HOKLAS Supplementary Criteria No. 37

'Food' Test Category – Chemical Testing

0 Introduction

(a) This document serves to clarify and supplement the requirements of ISO/IEC 17025:2017 and HKAS PD001 for the accreditation of laboratories performing chemical tests under the test category of 'Food'. This document shall be read in conjunction with ISO/IEC 17025:2017, HKAS PD001 and the relevant HKAS and HOKLAS supplementary criteria documents.

(b) Laboratories should note that fulfilling the requirements in this document might not necessarily meet all the requirements of test standards. Individual test standards may have specific requirements which shall be met when conducting the concerned tests.

1 Scope

(No additional explanation)

2 Normative reference

(No additional explanation)

3 Terms and definition

(No additional explanation)

4 General requirements

(No additional explanation)

5 Structural requirements

(a) The technical management of the laboratory shall include at least a member with in-depth knowledge of and extensive experience in chemical analysis of food. He/she shall be or part of the technical management that is responsible for the technical operation of the laboratory with respect to chemical analysis of food.

6 Resource requirements

6.1. General

(No additional explanation)

6.2. Personnel

(a) For tests involving the use of sophisticated analytical instruments such as AAS, ICP-OES, ICP-MS, GC-MS/MS, LC-MS/MS, etc., testing staff members are normally expected to have completed a post-secondary curriculum in chemistry such as higher diploma or above in chemistry or other relevant technical disciplines. Specific training shall be given to staff members operating specialised equipment such as high-resolution mass spectrometer, GC-ICP/MS, LC-ICP/MS, etc.

(b) A training programme for each testing staff member shall be documented. The programme shall include training on analytical techniques involved as well as test procedures and quality assurance plans.

(c) Approved signatories

(i) Approved signatories shall either have

(1) at least a Bachelor of Science degree, or equivalent, in chemistry or other relevant technical disciplines, with at least 3 years relevant testing experience; or
(2) an Associate Degree or a Higher Diploma, or equivalent, in chemistry or other relevant technical disciplines, with at least 5 years relevant testing experience.

Note: Alternatively, appropriate membership of professional bodies is acceptable. Special consideration may be given to persons without the above qualifications but with extensive experience (at least ten

years) in the test area concerned.

(ii) Irrespective of the person's academic qualifications, the nominee shall have at least six months experience in the areas of testing for which signatory approval is sought.

(iii) In all cases, candidates shall demonstrate to the assessors that his/her technical competence in the test areas under consideration before signatory approval can be granted.

(d) When training is conducted on specific analytical techniques, as opposed to specific test methods, the laboratory should define and document the technique-based competence required for an analyst to perform each test and the additional requirements related to the technique concerned.

(e) Laboratory personnel responsible for visual assessment of colour difference of test samples, including approved signatories for the visual tests concerned, shall not have colour vision problems that may affect the validity of results.

6.3. Facilities and environmental conditions

(a) The laboratory shall provide appropriate environmental conditions and controls necessary for particular tests, including temperature, humidity, freedom from vibration, freedom from airborne and dust-borne contamination, special lighting, etc. Acceptable ranges for the environmental conditions such as temperature and humidity shall be defined and documented. Cases where environmental conditions fall outside the acceptable ranges shall be recorded and the effects, if any, on test results shall be evaluated. Suitable corrective actions shall be taken to rectify the situation as soon as possible.

(b) It should be noted that both temperature and humidity fluctuations may affect the performance of some instruments. Laboratories shall identify instruments that require special environmental conditions. Common examples of this type of instruments include analytical balances, infrared spectrometers, etc. Laboratories shall ensure that these instruments are placed in an environment that ensures their optimum performance and fitness for the intended use. Voltage stabiliser should be used for instruments sensitive to voltage fluctuation.

(c) There shall be effective separation between neighbouring laboratory areas of incompatible activities, especially activities which are prone to interference from other work, or which present particular hazards. When selecting designated areas for special work, laboratories shall consider the previous use of the area and take appropriate measures to ensure that the area is free of contamination. Such laboratory areas include open benches, fume hoods, sample storage area, oven/furnace area, and apparatus soaking/washing area.

(d) Trace analyses are particularly susceptible to contamination. Laboratories shall document procedures and the precautions to be taken to prevent contamination from the environment. Particular attention should be given to the presence of dust in the laboratory environment for trace metal analyses. Precautions shall be taken to avoid the ingress of dust as far as possible. Materials used for furniture, hoods and other fixtures shall not cause contamination, by generation of air-borne particulates, to test samples, calibration standards and other reagents during the entire process of sample preparation and analysis. Good housekeeping is essential to minimise contamination by air-borne particulates.

