

Study protocol for proficiency testing

Pesticides in fruit 2021/22

Table of Contents

Foreword.....	3
Proficiency testing program	6
Scheme provider	6
Scheme Co-ordinator	6
Participation fees and charges	6
Reports provided to participants	6
Statistical analysis	7
Scheme details	7
Test samples	7
Test sample transport	8
Methods of analysis	8
Information required for reporting	8
Scheme dates	8
References	9

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 fruit PTS is included in our accreditation scope.

Please see the PTS registration form for the proficiency testing schemes on offer in the following year including PTSs for 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 pesticide residues in agricultural commodities. The PT will be used to assess the various matrices represented in the AOAC food composition triangle (**Figure 1**) over the course of five years. The matrices selected for this year are plums (stone fruit); pears (pome fruits) sweet peppers (fruiting vegetables, other than cucurbits); litchi and banana (assorted tropical and sub-tropical fruits – inedible peel).

Specific attention has been paid to tailoring the scheme to current export limits as well as maximum residue levels for the South African market. The target pesticide and commodities were selected to match the growth season within South Africa. Pesticides selected for analysis in this PT represents various chemical classes associated with the specific commodity selected. Thereby accommodating the routine analysis performed by the majority of laboratories at the time the proficiency test material is circulated.

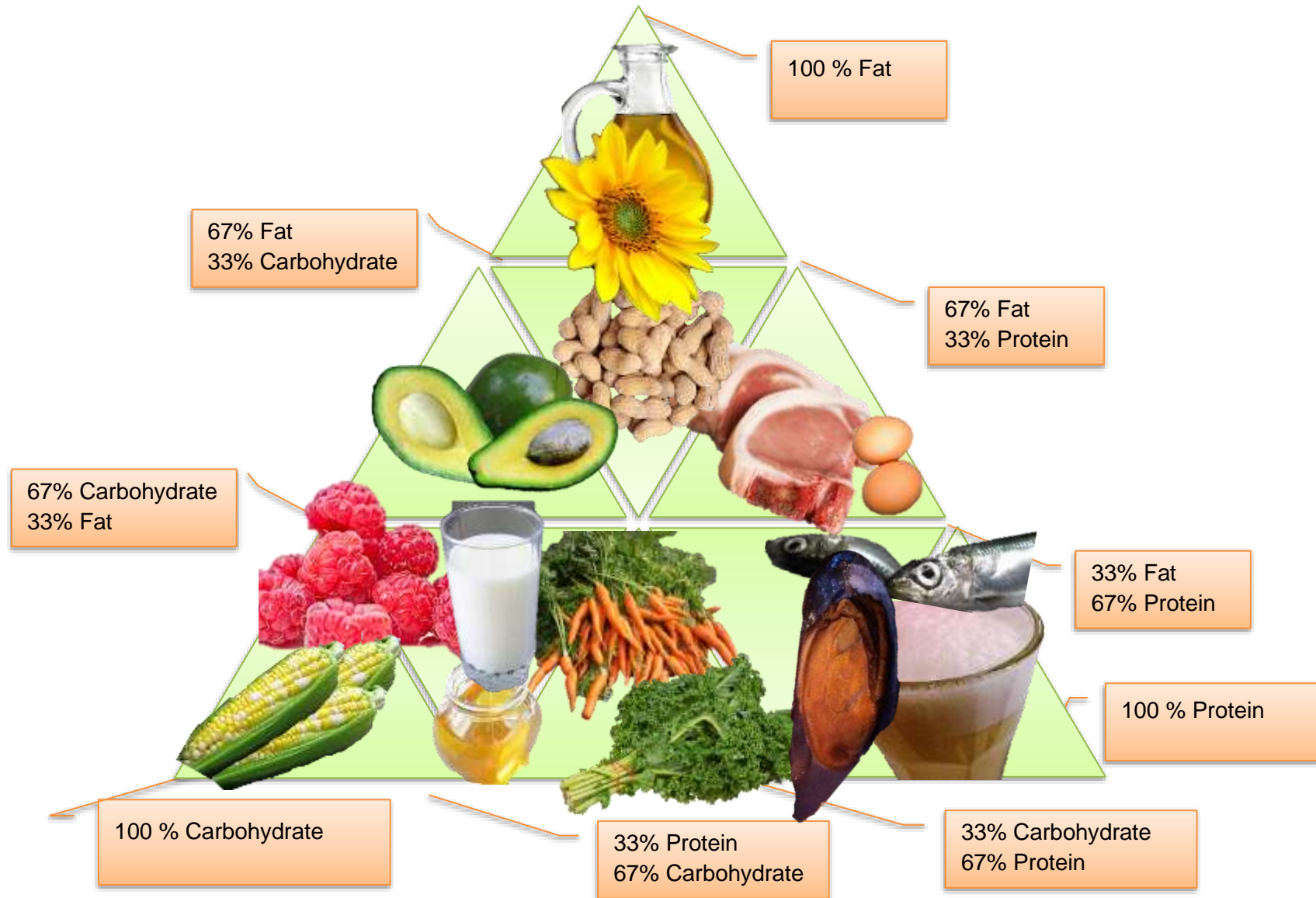


Figure 1: The AOAC food composition triangle (modified from Phillips *et al.*, 2013)

