# HOKLAS Supplementary Criteria No. 12

## "Environmental Testing" Test Category – Sampling / Testing of Ambient Air for Organic Compounds

### 1 Introduction

1.1 This document supplements HKAS 002 and HOKLAS 003 and provides specific guidance on HOKLAS accreditation of laboratories performing sampling and testing of ambient air for organic compounds. For aspects not covered in this document, the principles stipulated in HKAS 002 and HOKLAS 003 shall apply.

1.2 Only permanent laboratories with at least 2 years of practical experience in the captioned area are eligible to apply for accreditation in testing of ambient air for organic compounds.

1.3 Only permanent laboratories accredited for testing of ambient air for organic compounds can apply for extension of scope to cover site sampling / testing in this area. Organisations accredited for both testing at a permanent laboratory and the related site sampling / testing may, if they wish, undertake either one of the two activities separately and issue HOKLAS endorsed sampling reports or test reports as appropriate.

1.4 Site testing includes testing performed at a site laboratory, or testing performed in-situ (e.g. using potable GC/MS for direct measurement of test parameters).

1.5 Sampling, in this context, means sampling activities carried out on site in accordance with a pre-determined sampling plan and a set of documented and well-defined procedures to obtain representative air samples for subsequent testing.

1.6 The scope of accreditation for sampling will include only operations carried out in accordance with a set of documented and well-defined procedures. Accreditation will not be granted for the design of sampling programmes (i.e. sampling plans) or the subsequent interpretation of test results from such sampling programmes.

1.7 Assessment, reassessment and surveillance will be conducted at the permanent laboratory and individual or specific sites as appropriate.

## 2 Organisation

2.1 The technical management of the laboratory shall include at least a member with in-depth knowledge of and extensive experience in analytical chemistry. He/She is responsible for the laboratory and on-site technical operations with respect to air sampling and analysis for organic compounds.

## 3 Quality system

3.1 Sampling and site testing activities should be conducted in strict accordance with the quality system of the permanent laboratory.

3.2 The Quality Manual (QM) for the permanent laboratory shall cover all aspects of operation of sampling, site testing and the permanent laboratory. It shall have a separate section on site sampling / testing containing the following:

(a) an up-to-date register of long-term monitoring sites (with duration of more than three months).

(b) details of type of sampling / testing undertaken at each site, e.g. sampling, testing at site laboratory, in-situ testing, etc.

(c) the quality policy for sampling / testing on site, including a statement that a standard of service consistent with the requirements of HOKLAS shall be provided at all times for the HOKLAS accredited tests (including sampling).

(d) a separate list of sampling / testing performed on site and / or at the permanent laboratory.

3.3 The QM shall also contain details of how the quality system is applied to site sampling / testing, including:

(a) an organisation chart for the permanent laboratory showing lines of responsibility for staff authorized to conduct site sampling and

testing.

(b) the arrangements for the supervision of sampling / testing at sites including those not under the direct control of the laboratory.

(c) defined authority and responsibility of personnel designated for controlling all technical and quality aspects on site sampling / testing and their interrelationship with the technical management and the Quality Manager of the permanent laboratory.

## 4 Review of requests

4.1 Acceptance of sampling job requests shall be based on information submitted by customers in a work request form which should include a detailed sampling plan (or in-situ test plan) and the tests required (may be performed in permanent laboratory) for the samples.

4.2 The sampling plan (or in-situ test plan) shall include the sampling location, the exact sampling points as well as the frequency, time and type of sampling and number of samples required per sampling point.

## 5 Subcontracting of work

5.1 The subcontractor to which the accredited sampling and/or testing are/is subcontracted shall be a laboratory accredited under HOKLAS or by HOKLAS MRA partners for the relevant activities. Results of tests performed by subcontractors shall be clearly identified in test reports.

## 6 Access to sites

6.1 Access to the sampling / testing site shall be provided for HKAS representatives and assessors during HOKLAS assessment. If the site is not under the direct control of the laboratory, the permanent laboratory shall negotiate for their access to the appropriate parts of the site.