6.4. Equipment

(a) For chemical analysis, it is essential to avoid contamination of test samples and/or standard solutions by labware. Laboratories shall document procedures for washing labware and, where necessary, for using particular types of labware (glass, PTFE, etc.) for particular tests. Attention should also be given to the possible presence of analytes of interest in commercial detergents. Laboratories shall use, where necessary, different washing, storage and segregation procedures for labware used for different analyses. For example, labware should be soaked in acid bath to remove traces of metals for trace metal analysis. It is also necessary to have a set of labware dedicated for trace metal analysis to prevent possible cross-contamination. Similarly, dedicated labware is required for incompatible tests. Procedures or precautions for labware cleaning, if given in test standards, shall be followed.

(b) The grade of reagents used (including water) shall be stated in the methods together with guidance on precautions to be observed in their preparation or

use. The absence of analytes of interest in reagents, especially acids or solvents, is of particular importance for trace analyses. Laboratories shall ensure that reagents used are suitable for the applications. Critical reagents prepared by the laboratory shall be labelled to identify the substance, strength, solvent (other than water), any special precautions and restrictions of use, date of preparation and period of validity. The person responsible for the preparation of the reagent shall be identifiable from records.

(c) Water is one of the most widely used reagents in chemical analysis. Hence, means to ensure that reagent water is of the required quality is necessary. Performance of the water purification system shall be checked at suitable intervals to ensure that the water produced meets the testing requirements. Records of such checks shall be maintained.

(d) The calibration curve shall be constructed as specified in the test standards. As a general guideline, at least three standards (excluding blank) should be used to establish a linear calibration graph. The standards used shall bracket the entire range of concentration of test samples. The lowest standard shall be at a level at or below the reporting limit of the test method. Criteria for the correlation coefficient of linear calibration graph should be set and implemented. Guidelines given in ISO 11095 'Linear Calibration Using Reference Materials' should be consulted for further details. More calibration standards are required for non-linear calibration functions. Bracketing technique should be used, if appropriate. In case that the calibration procedure is given in the test standard, the procedure shall be followed.

(e) Calibration graphs shall be checked at suitable intervals using calibration standard. The frequency of such check depends on the stability of the equipment and a frequency of around 5 per cent is normally considered as adequate, except otherwise specified by the test standards or the stability of the equipment merits more frequent checking. Acceptance criteria shall be established and shall commensurate with the measurement uncertainty.

6.5. Metrological traceability

(a) HOKLAS Supplementary Criteria No. 2 'All Test Categories – Equipment Calibration and Verification' provides HKAS policy on metrological traceability of measurement results. The requirements relevant to chemical

analysis shall be followed.

(b) Reference materials used for calibration shall provide the necessary metrological traceability. The requirements given in HOKLAS Supplementary Criteria No. 1 'Acceptability of Chemical Reference Materials and Commercial Chemicals Used for the Calibration of Equipment' shall be followed.

6.6. Externally provided products and services

(No additional explanation)

7 Process requirements

7.1. Review of requests, tenders and contracts

(No additional explanation)

7.2. Selection, verification and validation of methods

7.2.1 Selection and verification of methods

(a) Where appropriate, laboratories shall preferably use national and international standard methods, or standard methods published by reputable professional bodies. Laboratories may also use laboratory-developed methods but they have to be validated. In all cases, laboratories shall be satisfied that each particular method is adequate for its intended purpose and that the needs of customers are met. When a piece of legislation, a standard or other published document specify the compliance testing method(s)/procedure(s) to be used, that specified test method(s)/procedure(s) shall be used for compliance testing against the corresponding regulatory or specification limits. It is essential that the limits of reporting are well below the compliance limits and that the method gives reliable results at the limits. Due regard shall also be given to the limitations, concentration ranges and sample matrices specified in the test standards. Laboratories carrying out tests on food should note that Codex Alimentarius (Codex) published test methods and guidance for food testing.

(b) The use of a technique 'more advanced' than that specified in the test

standard may sometimes constitute a deviation. This is particularly true when the analytes are defined by the analytical method. When modifications of this nature are made, laboratories shall assess the possible effects on the test results and, where necessary, obtain supporting evidence to justify the deviation. The deviation shall not affect the test results. HKAS Executive shall be informed of such deviations. The test method shall be described as the particular test standard with modifications. Test reports shall indicate the modifications.

