

Australian Government Department of Industry, Science and Resources National Measurement Institute

Study Protocol for Proficiency Testing

NMI North Ryde – CRV

Issue No.: Approved By: Prepared By: Amendments: Control: 4.9

CRV Manager

Refer to revision history

Raluca lavetz

Issued Date: Next Review: 8 October 2024 8 October 2029

The electronic copy on the WAN is the latest version of this document. Any paper copy is UNCONTROLLED and should be checked against the electronic copy before use.

© Commonwealth of Australia 2024.

Unless otherwise noted, the Commonwealth owns the copyright (and any other intellectual property rights, if any) in this publication.

All material in this publication is provided under a Creative Commons Attribution 4.0 International Licence (CC BY 4.0), with the exception of:

- the Commonwealth Coat of Arms;
- the logo of the Department of Industry, Science and Resources;
- photographs of our staff and premises; and
- content supplied by third parties.

Creative Commons Attribution 4.0 International Licence is a standard form licence agreement that allows you to copy, distribute, transmit and adapt this publication provided you attribute the work. A summary of the licence terms is available at: <u>creativecommons.org/licenses/by/4.0/</u>. Further details are available on the Creative Commons website, at: <u>creativecommons.org/licenses/by/4.0/legalcode</u>.

You may not copy, distribute, transmit or adapt any material in this publication in any way that suggests that this department or the Commonwealth endorses you or any of your services or products.

Attribution

Material contained in this publication is to be attributed to this department as:

© Commonwealth of Australia, Department of Industry, Science and Resources, Study Protocol for Proficiency Testing, 2024.

Third party copyright

Wherever a third party holds copyright in material contained in this publication, the copyright remains with that party. Their permission may be required to use the material.

This department has made all reasonable efforts to:

- clearly label material where the copyright is owned by a third party;
- ensure that the copyright owner has consented to this material being contained in this publication.

Using the Commonwealth Coat of Arms

The terms of use for the Coat of Arms are available on the Department of Prime Minister and Cabinet's website, at <u>www.pmc.gov.au/resource-centre/government/commonwealth-coat-arms-information-and-guidelines</u>

Contents

| 1 | Fore | eword | .4 |
|---|------|---|-----|
| 2 | Prof | ficiency Testing (PT) program | .4 |
| | 2.1 | Scheme Provider | 4 |
| | 2.2 | Scheme Coordinator | 4 |
| | 2.3 | Objective of the Scheme | 4 |
| | 2.4 | Impartiality | 4 |
| | 2.5 | Participation Fees and Charges | 5 |
| | 2.6 | Externally provided services | 5 |
| | 2.7 | NMI PT Program | 5 |
| | 2.8 | Test Samples | 5 |
| | 2.9 | Test Samples Transport | 5 |
| | 2.10 | Information to Participants | 5 |
| | 2.11 | Testing requirements for participants | 6 |
| | 2.12 | Due date adjustments and late results | 6 |
| | 2.13 | Establishing the Assigned Value | 6 |
| | 2.14 | Target Standard Deviation for Proficiency Asessment | 7 |
| 2 | 2.15 | Statistical Analysis | 7 |
| 2 | 2.16 | Reports and Performance Evaluation | 7 |
| | 2.17 | Technical Advice and Feedback from Participants | 8 |
| | 2.18 | Confidentiality and Ethical Considerations | 8 |
| 3 | Refe | erences | . 9 |
| 4 | Ann | ex 1 – Individual Scheme Details | 10 |
| | 4.1 | Pesticides in Fruit, Vegetables and Herbs | 11 |
| | 4.2 | Controlled Drugs | 12 |
| | 4.3 | Controlled drugs in Wipes | 13 |
| | 4.4 | Pesticides in Soil and Water | 14 |
| | 4.5 | Hydrocarbons and PAHs in Soil and Water | 15 |
| | 4.6 | Inorganic Analytes, and Physical Tests in Soil and Water and Chlorophyll a in Water | 16 |
| | 4.7 | Toxic and Essential Elements in Food and Pharmaceutical | 17 |
| | 4.8 | Folic Acid in Flour | 18 |
| | 4.9 | PFAS in Biota, Food, Soil and Water | 19 |
| , | 4.10 | Rapid PT Studies | 20 |
| 5 | Ann | ex 2 – Scope of Accreditation (ISO/IEC 17043) | 21 |
| 6 | Rev | ision/Review History | 22 |

1 Foreword

This Study Protocol sets outs the organisational and logistical aspects of the National Measurement Institute's (NMI) Chemical Proficiency Testing (CPT) programs. It has been prepared to comply with the requirements for a study protocol specified in the ISO/IEC 17043.¹

The protocol should be read in conjunction with the **Statistical Manual** for Proficiency Testing Program which sets out the procedures for data analysis and assessment of performance, and the **CPT Standard Terms and Conditions for the Proficiency Testing Schemes.**

2 Proficiency Testing (PT) program

2.1 Scheme Provider

Chemical Reference Values (CRV)

National Measurement Institute

105 Delhi Road

Riverside Corporate Park

North Ryde NSW 2113 Australia

CRV is an accredited provider of proficiency testing schemes (see Annex 1 for the Services, Products and Determinants that are a part of the Scope of Accreditation). CRV also provides PT schemes that are not part of the Scope of Accreditation, and this will be clearly identified in communications with participants.