## 7 Control of records

7.1 For sampling and site testing, procedures for recording and reporting results

obtained on site shall be available and in line with that for the permanent laboratory.

7.2 In case of sampling (or in-situ testing), the details of the sampling / testing event shall be recorded. For this purpose, a proforma record sheet including space for diagrams should be provided. Other media such as videotape and photography should be used where appropriate to supplement written description and to serve as supporting evidence of the event.

7.3 Procedures shall exist for ensuring commercial confidentiality and security of test data and samples held on site.

7.4 The person(s) performing the testing/sampling, and the supervisor present on site shall sign on the record sheet at the time when the activities are being carried out.

## 8 Internal audits

8.1 The laboratory shall conduct periodic internal audits in accordance with a predetermined schedule and procedure for sampling and testing carried out in both the permanent laboratory and on site.

8.2 For laboratories involved in sampling and / or site testing, the Quality Manager of the permanent laboratory or his/her deputy shall visit the sites as part of the audits.

8.3 The site staff performance shall be audited by their supervisor, the Quality Manager or his/her deputy at least annually or at more frequent intervals.

8.4 Each type of sampling / testing listed on the HOKLAS scope of accreditation for site sampling / testing shall be audited and reviewed by the Quality Manager and / or his/her deputy at least annually or at more frequent intervals, depending on the extent of sampling / testing performed.

8.5 The documented results of all audits so obtained for site sampling / testing are subject to examination by HKAS accreditation officer and assessors during their assessment visits to the permanent laboratory.

8.6 The Quality Manager of the permanent laboratory or his/her deputy shall monitor progress on remedial actions identified and recorded during audits and reviews. If necessary, he/she shall make extra visits to sites to ensure

these actions have been discharged and the audit records completed.

## 9 Personnel

9.1 Approved signatories shall have at least a bachelor of science degree in chemistry, chemical technology or applied science, with not less than three years relevant sampling / testing experience. Appropriate membership of professional bodies is also acceptable. In addition, they shall have more than six months experience in the area of sampling / testing for which signatory approval is sought. Special consideration may be given to persons without the above qualifications but with extensive experience (at least 10 years) in the sampling / testing area concerned.

9.2 In all cases, candidates for approved signatories shall demonstrate to the assessors their technical competence before signatory approval can be granted.

9.3 Operators of special analytical equipment like GC/MS or GC/ITD should have, as a minimum, either a bachelor degree in chemistry with at least six months of experience in the sampling / testing techniques involved, or a high diploma in chemical technology or equivalent, with more than three years of relevant sampling / testing experience.

9.4 Sampling / testing shall only be performed by personnel who are employed by, or under contract to, the laboratory. The laboratory shall ensure that sufficient supervision is provided by trained staff to sampling / testing staff on site and in the laboratory, and also to contracted personnel to assist in sampling / testing. There shall be procedures to ensure that staff deployed for sampling / testing are properly trained. The contract workers shall be trained by the laboratory on the proper use of equipment and they shall possess sufficient technical knowledge and skill necessary for the correct performance of the activities they are responsible. Means to ensure that they carry out the sampling activities in accordance with the documented procedures is required.

9.5 Training programme shall include criteria for acceptance of staff to become authorized site testing staff or sampler. Training and competence assessment records, including any raw data, shall be maintained by the laboratory. Records of authorisation shall also be maintained.

## 10 Accommodation and environment

10.1 The environment in which the sampling / testing is undertaken shall meet all sampling / test procedure requirements, not invalidate the sampling / test results, or adversely affect the required accuracy and precision of measurement. The environmental conditions shall be recorded.

10.2 Where sampling / testing is undertaken in a hostile environment, procedures shall be available for checking that the environment does not adversely affect the performance of sampling / test equipment.

10.3 Adequate measures shall be taken to ensure good housekeeping in the sampling / testing area.

10.4 In the case where automatic sampling / test equipment is used and where operators may not be present all the time, there shall be provisions to ensure security of such equipment and integrity of samples / results obtained.

