Study protocol for proficiency testing

Organic contaminants in drinking water

NMISA-PT-69

2021/22

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Foreword

The National Metrology Institute of South Africa (NMISA) was established under the Measurement Units and Measurement Standards Act No 18 of 2006. The NMISA is committed to supporting laboratories through the provision of proficiency testing schemes (PTs) that afford participating laboratories the opportunity to regularly demonstrate their continued analytical measurement competence. NMISA is an accredited proficiency testing scheme provider and the pesticides in water PTS is included in out accreditation scope.

Please see the PTS registration form for the proficiency testing schemes on offer in the following year including PTSs for pesticides, mycotoxin, heavy metal, food labelling and forensic blood alcohol testing

The NMISA provides a confidential service to participants that allows a laboratory to assess the accuracy of their test results using their routine laboratory methodologies, thereby testing the effectiveness of their methods and quality assurance programs. The provided PT report is generated to assist laboratories in identifying areas of improvement within their current quality system.

The current study protocol has been designed to support routine analytical laboratories testing organic contaminants in drinking water. The focus of this round will be pesticides and polycyclic aromatic hydrocarbons (PAHs).

Scheme provider

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Participation fees and charges

The cost of participation in the PT scheme is R 3 000.00 excluding delivery fees (0% VAT, please note that we are not a VAT registered company). This fee includes two 1 litre samples of purified water spiked with a selection (minimum of six) of the organic contaminants listed in **Table 1**. No dilution of the samples is required. For more than one participant per laboratory an additional participation fee will be levied, and additional material supplied. After conclusion of the PT round, a report will be issued. Please find registration form attached as an addendum to this document, for participation an official quotation will be provided. Transport costs are not included in these fees please refer to the section "Test sample transport" below. Upon registration for participation an official quotation will be provided

Reports provided to participants

Participant results, in the form of z-scores, will be issued to each participant as an indication of their performance in the PT within one week of the result submission deadline. A full PT report will be issued to each participating laboratory within one month of the submission deadline. The report will contain the following information: description of the material used and how it was prepared; verification of target values, a summary of participating laboratories analytical techniques and data evaluation. The PT assigned value will be determined using participant consensus or using the NMISA reference values depending on the number of participant results received.

Statistical analysis

Participant performance will be evaluated against the assigned value for each parameter. The assigned value for the PTS will be the gravimetric preparation or consensus values determined from participant results in accordance with ISO 13528:2015 statistical principals. For consensus results a minimum of five laboratories need to submit duplicate results.

A z-score will be used to determine the individual laboratories performance based on the following equation:

$$z = \frac{x_{laboratory} - x_{PT\ value}}{\sigma}$$

Where:

$x_{laboratory}$ = the result reported by the participant

$x_{PT\ value}$ = the PT value (NMISA reference value or participant consensus value)

σ = the standard deviation for the PT

The target standard deviation used to calculate the z-score will be based on the modified Horwitz performance model, where the maximum measurement result variation expected between laboratories at these concentration levels is 22%.

Scheme details

Test samples

The PT scheme consists of two 1 litre samples of purified water spiked with a minimum of six of the organic contaminants, listed in Table 1. The concentration range of contaminants will be between 10 – 5000 ng/ L.