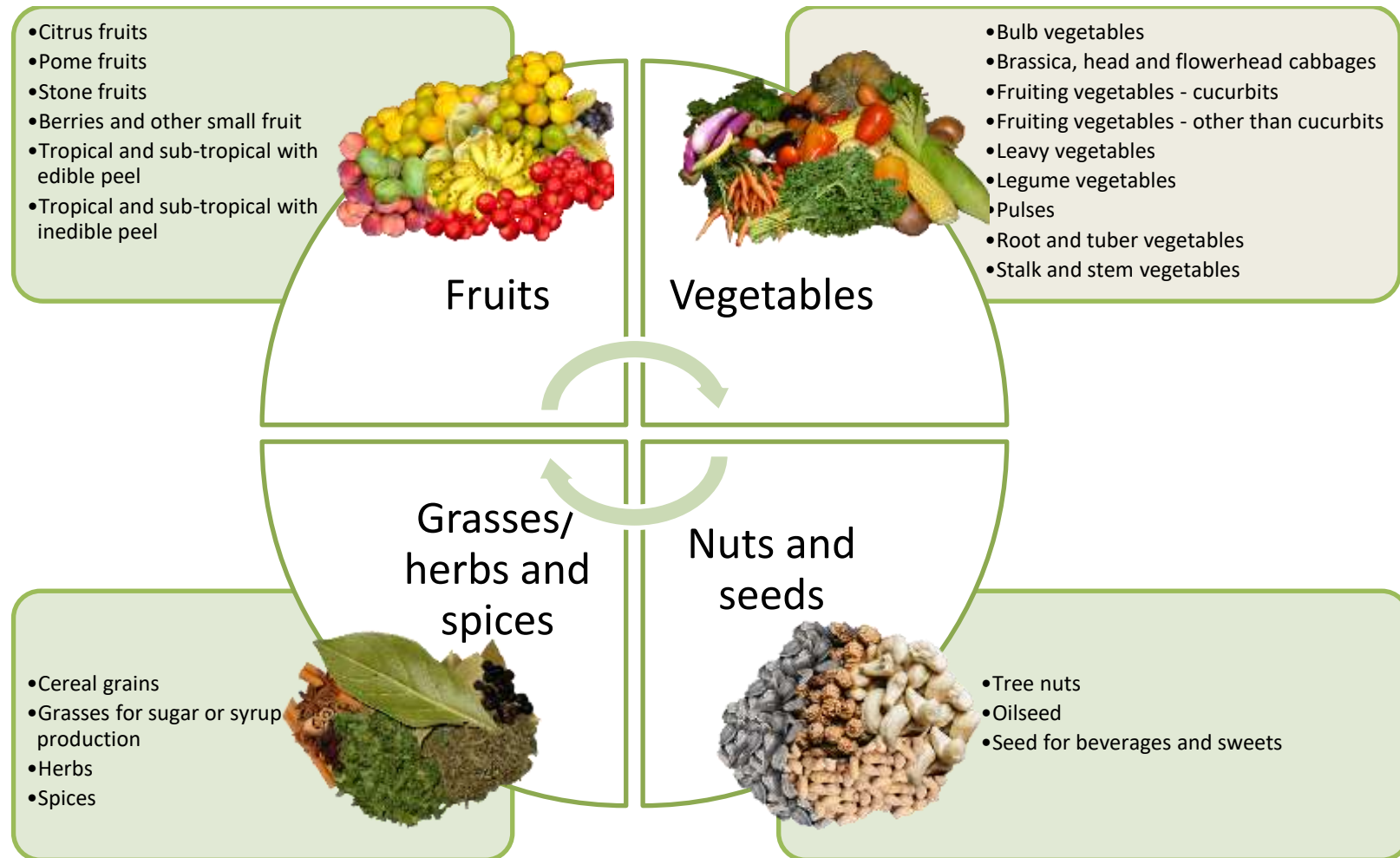


Figure 2: A summary of class A commodities, primarily of plant origin (modified from Codex *Alimentarius*, 2016)

Proficiency testing program

Scheme provider

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Participation fees and charges

The cost of participation has been significantly reduced to remain competitive and accessible. Participation in one round of the PT scheme is now only R 3800.00 excluding delivery (0% VAT, please note that we are not a VAT registered company) **Table 1**. This fee includes two 50 g portions of test sample and a blank sample of 50 g. 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.

Table 1: Summary of participation fees

Participation	Participation fees
Single round	R 3 800.00

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 value will be determined using participant consensus or using the gravimetrically spiked values depending on the number of participant results received.

Statistical analysis

The participant data will be processed according to ISO 13528:2005 (Statistical methods for use in proficiency testing by interlaboratory comparisons). 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 Horwitz performance model, where the maximum measurement result variation expected between laboratories is 22%.

Scheme details

The agricultural commodities and pesticide residues selected for this proficiency testing scheme have been selected based on routinely tested seasonal commodities and their regulated pesticide residue limits, respectively. Concentration levels range from below the regulated export limits to above the maximum residue limit for South Africa.

Test samples

The test samples will consist of a homogenised raw fruit sludge that has been spiked with a known concentration of **selected pesticides** from the pesticide list described in **Table 2**. For each round a participant will be provided with two 50 g portion of test sample. A portion of the un-spiked commodity that may contain pesticide residues not spiked in the round will also be provided to be used as a blank control.