10.5 There shall be effective separation between neighbouring laboratory areas of incompatible activities, especially tests which are likely to cause contamination to the test samples and equipment.

## 11 Sampling / Test methods and method validation

11.1 A sampling / test procedure manual shall be available to all staff performing sampling and / or testing in the laboratory and on site. Where available, procedures published in international and national standards should be employed.

11.2 The sampling procedure should be given in adequate detail with step-by-step instructions, taking into consideration the requirement of the sampling plan provided, specific parameter of interest, sampling techniques, sample volume, sample storage requirements, sampler certification requirements, type of equipment, and calibration (if required). Reference to any site testing procedures (if required) and to a proforma sampling record form should also be made.

11.3 Procedures shall exist for provision of current sampling / test specifications to staff working on site. Documented procedures and any amplification of standards and specifications shall be available to sampling / testing staff on

site for all non-standard tests (even if based on a national standard) and for all standards and specifications needing amplification.

11.4 Tests which are listed in the Scope of Accreditation for site sampling / testing shall be carried out on site in accordance with HOKLAS requirements and any specific requirements laid down by the customer. Any deviations, additions or exclusions from the accredited sampling / test procedures and conditions as imposed by customers shall be recorded in detail in permanent worksheets and on all documents containing the sample information or the test results. HOKLAS endorsed reports shall not be used in such cases because the procedures used have not been assessed by HKAS and hence are outside the scope of accreditation. Regulations on the inclusion of results of non-accredited activities in a HOKLAS endorsed report or certificate given in HKAS Supplementary Criteria No. 1 and HOKLAS Supplementary Criteria No. 33 apply to these cases.

11.5. Test procedure should include the acceptance criteria for identification and quantitation of each compound of interest, the number of significant figures for reporting of numerical results, and be in accordance with the test standards on which the procedures are based.

11.6 Test method performance characteristics including the following should be determined and recorded:-

(a) test parameters applicable

(b) the method precision at various concentration ranges of interest supported by data on replicate testing of samples and / or standard.

(c) the practical quantitation limit (PQL, lowest reporting limit) estimated from method detection limit (MDL).

Note : Normally PQL is about 5 times the MDL. The determination of MDL is based on seven replicate measurements (including sampling where applicable) performed at different times and by different operators on a test sample or a standard reference material, of low analyte concentration. The reference material used should be in the same form as the sample (e.g. canister for USEPA TO-14A). The standard deviation (SD) so obtained multiplied by 3.14 gives the MDL. The claimed MDL should be lower than one-fifth of the concentration of the analyte in the

test sample. Such limits should be suitably verified.

(d) method bias at various concentration ranges of interest.

Note : The method bias should be supported by sufficient data obtained by carrying out the complete test procedure (sampling and analysis as applicable) using a certified reference material either traceable to national standards or verified otherwise. The reference material should be introduced in the form which simulates that of real samples. Reference standard canister gas may be used to obtain the bias values for the analytical procedure for TO-14A (or, in the case of TO-2, absorption tube charged with known amount of analyte of interest). For sampling, an audit gas (from cylinder plus suitable dilution equipment) may be introduced into the sampling manifold to mimic the actual field sampling conditions. The acceptable bias should be within ±30% of the certified standard reference value.

11.7 Since there are various approaches to estimate measurement uncertainty and no consensus on the method to be used in analysis of air for organic compounds, any valid methods given by reputable professional and standard writing bodies will be accepted by HKAS. The uncertainty estimated should be in line with the definition given by "International Vocabulary of Metrology - Basic and General Concepts and Associated Terms" (VIM) and should include all major components of uncertainty. Reference to the EURACHEM/CITIC document "Quantifying Uncertainty in Analytical Measurement" and "Protocol for uncertainty evaluation from validated data" published by LGC, UK, may be useful.

11.8 The procedures shall be documented for contingency situation when equipment is inoperative or damaged at the site, including where necessary the halting of work and its resumption after repair or replacement of the equipment.

## 12 Equipment

12.1 The laboratory shall have procedures for operating, maintaining and calibrating equipment used in sampling / testing.