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

Table 1: List of analytes possibly present in the drinking water PT

Analyte	**CAS nr**	**Analyte**	**CAS nr**
2-Phenylphenol	90-43-7	Gamma HCH (Lindane)	58-89-9
Alachlor	15972-60-8	Heptachlor	76-44-8
Aldrin	309-00-2	Heptachlor epoxide	1024-57-3
Alpha HCH	319-84-6	Imazalil	35554-44-0
Anthracene	120-12-7	Imidacloprid	138261-41-3
Atrazine	1912-24-9	Indeno[1,2,3-cd]pyrene	193-39-5
Azoxystrobin	131860-33-8	Kresoxim methyl	143390-89-0
Benzo[a]pyrene	50-32-8	lambda-Cyhalothrin	91465-08-6
Benzo[b]fluoranthene	205-99-2	Linuron	330-55-2
Benzo[g,h,i]perylene	191-24-2	Methamidophos	10265-92-6
Benzo[k]fluoranthene	207-08-9	Methomyl	16752-77-5
Beta HCH	319-85-7	Metolachlor	51218-45-2
Bifenthrin	82657-04-3	Metribuzin	21087-64-9
Bromacil	314-40-9	Molinate (Ordram)	2212-67-1
Butachlor	23184-66-9	Nitempyram	150824-47-8
Carbaryl (Sevin)	63-25-2	*o,p'*-DDD	53-19-0
Chlorothalonil	1897-45-6	*o,p'*-DDT	789-02-6
Chlorpyrifos	2921-88-2	*p,p'*-DDD	72-54-8
Chlorpyrifos methyl	5598-13-0	*p,p'*-DDE	72-55-9
cis-Chlordane	5103-71-9	*p,p'*-DDT	50-29-3
cis-Permethrin	61949-76-6	Procymidone	32809-16-8
Clothianidin	210880-92-5	Prometon	1610-18-0
Cyanazine	21725-46-2	Prometryn	7287-19-6
Cyprodinil	121552-61-2	Propachlor	1918-16-7
Delta HCH	319-86-8	Pymetrozine	123312-89-0
Desethylatrazine	6190-65-4	Pyrene	129-00-0
Diazinon	333-41-5	Simazine	122-34-9
Dichlorvos (DDVP)	62-73-7	Tebuconazole	107534-96-3
Dicofol (Kelthane)	115-32-2	Thiabendazole	148-79-8
Dieldrin	60-57-1	Thiacloprid	111988-49-9
Endosulfan I (alpha-endosulfan)	959-98-8	Thiamethoxam	153719-23-4
Endosulfan II (beta-endosulfan)	33213-65-9	Thiobencarb	28249-77-6
Endosulfan sulfate	1031-07-8	Tolylfluanid	731-27-1
Endrin	72-20-8	*trans*-Chlordane	5103-74-2
Ethion	563-12-2	*trans*-Permethrin	61949-77-7
Fluoranthene	206-44-0	Trifluralin	1582-09-8

Test sample transport

Samples will be packaged and transported in a manner to minimise deterioration of the sample in transit. Transport costs are calculated depending on the location of the participating laboratory and are therefore not included in the PT cost price. Upon registration, a quotation will be issued including transport costs. Local laboratories may collect the sample from the NMISA premises.

For international laboratories, please note that any import or quarantine permits remains the responsibility of the participating laboratory and must be submitted to the NMISA prior to the shipment date. Participants are accountable for all customs and import duties.

All PT material shall be delivered and collected at the Applicant's own risk. The NMISA will not take responsibility for samples damaged during transport, although all due care will be exercised during packing to prevent this from occurring.

Methods of analysis

Participants are encouraged to perform the analysis using their normal laboratory procedures. Please note that the concentration levels of this PT are very low. Care should be taken with sample size and preconcentration techniques used to ensure instrument LOQ is exceeded. Measurement at these levels is typically achievable using chromatography with mass spectrometric analysis. An electronic result submission form will be sent to participants when samples are delivered/ collected.

Information required for reporting

An electronic result submission form will be sent to participants when samples are delivered/ collected. For each participant two results per pesticide/ PAH per sample should be submitted. Participants are encouraged to include an uncertainty estimate for each result obtained. Where applicable, participants must report whether a recovery correction was applied.

The following information will be requested from participating laboratories:

- Method validation information
- Quality control measures implemented
- Method of extraction used
- Sample size analysed
- Instrumentation specification
- Analytical method information
- Recoveries for method and if a correction for recoveries is applied
- The method limit of detection and limit of quantification

Scheme dates

In effort to improve our service a new automated system will be implemented allowing the initial z-score table to be issued within one week from the result submission deadline. Please note that no late submissions can therefore be accommodated.