(c) Laboratories using standard methods shall confirm that they can properly perform the methods. Such confirmation is called method verification. Verification is usually done by comparing the method performance data obtained by the laboratories when performing a standard method with those specified in the same method. Laboratories shall demonstrate that the specified limits of detection, selectivity, repeatability, reproducibility, etc., can be obtained. If specified in the standard method, the procedures for determination and/or verification of method performance characteristics shall be followed.

(d) The verification work to be carried out should be appropriate for the purpose of the method, such as identification or quantification of analytes at low and high concentrations. In general, the laboratory shall demonstrate their competence in performing the standard method such that the method performance characteristics, such as trueness, precision, limits of detection/quantitation, measurement uncertainty derived from the verification data, could meet the performance claims in the standard method, for all the matrices and concentrations that the laboratory will apply the method. International guidelines on method verification, such as ‘How to Meet ISO 17025 Requirements for Method Verification’ published by AOAC INTERNATIONAL, provide useful information on method verification.

(e) Confirmation of the identity of organic compounds is necessary for non-selective methods such as gas chromatographic methods employing electron capture detector, flame ionisation detector or liquid chromatographic methods employing refractive index detector or evaporative light scattering detector, etc. Some standard methods may have already specified the confirmation method required and the identity of organic compounds may be considered as confirmed if these instructions are followed. Procedures and criteria for confirmation of organic

compounds, if not specified in test standards, shall be documented. The confirmation method shall be able to reliably confirm the identity of the organic compound at the reporting limit. For mass spectrometric detection, the maximum permitted tolerances for relative ion intensities specified in references relevant to the particular field of application should be followed.

7.2.2 Validation of methods

(a) Standard test methods shall be strictly adhered to and only be used for the intended concentration ranges and sample matrices. If they are used outside their intended concentration ranges or applied to different sample matrices, validation is required. Laboratories shall confirm their competence to perform the test by the use of certified reference materials and participation in proficiency testing programmes, if available.

(b) Non-standard methods shall be validated and authorised before use. The validated non-standard methods shall be documented and the documentation shall include the scope of application, performance characteristics, quality control plans and calibration procedure. Reference to ISO 78-2 ‘Chemistry - Layouts for Standards - Part 2: Methods of Chemical Analysis’ may be useful.

(c) For chemical tests, some of the method performance characteristics are of particular importance. These include, for example, limits of detection/quantitation, precision and bias, applicable concentration ranges and sample matrices. It is thus important that laboratory-developed methods should be validated against, amongst others, these characteristics. Laboratories shall define and explain how the limits of detection/quantitation and reporting, if applicable, are derived. These procedures shall be in line with guidelines given by reputable professional bodies and the limits shall not give an unrealistic impression of the method’s capability. Reporting limits shall be set at a level at which quantitative results may be obtained with a specified degree of confidence. Limits of detection/quantitation and reporting shall be suitably verified.

(d) The following tables provide recommendations for establishing numeric values for applicable range, limit of detection, limit of quantification and recovery of test methods.

Minimum Applicable Range	For ML ≥ 0.1 mg/kg, [ML – 3 S_R, ML + 3 S_R] For ML < 0.1 mg/kg, [ML – 2 S_R, ML + 2 S_R]
Limit of Detection (LOD)	For ML ≥ 0.1 mg/kg, LOD ≤ ML • 1/10 For ML < 0.1 mg/kg, LOD ≤ ML • 1/5
Limit of Quantification (LOQ)	For ML ≥ 0.1 mg/kg, LOQ ≤ ML • 1/5 For ML < 0.1 mg/kg, LOQ ≤ ML • 2/5

Note: ML: Specified maximum and/or minimum level. The minimum applicable range of the method depends on the specified level and can either be expressed in terms of the reproducibility standard deviation (S_R), calculated from the Horwitz/Thompson equation, or in terms of LOD and LOQ.

	Unit	Recovery (%)
Recovery (R)	100% (100 g/100 g)	98 – 102
	≥ 10 % (10 g/100 g)	98 – 102
	≥ 1 % (1 g/100 g)	97 – 103
	≥ 0.1 % (1 mg/g)	95 – 105
	100 mg/kg	90 – 107
	10 mg/kg	80 – 110
	1 mg/kg	80 – 110
	100 µg/kg	80 – 110
	10 µg/kg	60 – 115
	1 µg/kg	40 – 120

Note: Other guidelines are available for expected recovery ranges in specific areas of analysis. In cases where recoveries have been shown to be a function of the matrix other specified requirements may be applied.