12.2 Sampling system should be certified by using appropriate zero air and

reference gases in accordance with standard test procedures. Sampling devices should be verified clean before being used for sampling.

12.3 For sampling device of disposable type e.g. adsorption tubes, not less than 5% of the total should be checked and confirmed free of contamination before use.

12.4 For re-usable sampling device like canisters, 100% of the canisters should be certified clean initially in accordance with TO-14A procedure before being used for sampling runs. These canisters should be re-certified clean prior to each subsequent sampling exercise. Only until there is sufficient evidence to confirm the reliability of the cleaning process can the checking frequency of canisters be reduced to a lower percentage.

12.5 Automatic site sampling / testing equipment should be checked (either on-site or in laboratory) at appropriate intervals to ensure all operating parameters are as specified.

12.6 Documented procedure should be available for handling of such re-usable sampling devices to ensure cross-contamination is reduced to minimum (e.g. source sampling canisters should not be used for ambient sampling). Detailed records on the history of use and re-certification of canisters should be maintained.

12.7 Precautions shall be taken to ensure that the equipment, after transportation to the site and during use, sampling, testing and calibration, remains in a serviceable state and in calibration.

12.8 Appropriate checks shall be performed on site to confirm calibration status before sampling / testing / calibration commences. Where reliable checks cannot be carried out on site, the calibration status should be established in the permanent laboratory before and after site sampling / testing and augmented by on-site checks with suitable check standards (see Section 15).

12.9 Equipment which is unfit for use or out of calibration shall be immediately withdrawn from service. Sampling / test reports shall not be issued for sampling / tests carried out with equipment being found out of calibration after use.

## 13 Measurement traceability

13.1. The calibration programme for the analytical system (e.g. GC/MS or GC/ITD plus cryogenic trap) should at least consist of the following features and be documented and implemented.

13.1.1 Full calibration upon commissioning and after any maintenance event which might affect the initial calibration curve or if the daily calibration criteria is not met.

(a) For parameters known to display linear response, at least a blank and three concentration levels with the lowest being just above the PQL (taken as 5 times the MDL), should be included. In addition, the concentration range should bracket the expected concentration of the analytes. No data should be taken beyond the range of calibration.

(b) For parameters known to display non-linear response, further calibration points should be obtained, depending on individual parameters.

13.1.2 Acceptance criteria for the calibration of the analytical system

(a) The acceptance criteria should be documented and should in any case be within the following limits for each test parameter.

| | |
| --- | --- |
| Relative standard deviation of the response factor (for 3 or more calibration points) | < 30% of the mean |

13.1.3 Daily calibration of the GC system

The calibration should consist of initial one-point (or more) check of the calibration curve followed by a blank sample which should be in the same form as the test samples (i.e. in the case of TO-14A, the blank should be a certified canister pressurized with humidified ultra pure zero air and carried through the same analytical procedures as the field sample). The sequence of calibration check and blank samples should be repeated for every 20 samples or per batch or every 24 hours whichever is more frequent.

13.1.4 The acceptance criteria for routine calibration check

The acceptance criteria should be documented and should in any case

be within the following limits for each analyte:

| | |
|---|---|
| response factor of calibration check standard | ± 30% of the mean response factor of most updated calibration curve |
| the retention time | ± 3 SD of the mean retention time of most updated calibration curve |

13.1.5 The routine blank sample

It should not contain any analyte at concentration level greater than MDL nor compounds with elution characteristics and mass spectral features that would interfere with identification and quantitation of the analyte.

13.2 Vacuum / pressure gauges shall be calibrated at intervals recommended by HOKLAS Supplementary Criteria No. 2, Appendix C.

13.3 Calibration of flow measuring / control devices shall be conducted and traceable to the International System of Units (SI) of measurement. In the event that the reference flowmeter is of manual soap-bubble type, the volume of such meter should be calibrated on commissioning and visual inspection for damage at regular intervals should be carried out.

