laboratories that may carry out the analysis of samples taken during official controls.

2. However, competent authorities may only designate laboratories that operate and are assessed and accredited in accordance with the following European Standards:

(a) EN ISO/IEC 17025 on “General requirements for the competence of testing and calibration laboratories”;

(b) EN 45002 on “General criteria for the assessment of testing laboratories”;

(c) EN 45003 on “Calibration and testing laboratory accreditation system – General requirements for operation and recognition”,

taking into account criteria for different testing methods laid down in Community feed and food law.

3. The accreditation and assessment of testing laboratories referred to in paragraph 2 may relate to individual tests or groups of tests.

4. The competent authority may cancel the designation referred to in paragraph 1 when the conditions referred to in paragraph 2 are no longer fulfilled.

CHARACTERISATION OF METHODS OF ANALYSIS

1. Methods of analysis should be characterised by the following criteria:

(a) accuracy;

(b) applicability (matrix and concentration range);

(c) limit of detection;

(d) limit of determination;

(e) precision;

(f) repeatability;

(g) reproducibility;

(h) recovery;

(i) selectivity;

(j) sensitivity;

(k) linearity;

(l)

2. The precision values referred to in 1(e) shall either be obtained from a collaborative trial which has been conducted in accordance with an internationally

4. In situations where methods of analysis can only be validated within a single laboratory then they should be validated in accordance with eg IUPAC Harmonised Guidelines, or where performance criteria for analytical methods have been established, be based on criteria compliance tests.

5. Methods of analysis adopted under this Regulation should be edited in the standard layout for methods of analysis recommended by the ISO.

Exactly the same requirements now apply to FSA surveys.

See

GUIDELINES FOR FOOD STANDARDS AGENCY TECHNICAL SURVEYS

On FSA website.

- It is also important to recognise the effect of the introduction of the criteria (performance based) approach for methods of analysis in the food sector and what this means for the analyst.

In particular the analyst must:

- Decide what is an acceptable method.
- Assess individual performance characteristics.
- Consider the effect of the development of an uncertainty function approach to methods of analysis.
- Consider the role of validation of methods within a single laboratory.

Methods of Analysis

Possible approaches to evaluating acceptable methods of analysis

Criteria Approach

The introduction of the criteria approach does mean that thought now has to be given to developing defining and quantifying the specific criteria required in each instance. This is often complex and an alternative approach has also been considered.

Thus two possible approaches to evaluating acceptable methods of analysis are:

- To identify specific performance parameters and assign numeric values to these (the traditional approach)
- To identify a maximum acceptable uncertainty.

Examples

Performance Criteria – Traditional Approach

"Specific methods for the determination of tin contents are not prescribed. Laboratories should use a validated method that fulfils the performance criteria indicated in Table 3*. The validation should ideally include a certified reference material in the collaborative trial test materials.

[* from EU Tin Sampling and Analysis Directive]

Table 3: Performance criteria of methods for tin analyses

Limit of quantification	No more than one 10 mg/kg
Precision	$HORRAT_r$ or $HORRAT_R$ values of less than 1.5 in the validation collaborative trial
Recovery	80% - 105%
Specificity	Free from matrix or spectral interferences

Performance Criteria – Uncertainty Function Approach

However, an uncertainty approach may also be used to assess the suitability of the method of analysis to be used by the laboratory. The laboratory may use a method which will produce results with a maximum standard uncertainty given by the following formula:

$$Uf \quad \sqrt{(CL/2)^2 \quad (0.1C)^2}$$

where: Uf is the maximum standard uncertainty
CL is the detection limit of the method
C is the concentration of interest

Results with an uncertainty less than that stipulated above will be produced by a method which is equivalent to one meeting the performance characteristics given in Table 3.

Measurement Uncertainty

Is still an issue for food analysts

One of the consequences of 17025 Accreditation

REPORT TO THE STANDING COMMITTEE ON THE FOOD CHAIN AND ANIMAL HEALTH ON THE RELATIONSHIP BETWEEN ANALYTICAL RESULTS, THE MEASUREMENT UNCERTAINTY, RECOVERY FACTORS AND THE PROVISION IN EU FOOD AND FEED LEGISLATION

CONSEQUENCES OF REPORTING RESULTS IN DIFFERENT WAYS

There are potential problems with the reporting of results for which there is a legislative specification. This is best explained by example:

Let us assume that there is a specification of 4 µ

Situation a

Here the level reported is below the specification. All countries would take the same view and accept the material.

Situation b

Here the level reported is above the statutory limit but the true value lies in the range 3.4 to 8.6 µg/kg. The level and its uncertainty would be reported.

Here some countries would report the sample as containing not less than 3.4 µg/kg of the analyte and because it is not beyond reasonable doubt that the limit has been exceeded, no action will be taken.