(e) Examples of guidelines on method validation are IUPAC Technical Report ‘Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis’, EURACHEM Guide ‘The Fitness for Purpose of Analytical Methods: A Laboratory Guide to Method Validation and Related Topics’, ISO 11843-2 ‘Capability of Detection – Part 2: Methodology in the Linear Calibration Case’ and AOAC INTERNATIONAL ‘AOAC Peer-verified Methods Program - Manual on Policies and Procedures’, ‘Commission Implementing Regulation (EU) 2021/808’ and ‘Analytical Quality Control and Method Validation procedures for Pesticide Residues Analysis in Food

and Feed'.

(f) Method bias shall be assessed using matrix certified reference materials (CRMs), if available. The CRMs used shall be of the same matrices as the intended sample matrices. The levels of the analytes shall also be within the ranges of applicability of the method. The procedure given by ISO Guide 33 'Reference materials - Good practice in using reference materials' should be used to assess the trueness of the test method. If suitable matrix CRMs are not available, recovery studies or comparisons with standard reference methods shall be carried out. The recovery studies should be carried out by spiking the analyte into matrix blank or sample blank. The variety of matrices used for method validation should be representative to serve the intended purpose of the method. Additional guideline is given in IUPAC Technical Report 'Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement'.

(g) Participation in proficiency testing (PT) activities is an external means of method validation. It provides independent evidence that the test results obtained by the proposed method are comparable to those obtained by the other laboratories. Where appropriate, method bias shall be assessed by participating in PT activities.

(h) For food analysis, the method validation required depends very much on the analytes of interest and the matrices. Common food matrices include those rich in protein, carbohydrate, oil, dietary fibre, etc. Validation on food composed mainly of water may also be relevant in some cases. If a method is to be accredited under 'general foodstuffs', satisfactory validation data shall be obtained for at least five different common food matrices (protein, carbohydrate, oil, dietary fibre and water), and at least three food types representative of each food matrix. The range of matrices shall be in line with those listed in relevant regulations. Due consideration shall also be taken for the food matrices with potential interferences e.g. high chloride effect on the ICP-MS determination. The test procedure shall document the food matrices used in the validation studies. During routine analysis of food samples, spike recovery shall be performed on food types previously not encountered in method validation. Laboratories should note that even if their test methods have been validated in accordance with the above requirements, and their methods are described as applicable to 'general foodstuffs', it does not however mean that their test methods are applicable to all food. Laboratories shall assess and determine the applicability of

their methods to the food samples received.

(i) The acceptability of method validation shall be determined based on the intended use of the test methods. Recommendations given by international organisations such as AOAC INTERNATIONAL, Codex Alimentarius and Food and Agriculture Organization (FAO), etc., shall be followed where relevant.

(j) Only test methods that have been validated to have a false compliant rate of no more than 5% at the level of interest shall be used for screening purposes. In the case of a suspected non-compliant result, the result shall be confirmed by a confirmatory method. The suitability of an acceptable false compliant rate shall be assessed against the purpose of the tests and a more stringent false compliant rate may be required in some cases.

(k) To validate qualitative or semi-quantitative screening methods for screening analyte(s) against regulatory/specification limit(s), laboratories may refer to European Union 'Guidelines for the validation of screening methods for residues of veterinary medicines (initial validation and transfer)'.

(l) For qualitative binary screening methods that give two possible outcomes of 'target compound(s) detected' or 'target compound(s) not detected', useful guidance for the method validation could be found in 'Guidelines for Validation of Qualitative Binary Chemistry Methods' published by AOAC INTERNATIONAL. Approaches on statistical treatment of the results of method validation are described in 'Probability of Detection (POD) as a Statistical Model for the Validation of Qualitative Methods'.

(m) The performance of a validated method may change due to many reasons. It is therefore necessary to review the performance characteristics of test methods regularly at suitable intervals and perform revalidation, if necessary. Such reviews may also be required when the performance of the method is affected by changes, such as changes in equipment or environmental conditions, etc.

(n) For the determination of pesticide and veterinary drug residues in food, a laboratory may wish to modify an existing accredited test procedure such that the modified test procedure is still considered as covered by its accreditation scope. In such case, the requirements as given in the Appendix apply.

7.3. Sampling

(a) Sampling from sample lot or site is not covered by this document. Customers taking their own samples should be made aware of proper storage, sampling and transportation procedures. Customers shall be made aware of the fact that the test results only relate to the sample as submitted.

