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Organochlorine pesticides in water

Proficiency testing scheme

Scheme description

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1 Foreword

This is the official call for participation in the NMISA proficiency testing scheme (PTS) for the determination of organochlorine pesticides (OCPs) in water following the trial PTS conducted in July 2012. A confidential report will be issued to all participants after completion of the PTS. Dates for the OCPs in water PTS are listed in Table 1.

This forms part of a range of services offered by NMISA. Current proficiency testing schemes offered by the NMISA Organic Analysis Laboratory include:

- Organochlorine pesticides in water
- Aqueous ethanol (forensic blood alcohol analysis)
- Aqueous ethanol (alcohol content in beverages)
- Aqueous sodium fluoride (blood sample preservative)

The NMISA will be expanding its offerings towards the end of 2013/2014 to include:

- Organophosphorus pesticides in water

The NMISA can also assist with the preparation of traceable gravimetrically prepared spike solutions for benchmarking *ad-hoc* analyses for which commercial PTSs are not available.

2 Scheme aims

This scheme will assist laboratories that routinely analyse OCPs in water to monitor their laboratory performance. This covers aspects such as the accuracy and comparability of measurement results produced; the continued competency of analytical staff and the maintenance and effectiveness of the current quality assurance systems within the laboratory. In addition, this information may also be used to provide accreditation bodies or clients with objective evidence of laboratory performance.

A technical advisory forum, incorporating members from industry, will be established through the NMISA. The aim of this forum will be to provide guidance on measurement challenges, traceability and estimation of measurement uncertainty for this analysis.

3 Participation fees and additional charges

The cost of participation in each round of the PTS is R1 500.00 (0% VAT). This fee includes two 1 litre water samples that have been gravimetrically spiked with a selection of organochlorine pesticides listed in Table 2, together with a confidential report upon completion of the round. Please note, this cost excludes additional analytical standards and delivery fees.

Seven South African water testing laboratories have indicated interest in the PTS. Since most are located within close proximity to the NMISA, the option of collecting the PTS samples from NMISA premises will be permitted.

International laboratories will have test samples sent by courier and appropriately packaged to maintain sample integrity. International participants must provide NMISA with any import or quarantine permits that might be required to complete sample delivery well in advance of the shipment date. Please note, international participants are liable for any customs or import duties charged.

Upon registration for participation an official quotation will be provided. A registration form should accompany this document.

4 Scheme distribution dates

Table 1 Organochlorine pesticide in water PTS sample distribution dates

| Round | Registration deadline | Despatch Date | Result submission Deadline | Samples |
|-------|-----------------------|------------------|----------------------------|---|
| 1 | 18 February 2013 | 25 February 2013 | 18 March 2013 | 1. 2 x 1 L water samples 2. Gravimetrically diluted analytical standard (optional) |
| 2 | 19 August 2013 | 26 August 2013 | 16 September 2013 | 1. 2 x 1 L water samples |

5 Test samples

The PTS round consists of:

- Two 1 litre samples of purified water spiked with a selection of OCPs that are listed in Table 2. No dilution of the samples is required.
- OPTIONAL:
At an additional cost, the NMISA can provide a gravimetrically diluted mixture of organochlorine pesticides in appropriate solvent, which laboratories may use for quantification of the PTS samples, or to verify their own in-house standards.

The organochlorine pesticides listed in Table 2, are those which are currently being tested by laboratories in South Africa¹. The listed concentration range encompasses the recommended WHO concentration limits for these analytes in drinking water² and/ or from

¹ NMISA Survey: Water PTS for organic contaminants, *M. Fernandes-Whaley*, March 2012

² WHO Guidelines for drinking water quality, 2011, (4th Edition), ISBN 978 92 4 154815 1

the South African water standard for protection of aquatic ecosystems³. These levels should be achievable using analytical methods typically applied (GC-MS or GC-ECD) for quantification of OCPs¹.

Studies conducted during the trial PTS confirmed homogeneity. Additional checks will be performed to detect any unforeseen problems that may arise during bottling and storage. This information will be included in the PTS report.

Sample preparation instructions will accompany the PTS samples. Participants should adhere to these instructions, particularly concerning sample storage conditions and time frames within which samples should be analysed.

6 Proficiency testing scheme assigned value

The assigned value for the PTS will be the purity corrected gravimetric values obtained during the formulation preparation.

Table 2 Organochlorine pesticides in water PTS sample list of analytes and expected concentration ranges

| Analyte | Concentration range | | unit |
|---------------------------------|---------------------|-----|------|
| | Max | Min | |
| Aldrin | 50 | 10 | ng/L |
| <i>cis</i> -chlordane | 50 | 10 | ng/L |
| <i>trans</i> -chlordane | 50 | 10 | ng/L |
| p,p'-DDT | 120 | 20 | ng/L |
| o,p-DDT | 120 | 20 | ng/L |
| p,p'-DDE | 120 | 20 | ng/L |
| p,p'-DDD | 120 | 20 | ng/L |
| Dieldrin | 50 | 10 | ng/L |
| Endosulfan I (alpha-endosulfan) | 300 | 100 | ng/L |
| Endosulfan II (beta-endosulfan) | 300 | 100 | ng/L |
| Endosulfan sulphate | 300 | 100 | ng/L |
| Endrin | 120 | 20 | ng/L |
| Heptachlor | 50 | 10 | ng/L |
| Heptachlor epoxide | 50 | 10 | ng/L |
| Alpha HCH | 120 | 20 | ng/L |
| Beta HCH | 120 | 20 | ng/L |
| Delta HCH | 120 | 20 | ng/L |
| Gamma HCH (Lindane) | 120 | 20 | ng/L |

³ Department of Water Affairs and Forestry (1996b). *South African Water Guidelines. Vol. 7: Aquatic Ecosystems* (1st Edition). DWAF: Pretoria.

7 Analysis

Participants are encouraged to perform the analysis using their normal laboratory procedures.

An electronic result submission form will be sent to participants when samples are delivered/collected.

Participants are expected to submit one result for each pesticide tested per sample, and will be expected to include an uncertainty estimate for each result obtained.

Where applicable, participants must report whether a recovery correction was applied.

8 Reporting

The data will be processed according to ISO 13528:2005 (Statistical methods for use in proficiency testing by interlaboratory comparisons). A report will be issued to each participating laboratory, 3 weeks following the submission deadline.

Reports will be provided in electronic format only (Adobe Acrobat- pdf) files.

The scheme is fully confidential. Each participant will be issued with a unique identification number, which will change for every round. The participating laboratory is required to identify its analysts by a code known only to the laboratory.