However, other countries may take action on the 6.0 µg/kg result, without taking uncertainty into account. For these countries, the material will be deemed to be non-compliant.

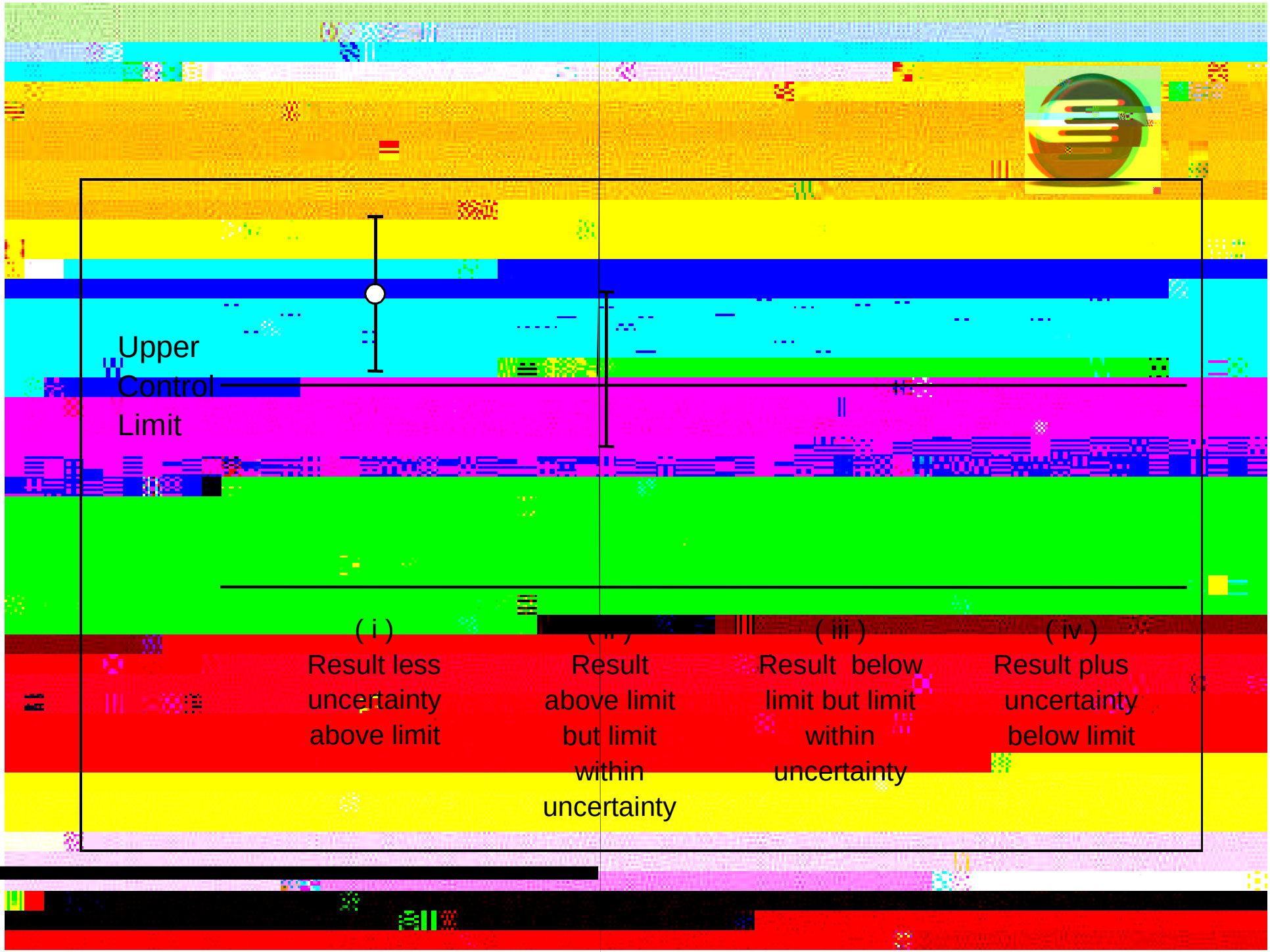
Situation c

Here the level reported is above the specification and the true value lies in the range 5.6 to 14.4 µg/kg. All countries will state that the material is non-compliant with the specification.

Conclusion

In situation ***b***, there is the possibility that different countries will make opposite decisions as to whether the material conforms with the specification. Various projects have shown this to be the situation.

Problem shown diagrammatically:


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

Recent EU Contaminant Regulations

require the deduction of the

Measurement Uncertainty

THE USE OF RECOVERY INFORMATION IN ANALYTICAL MEASUREMENT

A real example may result in the mycotoxin area where there may be a limit of 4µg/kg for total aflatoxin in nuts. Here the following situation may arise:

Country A will analyse a consignment and find a result of 3.5µg/kg total aflatoxin using a method which, in the analytical run, has a recovery of 70%. Country A does not correct for recovery corrections as a matter of policy and so the reported result will be 3.5µg/kg and so the sample will be in compliance with the 4µg/kg limit.