(b) Laboratories should never assume that a sample is homogeneous, even when it appears to be. Where a sample is clearly in two or more physical phases, the distribution of the analyte may vary within each phase. It may be appropriate to separate the phases and treat them as separate samples. Laboratories shall have documented procedures for taking test portions from laboratory samples and shall have measures to ensure that the test portion is representative of the sample. Equipment used for subsampling, packaging, sample extraction, etc. shall be selected in order to avoid unintended changes to the nature of the sample which may influence the final results. Preparation of laboratory samples and test portions, if not specified in test standards, should be based on national or international standards or regulatory guidelines specific to the tested samples. If necessary, customer's clarification should be sought.

7.4. Handling of test or calibration items

(a) Laboratories shall examine and record the condition and appearance of the samples upon receipt. Items to be checked should include, where appropriate, number, volume or amount of sample, temperature, colour, etc.

(b) Test samples shall be suitably stored as soon as practicable upon receipt. Laboratories shall define the storage conditions for different types of samples, particularly for perishable samples. Maximum holding time shall be set for samples of which the analytes to be determined may be affected on prolonged storage. Tests of such samples shall be performed within the set time limits.

(c) It is essential that sub-samples represent the original samples and that their identities are maintained at all times. Attention should be paid to possible contamination of samples by metals or plasticisers leached from containers or stoppers into the sub-samples. In choosing containers for sub-samples,

the properties of the analyte of interest should be taken into account.

(d) Access to the sample storage shall be controlled and only authorised persons shall have access to the sample storage. For litigation samples, procedures shall be in place both to ensure and to demonstrate that samples are secured and their integrity is maintained.

(e) Laboratory sample is the sample as received from the customer by the laboratory. Test sample is prepared from the laboratory sample and test portions are drawn from it for analysis. Test sample shall be representative of the laboratory sample and homogenised so that the uncertainty arising from taking of test portions is not significant. If necessary and appropriate, parts (e.g. adhering soil, bones, etc) that are not intended to be analysed shall be removed.

(f) If possible, the whole laboratory sample as received from the customers should be homogenised and test portions drawn from the homogenised laboratory sample. If the laboratory sample received is too large (for example, greater than 2 kg), sub-sampling may be required. The primary objective of sub-sampling is to obtain a test sample of suitable size which is representative of the laboratory sample as received. If random sub-sampling is used, adequate precautions shall be taken to ensure that each item has equal chance of being selected and each item is accessible to the sub-sampling process.

(g) If the number of items received is small, it may not be appropriate to perform tests on a test sample prepared from only some of the items. It may be necessary to use all the items received in order to obtain a representative test sample. If the size of each item of the laboratory sample is large, it may not be appropriate to directly select a number of items from the laboratory sample. It may be necessary firstly to reduce the size of each and every item before dividing the laboratory sample. The general principle is reduction in size first, then thoroughly mixed the resulting sample before dividing it.

(h) If the number of items is large, the laboratory should choose a reasonable number of items to be taken. The laboratory should be aware that the amount of sample needed depends on the size of the individual items. It may be necessary to have more than one stage of sample preparation. Each stage may consist of firstly reduction in size, then mixing and finally

sample division. Common procedures for sample division include coning and quartering.

(i) In view of the diverse types of samples, it may not be possible to document the sub-sampling procedure for each type of possible sample. However, the sub-sampling process shall address the factors to be controlled to ensure the validity of the test results and a protocol describing the general principles and requirements shall be documented. Laboratories shall record the data and operation relating to sub-sampling that forms part of the testing undertaken including, if relevant, the statistics the procedures are based upon.

(j) Where it is necessary for the proper interpretation of test results, the test reports shall include information on the sub-sampling plan and procedure.

7.5. Technical records

(No additional explanation)

7.6. Evaluation of measurement uncertainty

(a) HKAS Executive accepts approaches which are published by reputable professional bodies or standard writing bodies for evaluating measurement uncertainty of test methods. The measurement uncertainty obtained shall be in line with the definition given by JCGM 200 ‘International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM)’ and shall include all major components of uncertainty in its evaluation. ‘Quantifying Uncertainty in Analytical Measurement’ published by EURACHEM/CITAC and ‘VAM Project 3.2.1 Development and Harmonisation of Measurement Uncertainty Principles, Part (d): Protocol for uncertainty evaluation from validation data’ published by LGC, UK, provide useful guidance on evaluation of measurement uncertainty in chemical measurements.