2.2 Scheme Coordinator

Manager Chemical Reference Values

Phone +61 2 9449 0111

Email: proficiency@measurement.gov.au

The scheme coordinator is responsible for all aspects of the testing schemes.

2.3 Objective of the Scheme

The objective of the NMI studies is to:

- enable laboratories to assess the accuracy of their routine test results and hence the effectiveness of their test methods (including subsampling, dilution/calculation, reporting results and uncertainty in correct units and format) and quality assurance programs;
- give a snapshot of the state-of-practice of chemical measurements;
- provide accreditation bodies or others with object evidence of laboratory performance;
- provide laboratories with a means of establishing traceability of their measurements; and
- provide education and guidance on issues of metrology in chemistry.

CRV offers a special PT program – Rapid Proficiency Testing Studies (Rapid PT). These studies can be requested by participants any time. The purpose of the Rapid PT is to assist laboratories if they miss one of the yearly main studies (eg for auditing and accfreditation purposes, training and/or as a tool to assess performance of a new method). More details in section 4.10.

2.4 Impartiality

NMI is committed to impartiality and to providing services free from influences of coercion or other efforts to cause misrepresentation of the facts and any related expert opinion.

2.5 Participation Fees and Charges

For most studies, participation is open to all laboratories. Participation in controlled drug studies including wipes is restricted to laboratories that can provide appropriate licenses and permits.

Typical rounds include between seven and forty laboratories from Australia and overseas. The rapid studies are designed based on participants requests where a pre-set assigned value is used and may include only one participant.

Participation fees are charged in Australian dollars according to the current proficiency testing price list.

Proficiency test samples lost or damaged in transit will be replaced free of charge, if feasible. Proficiency test samples lost or damaged after receipt by the participant laboratory will be replaced at a cost (e.g. additional samples participant laboratory price).

2.6 Externally provided services

Analytical testing associated with the PT program such as provision of homogeneity and stability testing may be done externally from time to time by a competent provider (accredited to ISO/IEC 17025 or when this is not possible, then a proven track record such as satisfactory performance in PT).

2.7 NMI PT Program

CRV Manager provides a list of PT programs to be conducted yearly. This list is published on the NMI website (https://www.industry.gov.au/national-measurement-institute/chemical-and-biological-measurement-services/proficiency-testing-services).

2.8 Test Samples

The nature of the test samples is different for each scheme. Details are in Annex 1.

For Rapid PT studies the test samples are samples from previous PT studies with stability assessed before dispatch. These studies are designed based on participants' requirements and on samples' availabilities.

2.9 Test Samples Transport

Test samples are packaged to minimise deterioration in transit.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

NMI takes reasonable steps to ensure that samples arrive in a fit condition for analysis. Where logistical problems prevent the delivery of samples in a fit condition, the participation fee will be refunded.

Details of the transport arrangements for each sample type are presented in Annex 1.

2.10 Information to Participants

The invitation letter for each study round includes details of:

- samples, analytes and analytes' level
- key dates,
- fees,
- participation form for enrolling in the round.

Participants are given approximately four weeks to perform the analysis.

Sample dispatch documents include

• instruction for analysis,

- deadline for returning results,
- handling and storage conditions of test materials
- electronic results sheet.

2.11 Testing requirements for participants

Participants are usually requested to use their normal method of analysis and report a single result, together with an associated uncertainty and a coverage factor. Also in the result form participants are requested to provide information on the method of analysis used.

Occasionally participants are requested to perform certain analytical steps such as sampling or calibration according to specifications provided by NMI.

2.12 Due date adjustments and late results

NMI may adjust the results deadline to accommodate for exceptional circumstances. If the deadline is adjusted, all participants will be informed and will be allowed to submit or re-submit their results up until the new results deadline. The interim report issue date will also be adjusted accordingly. Examples of such circumstances include:

- Sample delivery delays to participants due to e.g. courier/distributor delays, or permit delays.
- Samples arrived not fit-for-purpose and new samples were dispatched.
- Uncontrollable events (e.g. pandemic, staff shortage, public holidays) affecting some or all participants, and/or NMI.

For any other exceptional circumstance the NMI PT team will assess each request on a case-by-case basis.

2.13 Establishing the Assigned Value

The assigned value is the value to which participants' results are compared, and must be the best available estimate of the true concentration of analyte.