Country B, however, uses recovery corrections as a matter of policy. That country could analyse the “same” sample using the “same” methodology and obtain the “same” analytical result but will report not 3.5 but 5µg/kg on a recovered basis. Here there is the possibility that because

CONCLUSIONS

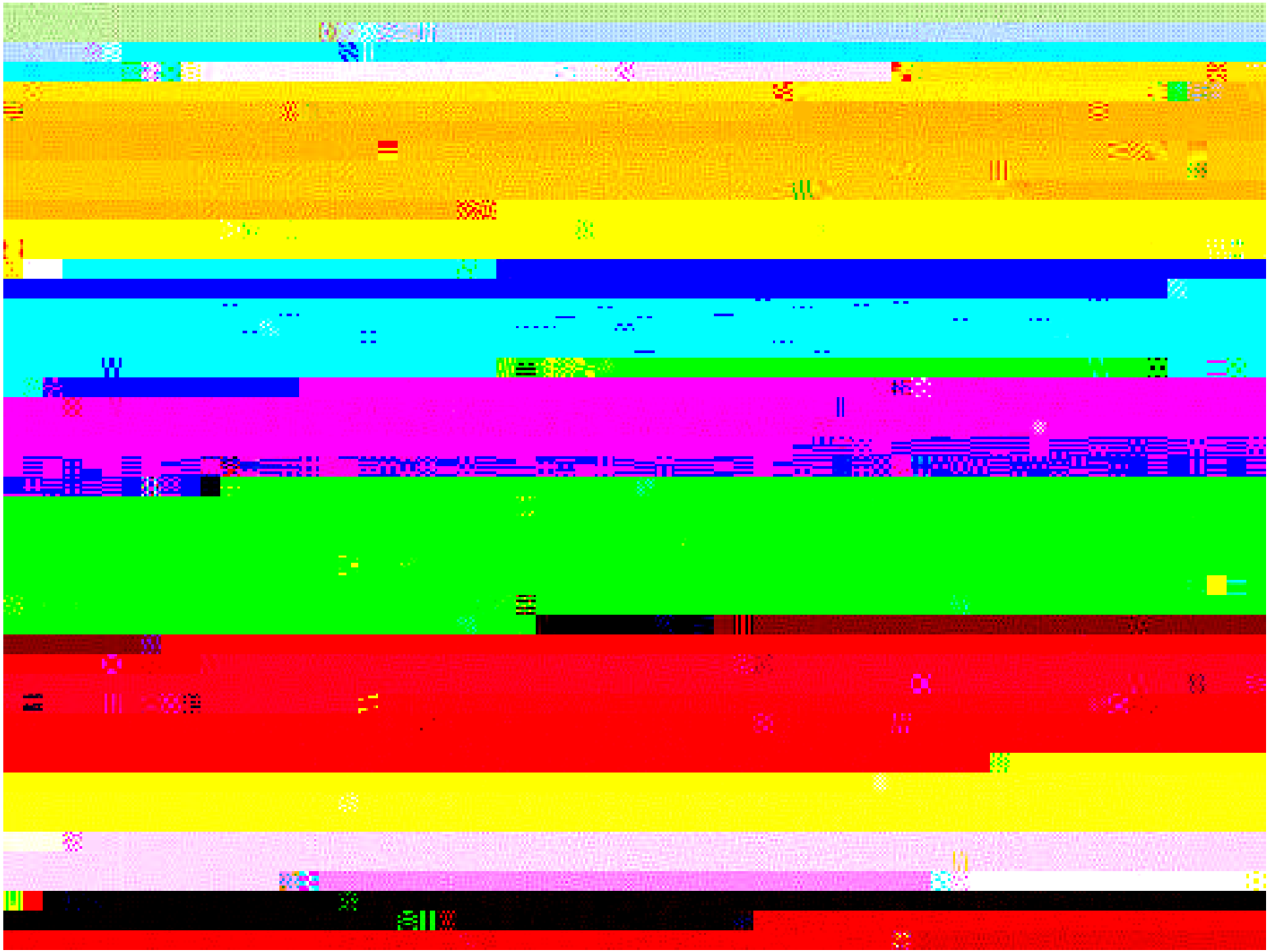
The analyst is increasingly being given more “freedom”,

Extracts from FSA letter of 8 May 2003

QUALITY ASSURANCE IN RESEARCH

– LAUNCH OF JOINT CODE OF PRACTICE FROM DEFRA, FSA, BBSRC AND NERC

This letter is important to all current and potential contractors of Defra, the UK Devolved Administrations and FSA as it will affect you in the future – please read and act



This Code has also been endorsed by the Department of Agriculture and Rural Development for Northern Ireland (DARDNI), the Scottish Executive Environment and Rural Affairs Department (SEERAD) and the Welsh Assembly Government Agriculture and Rural Affairs Department (WAGARAD). It is intended to provide a framework for auditing and research processes and will apply, where possible, to all research funded by Defra, the FSA, DARDNI, SEERAD and WAGARAD and to research funded by BBSRC and NERC in their own Institutes.