(b) In general, the degree of rigor relates to the level of risk. To properly evaluate safety, substantial property risk or financial risk, or for litigation purpose, a relatively rigorous uncertainty evaluation is required for the associated tests.

(c) Where sampling (or sub-sampling) is to be treated as part of the test, the

uncertainty arising from such sampling shall be considered by the laboratory. In other words, it is necessary to analyse the representativeness of the sub-sample (i.e. test portion) as part of the measurement uncertainty evaluation. EURACHEM/CITAC Guide 'Measurement uncertainty arising from sampling: A guide to methods and approaches' produced jointly by EURACHEM, EUROLAB, CITAC, Nordtest and the RSC Analytical Methods Committee may be followed.

(d) The uncertainty of physical measurements, the purity of calibration reference materials and their uncertainties, the uncertainties associated with recovery (bias) trials, as well as precision data, where applicable, shall be considered in the evaluation of measurement uncertainty.

(e) Where professional judgement has to be used for significant sources, it shall be based on objective evidence or previous experience. Evaluation of measurement uncertainty containing significant sources evaluated by professional judgement shall not be used for applications demanding the most rigorous evaluation of uncertainty.

Notes:

(1) Measurement uncertainty may be evaluated by rigorously considering individual sources, combined with mathematical combination to produce a measurement uncertainty. This approach is often considered appropriate for more critical work, including for the characterisation of reference materials.

(2) Another approach to evaluate measurement uncertainty is based on proficiency testing or interlaboratory studies, quality control and method verification/validation data, taking into consideration additional uncertainty sources. Additional sources that need to be considered may include sample homogeneity and stability, calibration/reference material, bias/recovery, equipment uncertainty (where only one item of equipment was used in obtaining the precision data). For evaluating measurement uncertainty of methods and laboratory bias from proficiency testing data, reference such as EUROLAB Technical Report 'Measurement uncertainty revisited: Alternative approaches to uncertainty evaluation' may be useful.

(3) In standard methods where method performance characteristics such as repeatability standard deviation and reproducibility standard deviation

determined in accordance with ISO 5725-2 are provided, the information may be adopted for uncertainty evaluation as per ISO 21748 'Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty evaluation'.

7.7. Ensuring the validity of results

(a) Laboratories shall establish and implement quality control plans to ensure and demonstrate that the measurement process is in-control and test results generated are valid and reliable. Common quality control procedures include the analysis of blanks, duplicates, spikes, and controls. The plans shall include frequency of performing quality control samples, their acceptance criteria and actions to be taken in cases of acceptance criteria not being met. Laboratories shall document their quality control plans and procedures for each test method and sample matrix.

(b) The quality control plans and procedures, including acceptance criteria whenever given in the relevant test standards shall be followed. In the absence of such plans, the following shall be followed where applicable.

(i) Blank
Method blank shall be performed at a minimum frequency of one per preparation batch of samples or one per twenty samples, whichever is more frequent. A method blank should consist of all reagents, in the same amounts as the test samples, that are in contact with or added to a sample during the entire analytical procedure. Method blank shall be processed through the entire analytical procedure simultaneously with other test samples within the same preparation batch. Values of method blank above the acceptance limit indicate possible contamination of the batch of samples analysed simultaneously with the method blank.

(ii) Laboratory control sample (LCS)
LCS shall be analysed at a minimum frequency of one per each batch of samples or one per twenty samples ~~or~~, whichever is more frequent. LCS should be prepared in a matrix and at a level that are normally encountered.

(iii) Spike
Matrix spike shall be performed at a minimum frequency of one per

batch of samples or type of matrix or twenty samples, whichever is more frequent. Spiking shall be done before the sample is analysed and should be at the concentration of the analyte present, or the concentration of the mid-range of the calibration curve, or other relevant concentrations. The amount of spike added shall not alter the matrix of the sample significantly. The spike and the calibration standards used in the same run should be prepared from different stock solutions or by different analysts, if possible.

(iv) Duplicate
Duplicate samples (or duplicate spike/LCS, if applicable) shall be analysed at a minimum frequency of one per batch of samples or type of matrix or twenty samples, whichever is more frequent.

(c) Control charts shall be used where appropriate to monitor the performance of the laboratory. Control and warning limits of such charts shall be based on statistical principles. Laboratories shall monitor trends indicated in the control charts. Recommendations given in ISO 5725-6 'Accuracy (trueness and precision) of Measurement Method and Results – Part 6: Use in Practice of Accuracy Values' and IUPAC Technical Report 'Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Laboratories', ISO 7870-2 'Control Charts – Part 2: Shewhart control charts', ISO 7870-4 'Control Charts – Part 4: Cumulative sum charts', ISO 7870-6 'Control Charts – Part 6: EWMA control charts', may be followed, if appropriate.