Several measures of concentration are available to establish the assigned value:²

- Consensus of participants. In a typical study the consensus of participants' results estimated as the robust average is used as the assigned value. The robust average and associated expanded uncertainties were calculated using the procedure described in ISO 13528, 'Statistical methods for use in proficiency testing by interlaboratory comparisons'.²
- Primary method of chemical measurement (e.g. isotope dilution mass spectrometry, IDMS). Measurements made using primary methods are traceable to SI units within their uncertainties. Where primary methods are available for a particular analyte, the results will be used as the assigned value.
- Formulation. Where samples are prepared by spiking an analyte-free matrix with a solution of a pure chemical, the formulated concentration often has the smallest uncertainty and maybe used as the assigned value.
- Direct comparison with certified reference materials. Where the test material is a Certified Reference Material (CRM), or can be directly compared to a CRM of similar composition, the certified value of the CRM will form the basis of the assigned value.
- NMI testing performed using a method other than a primary method.

The relevant mathematics are presented in the Statistical Manual for Proficiency Testing Program.

For the Rapid PT studies the assigned value and associated measurement uncertainty from the original PT study will be used to calculate performance scores.

2.14 Target Standard Deviation for Proficiency Asessment

The target standard deviation for proficiency asessment used to calculate z-scores is selected by the study coordinator and is not the same standard deviation calculated from results returned by participant laboratories.

The advantages of setting a target standard deviation for proficiency assessment are:

- it is the preferred approach specified in the international protocol;
- it enables z-scores to be used as a fixed reference point for assessment of laboratory performance, independent of variations in group results from one study to the next;
- z-scores derived from it can be compared from study to study, to demonstrate performance trends for an individual laboratory or group of laboratories; and
- calculated z-scores are indicative of the current state of practice. By setting a realistic target standard deviation, current laboratory performance can be compared to achievable performance. This can provide a benchmark for progressive improvement.

The target standard deviation for performance assessment is calculated as the product of the assigned value and the performance coefficient of variation (PCV). The PCV is a measure of the between-laboratory coefficient of variation that, in the judgment of the study organisers, would be expected from participants given the analyte, concentration and matrix.

The PCV is determined from the following:

- a fitness for purpose goal for performance, as determined by expert judgement or regulatory mandate
- acceptance criteria indicated by the methods
- experience from previous studies.
- an estimate from a statistical model (e.g. Horwitz-Thompson model).

Examples for various studies are presented in Annex 1.

For the Rapid PT studies the target standard deviation for performance assessment is the one used in the original PT study. Any changes to the target standard deviation for performance assessment must be documented and signed by the study coordinator.

2.15 Statistical Analysis

The statistical treatment used by CPT to assess laboratory performance is based on the procedures described in ISO 13528,² 'The International Harmonized Protocol for Proficiency Testing of Analytical Chemistry Laboratories',³ and 'Proficiency testing in analytical chemistry'.⁴ Laboratory performance is assessed by comparing reported test results to the assigned value using both z-scores and E_n-scores.

The calculations are presented in the Statistical Manual for Proficiency Testing Program.

2.16 Reports and Performance Evaluation

The Interim Report primary purpose is to provide each participant with an early indication of performance. We aim to issue this report within five working days from the due date for submission of results. The interim report is distributed by email as a PDF file.

A Preliminary Report will also be issued. We aim to issue this report within 10 working days of the due date for submission of results. The report will include: a summary of the results reported by laboratories, assigned values, performance coeficient of variations, z-scores and E_n-scores for each analyte tested by the participant laboratory. Some of the data from this report may change in the Final Report. The purpose of this report is to provide participants with a faster feedback on their laboratory's performance.

We aim to produce a detailed final report within ten weeks of the closing date for submission of results with exception of complex reports (eg large number of data or reference values to be included in the study). The final report includes:

- Summary;
- Introduction general description and study objectives;
- Test items test sample preparation, homogeneity and stability testing;
- Summary of participants' tests methods;
- Definition of terms and statistics used (z-scores and En-scores);
- Participants' results results tables including assigned values, statistical summary data, histograms of results, z-scores and E_n-scores, scatter plots; and
- Discussion of Results assigned value and traceability, measurement uncertainty, basis of measurement uncertainty estimates, interpretation of graphical data, individual analyte summaries, participant comments.

Final reports are emailed to participants together with the individual Performance Summary and Performance History reports.

No Interim and Preliminary Reports are issued for the Rapid PT Study. The aim is to produce the Final Report within five working days from receiving results unless otherwise agreed with the participant.

The final reports are public documents and are published on the NMI website (https://www.industry.gov.au/national-measurement-institute/chemical-and-biological-measurement-services/proficiency-testing-services).

2.17 Technical Advice and Feedback from Participants

NMI actively seeks advice from technical experts on aspects of its programs. This may be in the form of an advisory group formed for this purpose, or NMI may draw upon existing industry groups. The groups have no executive powers and coordination of the schemes remains the responsibility of NMI. Group members agree to hold in-confidence information about the scheme. Laboratory codes are not disclosed to members of the group. Advice is also sought from the technical experts of NMI.

Details of the advisory arrangements for particular programs are detailed in Annex 1.