In the period June 2003 to May 2004 research providers who have, or who might expect to seek, funding from Defra, the FSA, DARDNI, SEERAD or WAGARAD were asked to consider the Code carefully in relation to their research processes.

In that year, Defra undertook a series of baseline audits with a selection of their current contractors to help establish the current position and to give feedback and guidance on areas for development. Where these are also FSA contractors, Defra will share the information gained with the FSA.

Aspects of the

JOINT CODE OF PRACTICE FOR RESEARCH

Principles behind the Code of Practice

Contractors funded by the above Funding Bodies are expected to be committed to the quality of the research process (QP) in addition to quality of science (QS).

QS addressed the aims of the project, its approaches and the extraction of new knowledge and understanding from the scientific work.

QP underlies the research giving confidence that the processes and procedures used to gather and interpret the results of the research are appropriate, rigorous, repeatable and auditable.

Compliance with the Code of Practice

Contractors are encouraged to discuss with the Funding Body any clauses in the Code that they consider inappropriate or unnecessary in the context of the proposed research project.

Monitoring of compliance with the Code of Practice

Monitoring of compliance with the Code is necessary to ensure:

- Policies and managed processes exist to support compliance with the Code
- That these are being applied in practice.

In the short term, the Funding Bodies can require contractors to conduct planned internal audits although the Funding Bodies reserve the right to obtain evidence that a funded project is carried out to the required standard. The Funding Bodies may also conduct an audit of a Contractor's research system if deemed necessary.

In the longer term it is expected that most research organisations will assure the quality of their research processes by means of a formal system that is audited by an impartial and competent third party against an appropriate internationally recognised standard that is fit for purpose.

Specific requirements in the Code of Practice

1.

5. *Health and Safety*
6. *Handling of samples and materials*
7. *Facilities and equipment*
8. *Documentation of procedures and methods*
9. *Research/work records*

[Here all records must be of sufficient quality to present a complete picture of the work performed, enabling it to be repeated if necessary.]

9. Research/work records (contd)

[The Project Leader must ensure the validity of the work by carrying out regular reviews of the records of each scientist.

The location of all project records, including critical data, must be recorded. They must be retained in a form that ensures their integrity and security, and prevents unauthorised modification, for a period to be agreed by the Funding Body.]

DECLARATION TO ACCOMPANY RESEARCH PROPOSALS

I confirm that I am aware of the requirements of the Joint Code of Practice and, in the proposed

ANNEX – Examples of documentary evidence

Quality Issue	Evidence
1. Responsibilities	Organisation structure showing line management responsibilities.
	List of personnel associated with the project, including sub-contractors.

Extract from letter of 4 February 2004

To: All current contractors for FSA research/survey contracts:

JOINT CODE OF PRACTICE ON QA IN RESEARCH – UPDATE AND ACTIONS

It is important that you read this letter and in particular consider how your current procedures align with the requirements of the Code. From June 2004, all applications for Agency funding will be expected to make a declaration of compliance with the Code's provisions as part of the application.

Activities since May 2003

In the first stage of implementation from May 2003 to May 2004, contractors making applications for funding were requested to sign a declaration acknowledging awareness of the Code's provisions and that they would use best efforts to comply with its principles. Initial activities have therefore focussed on raising and maintaining awareness of the Code.

- The Agency's Research Coordination Unit (RCU) has issued background information on the Code and its implementation to internal Agency project/ programme officers and external programme advisers, together with guidance on maintaining awareness of the Code with Contractors.

The guidance made clear that project officers are not in a position to (or indeed expected to) carry out audits against the Code. However they are in a position to ask questions in the context of discussing progress on projects which can highlight aspects of the Code.

As another part of the implementation phase, Defra has contracted the United Kingdom Accreditation Service (UKAS) to undertake a series of baseline assessments with a selection of its contractors, to establish the current position in relation to the Code's provisions and to give feedback on areas for development.

Defra selected its top 20 contractors in terms of those receiving the most Defra funding, which also includes a significant number of Agency contractors. These cover different types of organisation, including Research Institutes and Agencies, University Departments and independent contractors.

What is coming up – Declaration of compliance and audit

As indicated when the Code was launched, from June 2004, contractors making applications for funding will be expected to make a more definitive declaration of compliance with the Code.