13.4 Gilian type electronic flowmeters should be calibrated at a frequency not less then once every year. Acceptance criteria for calibration of flowmeters should be documented and, in any case, the uncertainty should be within 2% of the reading for working flowmeters. Mass flow controllers should be calibrated at the range of interest once every 6 months with uncertainty within 5% of the reading.

13.5 Reference materials shall, where possible, be traceable to national or international certified reference materials. In cases where it is necessary to use commercially prepared chemical standards as reference materials, the certified values of each batch of the chemicals shall be verified (e.g. against certified reference gases) and verification records held.

13.6 Where it is necessary to use reference materials on site, adequate precautions shall be taken to ensure that their calibration status is maintained during transportation and while on site.

13.7 There should be documented procedure for use of reference materials outside the permanent laboratory for sampling, tests and calibrations.

## 14 Handling of test samples

14.1 Precautions against damage and contamination shall be taken during storage, handling, mounting and transportation of test samples.

14.2 Every sample obtained on site shall carry a unique sample identification, the sampler identification, date of sampling, time, location and environmental conditions of sampling, and any additional information as required by the test standard procedure, e.g. canister pressure for TO-14A.

14.3 When automatic site sampling equipment is used over a period of time, due consideration shall be given to sample storage requirements for individual target compounds.

## 15 Assuring the quality of sampling / test results

15.1 For sampling, the laboratory should have a quality control (QC) programme which includes the use of blanks, duplicates, control samples as appropriate. Given below for reference are common QC measures :

(a) Collection of duplicate samples at each long-term monitoring site on a regular basis.

(b) Preparation of field control QC samples with analyte(s) at appropriate level by introducing into the sampling manifold an audit gas (using a suitable dilution equipment where necessary) and the use of these samples on routine basis to identify any field and transportation effects.

(c) Routine preparation of field blanks for use in detection of presence or absence of possible contamination during sampling. The field blank must have negligible amount of the analyte(s) or any interfering compounds and be processed through the entire sampling and analysis process as if it is an actual sample.

15.2 For laboratory analysis, a routine data verification programme including the regular use of QC check samples, blanks, duplicates and spike samples as

appropriate should be documented and accordingly implemented. Acceptance criteria for bias and precision must be established and documented in each test method for individual analytes before accreditation may be considered.

(a) Blank samples, QC check samples and duplicate samples should be analyzed at a minimum of once for every batch of samples or 20 samples or every 24 hours whichever is more frequent.

(b) Blank samples and QC check samples should be in a form which simulates that of real samples (e.g. in the form of canisters for TO-14A, or absorption tubes for TO-2).

(c) The reference material used for preparing QC check sample should be obtained from a source of supply different from that of the calibration standards.

15.3 Maximum acceptance criteria values should be within ±25% of the mean for duplicate samples and within ±30% of the corresponding certified values for QC check samples.

15.4 Maximum acceptance criteria for blank samples should be < 0.2 ppbV of each analyte if sampling is conducted in accordance with TO-14A.

15.5 The QC data so obtained from such verification practices should be recorded in such a way that trends are detectable, e.g. in form of control charts, and, monitored and reviewed, where practicable, by statistical techniques.

15.6 Procedures for dealing with situations in which the acceptance criteria for sample blanks, duplicates or QC check samples are not met should be documented. Records of corrective actions should be kept.

15.7 The laboratory should participate on a regular basis in relevant external interlaboratory comparison or proficiency testing programmes, where available. The frequency of participation should, as far as possible, commensurate with the volume of work encountered and be at least once a year.

## 16 Reporting of sampling and test results

16.1 Results and information obtained from sampling / site tests shall be identified as such on reports and certificates issued by the laboratory. Such sampling / test reports and certificates shall contain, in addition to information required

in HOKLAS Supplementary Criteria No. 33 and HOKLAS 003 Section 5.10 (where appropriate), the following information:

(a) An indication as to whether the reported results refer to sampling and site testing performed by the laboratory or to samples as received in case where the sampling is carried out by customers.

(b) The reporting limits for test results

(c) Quality control data and, where necessary, the qualifying statements if required for proper interpretation of sampling / test results, requested by the customer or specified in the test standards.