Table 2 Organic contaminants in drinking water PT scheme sample distribution dates

Matrix	Sample Distribution	Reporting	Samples
Purified water	November 2021	December 2021	2 x 1 L

NMISA PROFICIENCY TESTING SCHEMES

REGISTRATION FORM

PLEASE COMPLETE AND RETURN BY FAX OR E-MAIL TO pt@nmisa.org

Send test material to:	
Name	
Company	
Department / Laboratory	
Physical address	
Town/City	
Postal code	
Country	
Telephone	
Email	
Send PT report to (e-mail address):	
Name	
Email	
Send invoices to (e-mail address):	
Name	
Telephone	
Email	

I,_____ herewith confirm that a NMISA Customer Registration Information Form has been completed and submitted to NMISA for official quotation and invoicing purposes. It is understood that **registration for the proficiency testing scheme will only be confirmed upon receipt of a purchase order or EFT proof of payment.** Customs clearance and duties for international participants is for the customer's account.

_____ _____

Signature Date

PT Schemes available from the NMISA in 2021/22

Dispatch date	PT Scheme No.	PT Scheme Description	Approx. Sample Size	Registration fee*
Mycotoxins				
Oct 2021	NMISA-PT-65	Aflatoxin M1 in milk	50 mL	R 3 000.00
Jan 2022	NMISA-PT-66	Mycotoxins in feed/grain	100 g	R 3 000.00
Pesticides				
Jun 2021	NMISA-PT-67 Round 1	Pesticides in oranges	50 g + 50 g blank	R 3 800.00
Nov 2021	NMISA-PT-67 Round 2	Pesticides in apples	50 g + 50 g blank	R 3 800.00
Nov 2021	NMISA-PT-67 Round 3	Pesticides in strawberries	50 g + 50 g blank	R 3 800.00
Dec 2021	NMISA-PT-67 Round 4	Pesticides in apricots	50 g + 50 g blank	R 3 800.00
Mar 2022	NMISA-PT-67 Round 5	Pesticides in peaches	50 g + 50 g blank	R 3 800.00
Antibiotic drug residues				
Nov 2021	NMISA-PT-68	Antibiotic drug residues in chicken	50 g + 50 g blank	R 5 000.00
Water analysis				
Nov 2021	NMISA-PT-69	Contaminants in water	2 x 1 L	R 3 000.00
Nutritional Content/ Food labelling				
Sept 2021	NMISA-PT-75	Elements, proximates and amino acids in fortified milk powder	2 x 20 g	R 3 500.00
Jan 2022	NMISA-PT-70	Vitamin A palmitate, nutritional elements in a fortified food matrix	100 g	R 4 000.00

Dispatch date	PT Scheme No.	PT Scheme Description	Approx. Sample Size	Registration fee*
Forensic Alcohol Analysis				
Jun 2021	NMISA-PT-71 Round 1	Forensic Blood Alcohol testing: Ethanol	3 levels x 25 mL each	R 6 615.00
Aug 2021	NMISA-PT-71 Round 2	Forensic Blood Alcohol testing: Ethanol	3 levels x 25 mL each	R 6 615.00
Jan 2022	NMISA-PT-71 Round 3	Forensic Blood Alcohol testing: Ethanol	3 levels x 25 mL each	R 6 615.00
Sept 2021	NMISA-PT-72	Forensic Blood preservation: Sodium fluoride	2 levels x 100 mL each	R 4 000.00
June 2021	NMISA-PT-73	Breathalyser calibration using waterbath method	1 level x 4 x 500 mL	R 7 000.00
	NMISA-PT-74	Beverage alcohol content (on request)		

Register for these PTs by visiting our on-line store or sending us an e-mail at pt@nmisa.org

*Please note that the registration fee excludes delivery charges.