

Study protocol for proficiency testing Pesticides in fruit 2020/21

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	9
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.

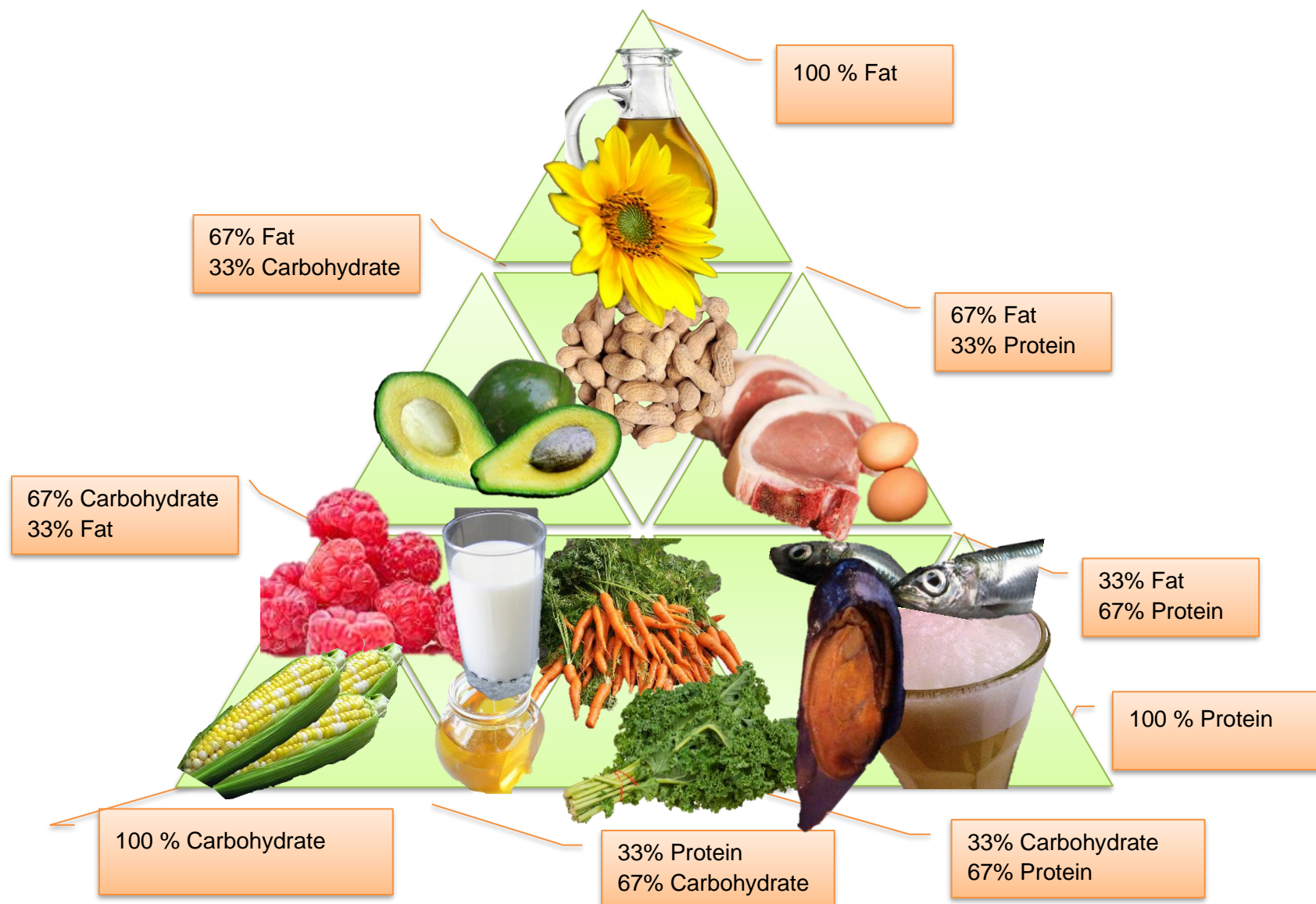


Figure 1: The AOAC food composition triangle (modified from Phillips *et al.*, 2013)

