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National Measurement Institute

Study Protocol for Proficiency Testing

NMI North Ryde – Chemical Reference Values

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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.

2.2 Scheme Coordinator

Manager Chemical Reference Values

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Email: proficiency@measurement.gov.au

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

2.3 Nature and Purpose of the Scheme

The purpose of the NMI studies is to:

- enable laboratories to assess the accuracy of their test results and hence the effectiveness of their test methods 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.

2.4 Participation Fees and Charges

For most studies, participation is open to all laboratories. Participation in controlled drug studies is restricted to bona-fide forensic laboratories that can provide appropriate licenses and permits.

Typical rounds include between fifteen and forty laboratories from Australia and overseas.

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 – see Price List Work Instructions 12).

2.5 Subcontracting

Analytical testing associated with the PT program such as provision of homogeneity and stability testing may be subcontracted from time to time to a competent provider.

2.6 Planning of a PT study

CRV Manager provides a list of PT programs to be conducted yearly. This list is published on the NMI Website.The list with the options available for the NMI Rapid Studies (studies that are not run on pre-defined schedules) is also published on the NMI website (https://www.industry.gov.au/national-measurement-institute/chemical-and-biological-measurement-services/proficiency-testing-services).

2.7 Test Samples

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

2.8 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.

2.9 Information to Participants

The invitation letter for each study round includes details of:

- key dates,
- fees,
- participation form for enrolling in the round.

Participants are allowed approximately four weeks to perform the analysis.

Participants in Rapid Studies are asked to report results at their earliest convenience. These studies are designed by participants based on their stringent requirements.

Sample dispatch documents include

- instruction for analysis,
- deadline for returning results,
- electronic results sheet, and
- list of possible analytes.

2.10 Methods of Analysis

Participants are usually requested to use their normal method of analysis and report a single result, together with an associated uncertainty.

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

2.11 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 (eg pandemic) affecting some or all participants, and/or NMI.

An individual participant may require extension of TAT by email. The NMI PT team will assess each request on a case-by-case basis. An extension of up to 4 business days may be granted. It is expected that the interim report issue date will not be adjusted for this circumstance.

2.12 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:

- 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 ISO13258, "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 NMI Rapid PT Studies the assigned value is a pre-set assigned value, an assigned value set in a previous study.

2.13 Target Standard Deviation for Proficency 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 asessment 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
- an estimate from expectations based on experience
- experience from previous studies.
- an estimate from a statistical model (e.g. Horwitz-Thompson model).

Examples for various studies are presented in Annex 1.

2.14 Statistical Analysis

The statistical treatment used by CPT to assess laboratory performance is based on the procedures described in the ISO13258,[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 En-scores.

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

2.15 Reports and Performance Evaluation

An Interim Report is issued within five working days of the due date for submission of results. The primary purpose of this report is to provide each participant with an early indication of performance. The interim report is distributed by e-mail 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 in eight weeks of the closing date for submission of results. The final report includes:

- Summary;
- Introduction - general description and study objectives;
- Test items - test sample preparation, homogeneity and stability testing;
- Summary of test participants' methods;
- Explanation of result presentation, definition of terms and statistics used (z-scores and E_n-scores);
- Laboratory codes and test results;
- Presentation of Results - results tables including assigned values, statistical summary data, histograms of results, z-scores and En-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.
- Reports are emailed to participants together with the individual Performance Summary report.

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).

2.16 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. 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.

2.17 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 expunged 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, https://www.legislation.gov.au/Series/F2015L00468

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

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.

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

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

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 terms of reference of the group are identical to those in ISO/IEC 17043 clause 4.4.1.4 and 4.4.1.5.[1] The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time.

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 (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 asessment may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

Table 2 Performance Coefficient of Variation for Controlled Drugs

Sample	Analyte	Assigned Value (% base (m/m))	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)
S1	MDMA	63.0	1.3	3
S2	MDMA	31.4	1.8	3
S3				
S4	Methamphetamine	45.3	1.5	3

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.

Table 3 Performance Coefficient of Variation for Controlled Drugs in Wipes

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

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 NMI 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.

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

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

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 terms of reference of the group are identical to those in ISO/IEC 17043 clause 4.4.1.4 and 4.4.1.5.[1] The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time.

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.

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

Sample	Analyte	Assigned value (mg/kg)	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)
S1	>C10-C16	1590	5.3	20
	>C16-C34	2520	4.9	20
	>C34-C40	317	6.7	20
	TRH	4400	4.5	15

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 asessment may vary sometimes with the concentration of the analyte in the sample or difficult matrices.

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

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%

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

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 terms of reference of the group are identical to those in ISO/IEC 17043 clause 4.4.1.4 and 4.4.1.5.[1] The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time.

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.

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

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	Ba	3.62	13%	15%
S1	Ca	1830	5.2%	10%

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

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 terms of reference of the group are identical to those in ISO/IEC 17043 clause 4.4.1.4 and 4.4.1.5.[1]The study coordinator may seek advice from members of the group about a particular study round or other issue from time to time.

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 terms of reference of the group are identical to those in ISO/IEC 17043 clause 4.4.1.4 and 4.4.1.5.[1] 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 5 - 50 g sieved and homogenised biota and/or food test samples
- two 10 g sieved and homogenised soil test samples
- two 2x50 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.

Table 8 Performance Coefficient of Variation for PFAS

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

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 be run on a pre-defined schedule; laboratories can request this type of study at any time (e.g. if they have an audit, validate methodology or check the performance of a new analyst). Participants have the option to design their own study.

4.10.1 Test Sample

Each round includes one sample from a previous study. The sample may contain either spiked or incurred analytes. Participants select the matrix and the analytes (up to 6) from a list with available options.

4.10.2 Test Sample Transport

The test sample is sent by courier packaged to prevent losses 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 (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.

4.10.3 Target Standard Deviation for Proficiency Asessment

The target standard deviation for proficiency assessment used for calculation of z-scores is the same as the PCV used in the original study. PCVs in the rapid studies might need to be adjusted to account for any changes in the analytes' level over time as per ISO 13528 instructions clause 6.1.[2]

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 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).