NMI also seeks feedback from participants in the program. Results sheets and other documents include a section for participants' comments. Such comments are reported in the final report, together with the study coordinator's response (where appropriate).

Complaints from customers are dealt with promptly, confidentially and in accordance with NMI procedures.

Appeals received from participants are sent to the ChemBio Branch Manager for resolution. The ChemBio Manager may call upon other sections in NMI that are not involved with the PT study, or engage outside scientists for expert advice.

For both complaints and appeals, the NMI Corrective Action, Preventative Action and Continual Improvement procedure will be followed.

2.18 Confidentiality and Ethical Considerations

The identity of participants is protected by means of a laboratory code, which is randomly assigned when a laboratory enrols in a PT round. These codes are confidential and are not disclosed to other persons. The codes are sent to each individual lab in the instructions letter that accompanies the samples, with the exception of international participants, who are handled by distributors. For international participants who are handled by distributors, the identification code will be advised after samples dispatch in an email sent directly to participants.

The CRV Team is a part of NMI. However if an NMI laboratory participates in a round they do so on the same basis as other participants. Other parts of NMI do not have access to CPT participant details. The CPT team in the CRV section and other NMI staff are bound to uphold Australian Public Service Values and abide by the Code of Conduct (https://www.apsc.gov.au/integrity-aps).

NMI conducts its program in the belief that participants perform the analysis and report results with scientific rigour. However NMI will take steps to prevent collusion or falsification of results by participants. Where any collusion or falsification is proven, the results for that participant for the PT round concerned will be expunded and the laboratory manager will be notified.

3 References

Note: For all undated references, the latest edition of the referenced document (including any amendments) applies.

1. ISO/IEC 17043, Conformity assessment – General requirements for proficiency testing, ISO, Geneva, Switzerland.

2. ISO 13528, Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO, Geneva, Switzerland.

3. Thompson, M. Ellison S. L. R and Wood, R., The international harmonized protocol for proficiency testing of analytical chemistry laboratories, Pure Appl. Chem. 78, 145-196, 2006.

4. Lawn, R. E., Thompson, M. and Walker, R. F., Proficiency testing in analytical chemistry, LGC, Teddington, UK, 1997.

5. Australian Government Federal Register of Legislation, *Australia New Zealand Food Standards Code – Schedule 20*, viewed March 2023, <u>https://www.legislation.gov.au/Series/F2015L00468</u>

6. Australian Government, 2011, Clandestine Drug Laboratory Remediation Guidelines

Page 9 of 22

4 Annex 1 – Individual Scheme Details

- 4.1 Pesticide in Fruit, Vegetables and Herbs
- 4.2 Controlled Drugs
- 4.3 Controlled Drugs in Wipes
- 4.4 Pesticide in Soil and Water
- 4.5 Hydrocarbons and PAHs in Soil and Water
- 4.6 Inorganic Analytes, Chlorophyll a and Physical Test in Soil and Water
- 4.7 Toxic and Essential Elements in Food and Pharmaceutical
- 4.8 Folic Acid in Flour
- 4.9 PFOS/PFOA in Food, BiotaSoil and Water
- 4.10 Rapid PT Studies

4.1 Pesticides in Fruit, Vegetables and Herbs

4.1.1 Test Samples

Each round may include two to four approximately 100 g of test samples consisting of a homogenised vegetable, fruits and/or herbs commodity, spiked with selected pesticides. Portions of the un-spiked (blank control) commodity are also supplied.

The commodities and analytes are selected from those typically encountered in residue testing. Residues of current concern in trade or agriculture are given priority. Concentrations of pesticide range from near the limit of detection of typical testing methods, to above the relevant Australian Maximum Residue Limit.⁵

4.1.2 Test Samples Transport

Test samples are sent by courier packaged in an insulated foam box with gel ice-packs, Although the samples are frozen when sent, NMI cannot guarantee that the samples will arrive frozen. Matrices and pesticides are chosen so that short periods (up to 1 week) at room temperature will not invalidate study results.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.1.3 Target Standard Deviation for Performance Asessment

The target standard deviation for performance assessment used to confirm sufficient homogeneity and for calculation of z-scores is equivalent to a performance coefficient of variation of 15%. That is 0.15 * assigned value. Examples are presented in Table 1. The target standard deviation for performance assessment may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

| Sample | Analyte | Assigned value (mg/kg) | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|--------------------|---------------------------|----------------------------|---------------------------|
| S1 | Azoxystrobin | 1.17 | 16 | 15 |
| | Endosulfan sulfate | 0.741 | 17 | 15 |
| | Methamidophos | 0.925 | 16 | 15 |
| | Permethrin | 0.70 | 17 | 15 |

Table 1 Performance Coefficient of Variation for Pesticides in Fruit and Vegetables

4.1.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each pesticide that is detected. NMI does not specify whether or not the results should be corrected for recovery, however participants are asked to report the percent recovery if it has been determined. A list of pesticides from which those spiked into the samples is provided. Participants may elect to test for all or for only some of these.