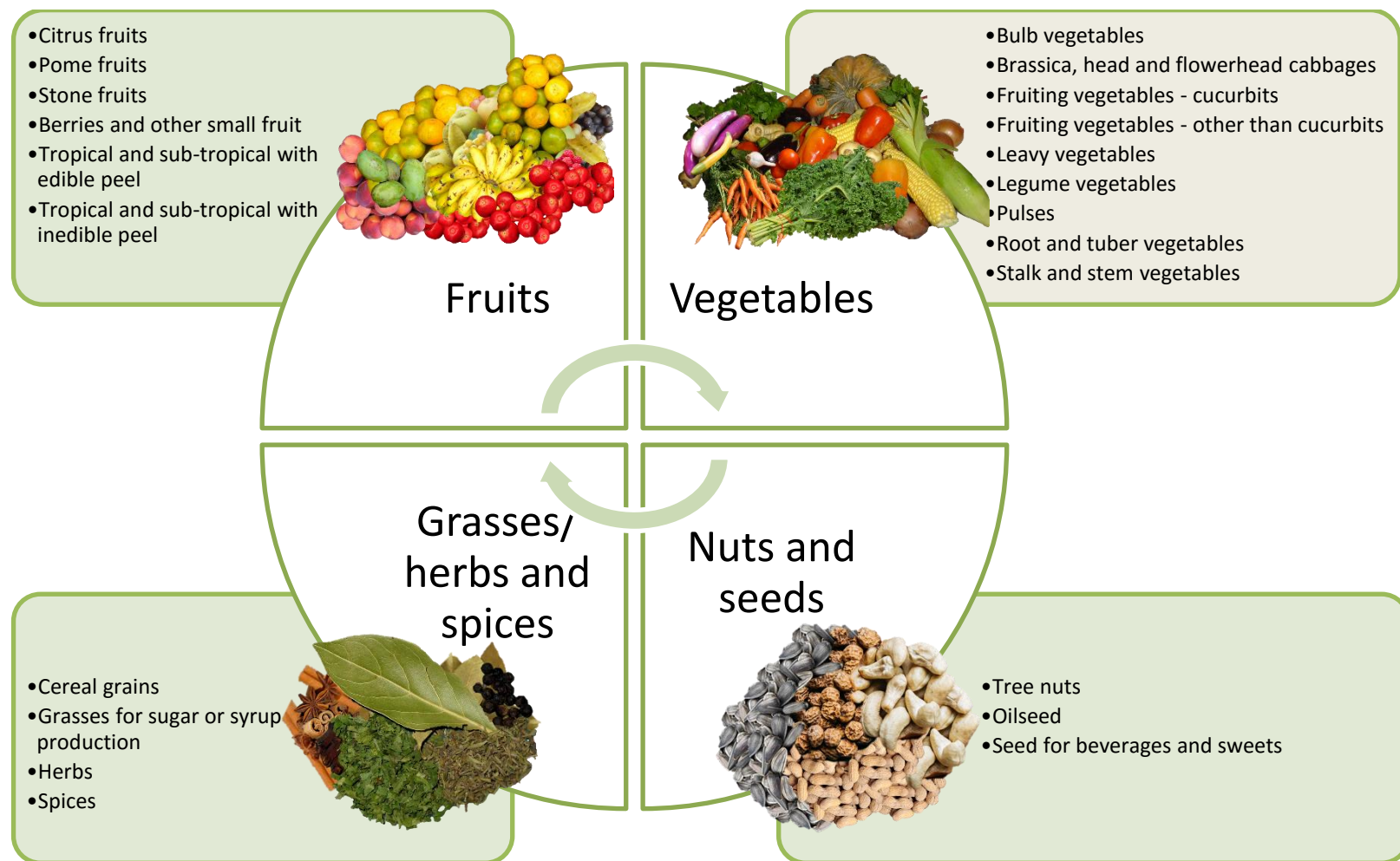


Figure 2: A summary of class A commodities, primarily of plant origin (modified from Codex *Alimentarius*, 2016)

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 3000.00 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 3 000.00

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 2020/21 proficiency testing scheme