Table 2: List of possible matrix specific pesticides included within the 2021/22 proficiency testing scheme.

Pesticide	Pesticide	Pesticide	Pesticide
2,4 D	Cyprodinil	Hexythiazox	Propyzamide
Abamectin	Dichlorprop	Indoxacarb	Prothiophos
Acetamiprid	Difenoconazole	Isopyrazam	Pymetrozine
Atrazine	Dimethoate	Isoxaben	Pyraclostrobin
Azoxystrobin	Dioxathion	Kresoxim-Methyl	Pyrimethanil
Bifenazate	Diphenylamine	Mepanipyrim	Quinoxifen
Boscalid	Etoxazole	Methomyl	Tebuconazole
Bromopropylate	Fenarimol	Methoxyfenocide	Tetradifon
Captan	Fenoxycarb	Metrafenone	Thiacloprid

Pesticide	Pesticide	Pesticide	Pesticide
Carbaryl	Fenthion	Omethoate	Triadimefon
Chinomethionat	Flubendiamide	Penthiopyrad	Triadimenol
Clopyralid	Flurochloridon	Piperonyl butoxide	Trichlorfon
Coumaphos	Flutriafol	Pirimicarb	Triforine
Cyantraniliprole	Folpet	Procymidone	Vinclozolin
Cyhalothrin (Lambda)	Haloxyfop		

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 requested to use the methods/ procedures used during routine sample analysis.

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 per sample should be submitted.

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 submission can therefore be accommodated.

Table 3: Pesticide in fruit PT scheme important dates

Round	Matrix	Sample distribution	Reporting
1	Oranges	June 2021	August 2021
2	Apples	November 2021	January 2022
3	Strawberries	November 2021	January 2022
4	Apricots	December 2021	February 2022
5	Peaches	March 2022	April 2022

References

- F. Cordeiro. Statistical methods for use in proficiency testing (2009). Institute for Reference Materials and Measurements. JRC –EC
- M.M. Phillips, K.E. Sharpless and S.A. Wise. Standard reference materials for food analysis (2013). *Analytical and Bioanalytical Chemistry*, 405: 4325 – 4335
- Codex Alimentarius International Food Standards: Codex pesticides residues in food online database (July 2016).

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**. 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.