4.1.5 Advisory Committee

The advisory group consists of individuals with relevant technical expertise. Members volunteer, and sit in a personal capacity not as representatives of any specific organisations. There is no formal assessment of the qualification to sit, however members will typically be current or former senior scientists from residue testing laboratories. Group members agree to hold in confidence information about the scheme. Laboratory codes are not disclosed to members of the group.

The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time.

Advice is also sought from the technical experts of NMI.

4.2 Controlled Drugs

4.2.1 Test Samples

Each study includes three to four test samples consisting of 150 mg of powder each. Samples are usually prepared from seizures of controlled drug and typically contain between 5 and 80% w/w of drug as the base. Drugs studied include heroin, cocaine, amphetamines and amphetamine type substances (e.g. MDA, MDMA, MDEA). Participants are informed of the identity of the drug and so the studies focus on quantitation.

4.2.2 Test Samples Transport

Test samples are sent by courier packaged in a sealed bag. Controlled drugs samples do not require ice packs when dispatched. Samples will only be sent to bona-fide laboratories that provide appropriate documentation:

- Australian participants must provide a permit to hold controlled drugs.
- Overseas participants must provide an import permit acceptable to the Therapeutic Goods Administration, the Australian authority that issues export licences.

4.2.3 Target Standard Deviation for Performance Asessment

The target standard deviation for performance assessment used to confirm sufficient homogeneity and for calculation of z-scores is equivalent to a performance coefficient of variation of 3%. That is 0.03 * assigned value. Examples are presented in Table 2. The target standard deviation for performance assessment may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

| Sample | Analyte | Assigned Value (% base (m/m)) | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|-----------------|--|----------------------------|---------------------------|
| S1 | MDMA | 63.0 | 1.3 | 3 |
| S2 | | 31 / | 1.8 | 3 |
| S3 | IVIDIVIA | 51.4 | 1.0 | 5 |
| S4 | Methamphetamine | 45.3 | 1.5 | 3 |

Table 2 Performance Coefficient of Variation for Controlled Drugs

4.2.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result together with an associated uncertainty. In some cases the identity of any diluent may also be requested.

4.2.5 Advisory Committee

There is no formal advisory committee for the controlled drug scheme. Advice is sought from the senior advisory group (SAG) of Australian forensic laboratories. The NMI also has access to technical experts within the Australian Forensic Drug Laboratory and through them to the Australian Federal Police, who provide advice on drugs of current concern.

4.3 Controlled drugs in Wipes

Wipes are used to swipe non-porous surfaces inside a building that was used to manufacture illegal drugs (clandestine laboratories, 'clan labs') in order to check the extent of contamination.

This scheme enables laboratories to assess their ability to measure methamphetamine, MDMA and or pseudoephedrine in wipes at investigation levels specified in Clandestine Drug Laboratory Remediation Guidelines 2011.⁶

4.3.1 Test Samples

Each study includes two to four test samples consisting of wipes. Samples contain spiked analytes. Drugs studied may include methamphetamine, MDMA and/or (pseudo)ephedrine. Participants are informed of the identity of the drug spiked on the wipes and so the study focuses on quantitation.

4.3.2 Test Samples Transport

Test samples are placed in amber glass jars and are sent by courier packaged with a gel ice-pack. Samples will only be sent to bona-fide laboratories that provide appropriate documentation:

- Australian participants must provide a permit to hold controlled drugs.
- Overseas participants must provide an import permit acceptable to the Therapeutic Goods Administration, the Australian authority that issues export licences.

4.3.3 Target Standard Deviation for Performance Asessment

The target standard deviation for performance assessment used to confirm sufficient homogeneity and for calculation of z-scores is equivalent to a performance coefficient of variation of 20%. That is 0.20 * assigned value. Examples are presented in Table 3.

| Sample | Analyte | Assigned Value (µg/wipe as base) | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|-----------------|--|----------------------------|---------------------------|
| S1 | Methamphetamine | 1.14 | 22% | 20% |

Table 3 Performance Coefficient of Variation for Controlled Drugs in Wipes

4.3.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result together with an associated uncertainty.

4.3.5 Advisory Committee

There is no formal advisory committee for the controlled drug scheme. Advice is sought from the senior advisory group (SAG) of Australian forensic laboratories. The PT group also has access to technical experts within the Australian Forensic Drug Laboratory.

4.4 Pesticides in Soil and Water

4.4.1 Test Samples

Each round includes:

- two 50 g sieved and homogeneised soil test samples
- two or three 500 mL homogenised water test samples.

Each test sample may contain either spiked or incurred residues.

The pesticides are selected from those typically encountered in residue testing. Residues of current concern in trade or agriculture are given priority. Concentrations of pesticide range from near the limit of detection of typical testing methods, to above the relevant Australian environmental standards.