Pesticide	Pesticide	Pesticide
2-phenylphenol	Ethirimol	Metconazole
Acephate	Etofenprox	Methiocarb
Acequinocyl	Etoxazole	Myclobutanil
Acetamiprid	Etridiazole	Napropamide
Acibenzolar-S-methyl	Fenamiphos	Oxamyl
Ametryn	Fenazaquin	p,p-DDE
Azinphos methyl	Fenbuconazole	Penconazole
Benomyl	Fenbuconazole	Penthiopyrad

Pesticide	Pesticide	Pesticide
Benzovindiflupyr	Fenbuconazole	Phosalone
Bupirimate	Fenbuconazole	Propamocarb
Chlorantraniliprole	Fenhexamid	Pyrimethanil
Chlordecone	Fenpyroximate	Pyriproxyfen
Clethodim	Fluopyram	Tebufenozide
Clofentezine	Flutriafol	Teflubenzuron
Clothianidin	Fluxapyroxad	Tetraconazole
Cyflufenamid	Hexythiazox	Thiabendazole
Cyflumetofen	Imazalil	Thiacloprid
Deltamethrin	Imidacloprid	Trichlorfon
Diazinon	Indoxacarb	Triclopyr
Emamectin benzoate	Lufenuron	Triflumuron
Endosulfan sulphate		

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	Freeze-dried plums	March 2021	April 2021
2	Sweet peppers	July 2020	August 2020
3	Litchi	October 2020	November 2020
4	Banana	January 2021	February 2021
5	Pears	February 2021	March 2021

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 or EFT proof of payment.** Customs clearance and duties for international participants is for the customer's account

Signature

Date

Dispatch date	PT scheme no.	PT scheme description	Sample Size	Select PT	Delivery	Self-collection
Mycotoxins						
May 2020	NMISA-PT-63	Aflatoxins in peanut slurry	50 g			
Aug 2020	NMISA-PT-50	Aflatoxin M1 in milk	50 mL			
Jan 2021	NMISA-PT-51	Mycotoxins in cassava	50 - 100 g			
Pesticides						
Jan 2020	NMISA-PT-ORG41 Round 4	Pesticides in macadamia nuts	50 g + 50 g blank			
March 2021	NMISA-PT-53 Round 1	Pesticides in plums	50 g + 50 g blank			
July 2020	NMISA-PT-53 Round 2	Pesticides in sweet peppers	50 g + 50 g blank			
Sept 2020	NMISA-PT-54	Organic contaminants in drinking water	2 x 1 L			
Oct 2020	NMISA-PT-53 Round 3	Pesticides in litchie	50 g + 50 g blank			
Jan 2021	NMISA-PT-53 Round 4	Pesticides in banana	50 g + 50 g blank			
Feb 2021	NMISA-PT-53 Round 5	Pesticides in pears	50 g + 50 g blank			
Heavy metals						
Nov 2020	NMISA-PT-49	Toxic elements in cocoa powder	50 g			
Nov 2020	NMISA-PT-55	Toxic elements in maize flour	50 g			
Nutritional Content/ Food labelling						
August 2020	NMISA-PT-52	Corn-soya fortified food matrix (Vitamins, elements, proximates)	150 g			
Oct 2020	NMISA-PT-57	Capsaicin in spices/chilli	50 g			
Nov 2020	NMISA-PT-49	Nutritional elements in cocoa powder	50 g			
Nov 2020	NMISA-PT-55	Nutritional Elements in maize flour	50 g			
Feb 2021	NMISA-PT-58	Sugar content determination	100 - 200 g (mL)			
Mar 2021	NMISA-PT-59	Free Fatty Acids in peanut oil	50 - 250 mL			
Forensic alcohol analysis						
June 2020	NMISA-PT-60 Round 1	Forensic Blood Alcohol testing: Ethanol	3 levels x 25 mL each			
Aug 2020	NMISA-PT-60 Round 2	Forensic Blood Alcohol testing: Ethanol	3 levels x 25 mL each			
Jan 2021	NMISA-PT-60 Round 3	Forensic Blood Alcohol testing: Ethanol	3 levels x 25 mL each			
Sept 2020	NMISA-PT-61	Forensic Blood preservation: Sodium fluoride	2 levels x 100 mL each			
June 2020	NMISA-PT-62	Breathalyser calibration using waterbath method	1 level x 4 x 500 mL			
	NMISA-PT	Beverage alcohol content (on request)				