(d) Proficiency testing (PT) activities

(i) Laboratories shall establish schedules for verifying their performance by analysing matrix CRMs, where available. The laboratory shall also participate in appropriate PT activities for each area of technical competence, as defined by a minimum of one measurement technique, parameter and matrix which are related (please refer to Appendix C of ILAC-P9:01/2024 for more details).

(ii) The frequency of participation in PT activities shall commensurate with the outcome of the laboratory's risk assessment and shall be minimum once per year for each area of technical competence. If significant change is introduced to a verified/validated method, the performance of the method shall be demonstrated by participation in

PT activities.

7.8. Reporting of results

(a) A description of the samples as received shall normally be given in test reports. The description shall include, where relevant to the interpretation of test results, a description of the number/set, appearance and volume/amount of samples, type of container and condition when received. Any deviation from the test standard requirements or abnormality shall be reported.

(b) When test results are below the reporting limits, an indication of the reporting limits shall be given in test reports.

(c) If result to be reported is a numerical value, policy and instructions shall be given on the required number of significant figures and rounding of numbers.

(d) Other information necessary for the proper interpretation of the test results (e.g. quality control results, relevant information provided by the customers, measurement uncertainty, etc.) shall be reported. Qualifying statements on test results shall be given, if necessary.

(e) The sample preparation procedure shall be given if it is required for the proper interpretation of test results. For solid samples, the weight basis on which test results are calculated (e.g. dried basis, as received basis or wet basis) shall be given. Dry to wet weight ratios of samples shall be reported, where necessary.

(f) Some test standards require the reporting of additional information. In these cases, the test reports shall include all the information required by the test standards.

(g) In determining the decision rule to be applied when stating conformity with a legislation, specification or standard, international guideline, such as ILAC-G8 ‘Guidelines on Decision Rules and Statements of Conformity’ and EURACHEM/CITAC Guide ‘Use of uncertainty information in compliance assessment’ and EUROLAB Technical Report ‘Decision rules applied to conformity assessment’ may be followed.

7.9. Complaints

(No additional explanation)

7.10. Nonconforming work

(No additional explanation)

7.11. Control of data and information management

(No additional explanation)

8 Management system requirements

(No additional explanation)

Annex
(Informative)

Bibliography

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3 ISO Guide 33 *Reference materials – Good practice in using reference materials*

4 EURACHEM Guide *The Fitness for Purpose of Analytical Methods: A Laboratory Guide to Method Validation and Related Topics*

5 ISO 11843-2 *Capability of Detection – Part 2: Methodology in the Linear Calibration Case*

6 AOAC INTERNATIONAL *AOAC Peer-verified Methods Program – Manual on Policies and Procedures*

7 ISO 11095 *Linear Calibration using Reference Materials*

8 EURACHEM/CITAC Guide CG4 *Quantifying Uncertainty in Analytical Measurement*

9 EURACHEM/CITAC Guide *Measurement uncertainty arising from sampling: A guide to methods and approaches*

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22 AOAC International Stakeholder Panel on Alternative Methods *Guidelines for Validation of Qualitative Binary Chemistry Methods*

23 Community Reference Laboratories Residues (CRLs) 20/1/2010 *Guidelines for the Validation of Screening Methods for Residues of Veterinary Medicines (Initial Validation and Transfer)*

24 The Codex Alimentarius (CODEX), http://www.codexalimentarius.net

25 Official Journal of the European Communities: 2002/657/EC

26 Official Journal of the European Union: Commission Implementing Regulation (EU) 2021/808

27 EU Reference Laboratories for Residues of Pesticides: *Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed*

28 ILAC-G8: *Guidelines on Decision Rules and Statements of Conformity*

29 ILAC-G18: *Guideline for the Formulation of Scopes of Accreditation for Laboratories*

30 ILAC-P9: *ILAC Policy for Proficiency Testing and/or Interlaboratory comparisons other than Proficiency Testing*

31 ISO 7870-2 *Control charts – Part 2: Shewhart control charts*

32 ISO 7870-4 *Control charts – Part 4: Cumulative sum charts*

33 ISO 7870-6 *Control charts – Part 6: EWMA control charts*

Remark: For dated references in the whole Annex, only the edition cited applies. For undated references cited, the latest edition (including any amendments) applies.