4.4.2 Test Samples Transport

Test samples are sent by courier packaged in an insulated foam box with gel ice-packs. Although the samples are chilled when sent, NMI cannot guarantee that the samples will arrive chilled. Matrices and pesticides are chosen so that short periods (up to 1 week) at room temperature will not invalidate study results.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.4.3 Target Standard Deviation for Proficiency Asessment

The target standard deviation for proficiency asessment used to confirm sufficient homogeneity and for calculation of z-scores is equivalent to a performance coefficient of variation of 15%. That is 0.15 * assigned value. Examples are presented in Table 4. The target standard deviation for proficiency may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

| Sample | Analyte | Assigned value (mg/kg) | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|------------|---------------------------|----------------------------|---------------------------|
| | Bifenthrin | 0.063 | 22 | 15 |
| S1 | Dicamba | 0.57 | 17 | 15 |
| | p,p'-DDE | 0.902 | 16 | 15 |

Table 4 Performance Coefficient of Variation for Pesticides in Soil and Water

4.4.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each pesticide that is detected. NMI does not specify whether or not the results should be corrected for recovery, however participants are asked to report the percent recovery if it has been determined. A list of pesticides from which those spiked into the samples is provided. Participants may elect to test for all or for only some of these.

4.4.5 Advisory Committee

The advisory group consists of individuals with relevant technical expertise. Members volunteer, and sit in a personal capacity not as representatives of any specific organisations. There is no formal assessment of the qualification to sit, however members will typically be current or former senior scientists from residue testing laboratories. Group members agree to hold in confidence information about the scheme. Laboratory codes are not disclosed to members of the group.

The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time. Advice is also sought from the technical experts of NMI.

4.5 Hydrocarbons and PAHs in Soil and Water

4.5.1 Test Samples

Each round includes two to four test samples consisting of sieved and homogenised soil, or two to four samples of homogenised water. The samples may contain either spiked or incurred analytes.

The hydrocarbons are selected from those typically encountered in environmental testing associated with the remediation of contaminated sites. They cover volatile (C6 - C10, BTEX) and semi volatile (>C10 - C40) hydrocarbon ranges as well as a range of PAHs. Concentrations range from near the limit of detection of typical testing methods, to above the relevant Australian environmental standards.

4.5.2 Test Samples Transport

Test samples are sent by courier packaged to prevent volatile losses and are despatched in an insulated foam box with a gel ice-pack. Although the samples are chilled when sent, NMI cannot guarantee that the samples will arrive chilled. Stability testing has demonstrated that short periods (up to 1 week) at room temperature will not invalidate study results.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.5.3 Target Standard Deviation for Performance Asessment

The target standard deviation for performance assessment used to confirm sufficient homogeneity and for calculation of z-scores is equivalent to a performance coefficient of variation of 15% to 20%. Examples are presented in Table 5. The target standard deviation for performance assessment may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

| Sample | Analyte | Assigned value (mg/kg) | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|----------|---------------------------|----------------------------|---------------------------|
| | >C10-C16 | 1590 | 5.3 | 20 |
| 61 | >C16-C34 | 2520 | 4.9 | 20 |
| 51 | >C34-C40 | 317 | 6.7 | 20 |
| | TRH | 4400 | 4.5 | 15 |

 Table 5
 Performance Coefficient of Variation for Hydrocarbons in Soil and Water

4.5.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each analyte. NMI does not specify whether or not the results should be corrected for recovery, however participants are asked to report the percent recovery if it has been determined.

4.5.5 Advisory Committee

There is no formal advisory committee for the Hydrocarbons in Soil and Water schemes. Advice is sought from the technical experts in the environmental testing area of NMI.

4.6 Inorganic Analytes, and Physical Tests in Soil and Water and Chlorophyll a in Water

4.6.1 Test Samples

Each round includes one to six test samples consisting of sieved and homogenised soil, homogenised water and/or filters. The samples may contain either spiked or incurred analytes.

The analytes are selected from those typically encountered in environmental testing. Concentrations range from near the limit of detection of typical testing methods, to above the relevant Australian environmental standards.

4.6.2 Test Samples Transport

Test samples are sent by courier packaged to prevent losses. Water and filter samples are despatched in an insulated foam box with gel ice-packs, soil samples do not require ice pack. Although the water samples are chilled or frozen when sent, NMI cannot guarantee that the samples will arrive chilled/frozen. Stability testing has demonstrated that short periods (up to 1 week) at room temperature will not invalidate study results.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.6.3 Target Standard Deviation for Proficiency Asessment

The target standard deviation for proficiency assessment used to confirm sufficient homogeneity and for calculation of z-scores is equivalent to a performance coefficient of variation of 3.5% to 20%. Examples are presented in Table 6. The target standard deviation for performance assessment may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

| Sample | Test | Assigned value (mg/kg) | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|------|------------------------------|----------------------------|---------------------------|
| S1 | As | 3.29 | 13% | 15% |
| S1 | Bi | 3.28 | 13% | 15% |
| S1 | Cd | 0.840 | 16% | 15% |
| S1 | Cr | 25.4 | 9.8% | 10% |

 Table 6 Performance Coefficient of Variation for Inorganic Analytes and Chlorophyll a in

 Soil or Water

4.6.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each analyte. Advisory Committee

4.6.5 Advisory Committee

The advisory group consists of individuals with relevant technical expertise. Members volunteer, and sit in a personal capacity not as representatives of any specific organisations. There is no formal assessment of the qualification to sit, however members will typically be current or former senior scientists from residue testing laboratories. Group members agree to hold in confidence information about the scheme. Laboratory codes are not disclosed to members of the group.

The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time. Advice is also sought from the technical experts of NMI.

4.7 Toxic and Essential Elements in Food and Pharmaceutical

4.7.1 Test Samples

Each round includes two test samples consisting of homogenised material. The samples may contain either spiked or incurred analyte.

Analytes are selected from those typically encountered in food and pharmaceutical testing. Concentrations range from near the limit of detection of typical testing methods, to above the relevant Australian food standards.

4.7.2 Test Samples Transport

Test samples are sent by courier packaged to prevent losses.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.7.3 Target Standard Deviation for Proficiency Asessment

The target standard deviation for proficiency assessment used to confirm sufficient homogeneity and for calculation of z-scores is equivalent to a performance coefficient of variation of 10% to 20%. Examples are presented in Table 7. The target standard deviation for proficiency assessment may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

| Sample | Test | Assigned value (mg/kg) | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|------|------------------------------|----------------------------|---------------------------|
| S1 | Ag | 0.161 | 21% | 15% |
| S1 | As | 2.60 | 14% | 15% |
| S1 | Ва | 3.62 | 13% | 15% |
| S1 | Ca | 1830 | 5.2% | 10% |

Table 7 Performance Coefficient of Variation for Inorganic Analytes in Food and Pharmaceuticals

4.7.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each analyte. Advisory Committee

4.7.5 Advisory Committee

The advisory group consists of individuals with relevant technical expertise. Members volunteer, and sit in a personal capacity not as representatives of any specific organisations. There is no formal assessment of the qualification to sit, however members will typically be current or former senior scientists from residue testing laboratories. Group members agree to hold in confidence information about the scheme. Laboratory codes are not disclosed to members of the group.

The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time.

Advice is also sought from the technical experts of NMI.

4.8 Folic Acid in Flour

4.8.1 Test Samples

Each study includes three to five test samples consisting of 20 g of flour. Samples are prepared by adding fortified flour pre-mix to the unfortified flour and mixing thoroughly. Concentrations covers the range from 0 to 5 mg/kg folic acid to include the Food Standards Code mandatory level of between 2 to 3 mg/kg as well as below and above the mandatory levels.

4.8.2 Test Samples Transport

Test samples are sent by courier packaged in a sealed bag at room temperature.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.8.3 Target Standard Deviation for Performance Asessment

The target standard deviation for performance asessment used to confirm sufficient homogeneity and for calculation z-scores is equivalent to a performance coefficient of variation of 15%. That is 0.15 * assigned value.

4.8.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each analyte. NMI does not specify whether or not the results should be corrected for recovery, however participants are asked to report the percent recovery if it has been determined.

4.8.5 Advisory Committee

The advisory group consists of individuals with relevant technical expertise. Members volunteer, and sit in a personal capacity not as representatives of any specific organisations. There is no formal assessment of the qualification to sit, however members will typically be current or former senior scientists from residue testing laboratories. Group members agree to hold in confidence information about the scheme. Laboratory codes are not disclosed to members of the group.

The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time.

4.9 PFAS in Biota, Food, Soil and Water

4.9.1 Test Samples

Each round includes:

- two to three 5 50 g sieved and homogenised biota and/or food test samples
- two to three 10 g sieved and homogenised soil test samples
- two to three 2 x 50 mL homogenised water test samples.

Each test sample may contain either spiked or incurred analytes.

Concentrations range from near the limit of detection of typical testing methods, to above the relevant Australian standards.

4.9.2 Test Samples Transport

Test samples are sent by courier packaged in an insulated foam box with gel ice-packs. Although the samples are chilled when sent, NMI cannot guarantee that the samples will arrive chilled. Short periods (up to 1 week) at room temperature will not invalidate study results.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.9.3 Target Standard Deviation

The target standard deviation used to confirm sufficient homogeneity and for calculation z-scores is equivalent to a performance coefficient of variation of 20%. Examples are presented in Table 8. The target standard deviation may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

| Sample | Analyte | Assigned value | Unit | Thompson-Horwitz CV (%) | Target SD (as PCV) (%) |
|--------|---------|----------------|------|----------------------------|---------------------------|
| S1 | PFDS | 2.28 | µg/L | 22 | 20 |
| S1 | PFDA | 6.80 | µg/L | 22 | 20 |

Table 8 Performance Coefficient of Variation for PFAS

4.9.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each analyte. NMI does not specify whether or not the results should be corrected for recovery, however participants are asked to report the percent recovery if it has been determined.