Appendix

Requirements for laboratories making modifications to an accredited test procedure for the determination of pesticide and veterinary residues in food

(i) The bounds within which the laboratory may modify its test procedures shall be clearly defined and approved by the HKAS Executive, and normally, a laboratory may modify its accredited test procedures to (a) include additional analytes which are of the same or similar chemical nature (such as pesticides within the same class e.g. organophosphorus, organochlorine) to those already within its scope of accreditation; (b) include additional sample matrices; (c) change the method performance characteristics for a given sample matrix and a given parameter e.g. modification of measurement range and uncertainty; and (d) include additional technically equivalent procedures which adopt analytical techniques or measurement principles already covered by accreditation.

(ii) Any modifications shall not involve new analytical technique or measurement principle of testing not previously covered under the scope of accreditation of the laboratory in the particular sub-test area concerned, i.e. determination of pesticide residues in food or determination of veterinary drug residues in food.

(iii) The laboratory shall have demonstrated good system maturity and meet the accreditation criteria for Monitoring Plan B or C (as given in HKAS Supplementary Criteria No. 4) in the particular sub-test area concerned, i.e. determination of pesticide residues in food or determination of veterinary drug residues in food.

(iv) The laboratory shall demonstrate technical competence by obtaining satisfactory results in the latest two of any relevant proficiency test programmes or inter-laboratory comparisons participated by the laboratory within the previous two years using the accredited test procedures.

(v) The laboratory shall have gone through at least three assessments for scope extension in the particular sub-test area concerned with no significant nonconformity identified.

(vi) The laboratory shall have been accredited to perform the determination of at least

20% of the analytes anticipated to be covered by the test procedure.

(vii) The staff who are responsible for the development and modification of test procedures shall have the sufficient technical understanding of the test and the technology used. They shall be able to judge the suitability of the test and the validity of the results obtained. They shall be approved signatories in the test concerned and have at least 1 year of experience in the sub-test area under consideration. HKAS Executive will specifically assess the competence of the staff who are authorised to undertake method development and modification during assessments, taking into consideration factors such as the staff's (a) formal education and training received; (b) experience within the field; (c) participation in research or development projects; (d) participation in standardisation committees; and (e) participation in scientific or authoritative committees.

(viii) The process for developing, validating and authorising modified test procedures shall be controlled and documented. The process shall be reviewed at suitable intervals for adequacy and the related activities shall be monitored by incorporation into the laboratory's internal audit programme.

(ix) The laboratory shall maintain a record system that can demonstrate how a test procedure was modified, validated and accepted, the justification for any modification, and who was responsible for each key activity. The information recorded shall be sufficient to allow audits to clearly follow the events leading to the introduction of each modified test procedure.

(x) The laboratory shall demonstrate their technical competence to validate modified procedures in accordance with Cl. 7.2.2 of ISO/IEC 17025:2017 as well as Cl. 7.2.2 of this document.

(xi) The laboratory shall ensure that modified procedures have been fully validated before they are introduced in its scope of accreditation.

(xii) The laboratory shall implement sufficient quality control measures to ensure the validity of the test results obtained from the modified procedures.

(xiii) If nonconforming testing work is identified in association with the use of any modified procedures, the work shall be handled in accordance with Cl. 7.10 of ISO/IEC 17025:2017, and in this connection, if any invalid results are suspected or found to have been reported to customers, the laboratory shall report the matter to HKAS Executive immediately.

(xiv) The laboratory shall notify HKAS Executive of any newly modified test procedures for incorporation into its scope of accreditation by submitting the modified procedures, the proposed scope and also the duly completed HKAS 009 form within 10 working days from the effective date of the modified procedures to HKAS Executive for review.

(xv) The laboratory shall keep an updated scope of the tests the laboratory accredited to perform in the sub-test area(s) concerned, including any modified test procedures, the associated analytes and matrices.

(xvi) The laboratory shall submit in full the validation report with relevant raw data records, uncertainties and other pertinent information as appropriate e.g. staff training records, for any newly modified test procedures since the last reassessment visit for review by HKAS Executive upon request.

(xvii) HKAS Executive will closely monitor the performance of the laboratory, e.g. through unannounced or scheduled visits to the laboratory, matters arising as per (xiii) above, etc., and may amend or delete any items proposed by laboratory for inclusion into its scope of accreditation or terminate the practice of the laboratory under Cl. 7.2.2(n) of this document at its discretion.