4.9.5 Advisory Committee

There is no formal advisory committee for PFOS/PFOA in Biota, Soil and Water samples. Advice is sought from the technical experts of NMI.

4.10 Rapid PT Studies

Rapid PT Studies are not run on a pre-defined schedule; laboratories can request this type of study at any time (e.g. if they have an immediate audit, missed enrolment or reporting results in a routine PT study or if they need to validate methodology and check the performance of a new analyst). Participants have the option to design their own study based on sample availability.

4.10.1 Test Samples

Each round includes one or more samples from previous PT studies and are assessed for stability before dispatch. The samples may contain either spiked or incurred analytes.

4.10.2 Test Samples Transport

The test samples are sent by courier packaged with gel ice-packs. Although the water samples are chilled or frozen when sent, NMI cannot guarantee that the sample will arrive chilled/frozen. Stability testing has demonstrated that short periods of time at room temperature will not invalidate the study results.

Participants must provide NMI with any import or quarantine permits that might be necessary. Participants are liable for any customs or import charges levied.

4.10.3 Target Standard Deviation for Performance Asessment

The target standard deviation used for calculation of z-scores is the same as the one used in the original study. Any changes to the target standard deviation for performance assessment must be documented and signed by the study coordinator.

4.10.4 Analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each analyte.

4.10.5 Advisory Committee

There is no formal advisory committee for this type of PTs.

5 Annex 1 – Scope of Accreditation (ISO/IEC 17043)

Environment

| SERVICE | PRODUCT | DETERMINANT |
|--|---|---|
| Proficiency testing scheme for biological composition | Waters | Photosynthetic pigments |
| Proficiency testing scheme for chemical composition, residues and contaminants | Sediments; Soils, Clays, Compost, Sludges, Biosolids, Waters | Aliphatic hydrocarbons; Aromatic hydrocarbons; Chemical and physical properties; Halogenated compounds; Hormones; Inorganic non-metallic compounds; Per- and polyfluoroalkyl substances (PFAS); Pesticides; Phenols; Phthalates; Semi-volatile organic compounds (SVOCs); Trace elements; Volatile organic compounds (VOCs) |

Food and Beverahe

| SERVICE | PRODUCT | DETERMINANT |
|--|--|--|
| Proficiency testing scheme for chemical composition, residues and contaminants | Beverages; Food products; Food surrogates; Wine | Allergens; Chemical and physical properties; Halogenated compounds; Hormones; Inorganic non- metallic compounds; Mycotoxin; Per- and polyfluoroalkyl substances (PFAS); Pesticides; Phenols; Phthalates; Semi-volatile organic compounds (SVOCs); Trace elements; Vitamin |

Healthcare, Pharmaceutical and Media Products

| SERVICE | PRODUCT | DETERMINANT |
|--|-----------------------------|--|
| Proficiency testing scheme for pharmaceuticals | Pharmaceitucal preparations | Active pharmaceutical ingredients; Contaminants; Trace elements |

Legal

| SERVICE | PRODUCT | DETERMINANT |
|---|---|---|
| Proficiency testing scheme for controlled substances | Powders containing illicit drugs; Wipes | Powders containing illicit drugs; Wipes |

6 Revision/Review History

| Date | Issue Number | Reasons for revision |
|----------------|--------------|--|
| April 2006 | 1.0 | First issue after move to NSW |
| August 2006 | 1.1 | NATA audit |
| November 2007 | 1.2 | Internal audit |
| March 2009 | 2.0 | Internal audit |
| December 2010 | 3.0 | Complete revision for ISO 17043 |
| August 2012 | 3.1 | Changed Pymble to NMI North Ryde |
| September 2012 | 3.2 | Internal audit |
| October 2012 | 3.2.1 | NATA audit |
| February 2014 | 3.3 | Review and minor alterations |
| April 2014 | 3.4 | Info about subcontracting in Sect 2.1. |
| September 2016 | 4.0 | Complete revision. |
| January 2019 | 4.1 | Section 4.5 and 4.6 renamed |
| March 2019 | 4.2 | Added chlorophyll a and wipes |
| July 2020 | 4.3 | Updated Department Logo on title page and CRV Manager phone number (Section 2.2). |
| January 2021 | 4.4 | Review and minor alterations |
| June 2021 | 4.5 | Added a section on subcontracting (Section 2.5). Added information regarding results deadline adjustments (Section 2.11). |
| March 2023 | 4.6 | Added information regarding Rapid Studies (Section 2.6, 2.9, 2.12). Added information regarding Preliminary Reports. Added a section on Rapid Studies (Section 4.10). |
| November 2023 | 4.7 | Minor amendments throughout. |
| April 2024 | 4.8 | Information regarding copyright added and minor updates and amendments throughout. Added Annex 2. |
| October 2024 | 4.9 | Added information regarding Rapid Studies (Section 2.3, 2.7, 2.8, 2.10, 2.14, 2.16 and 4.10). |