

Study protocol for proficiency testing

**Nutritional and toxic elements in
cocoa powder
NMISA-PT-49
2020/21**

Table of Contents

Foreword.....	3
Scheme provider	3
Scheme Co-ordinator	3
Participation fees and charges	4
Reports provided to participants	4
Statistical analysis	4
Scheme details	4
Test samples	4
Test sample transport	5
Methods of analysis	5
Information required for reporting	5
Scheme dates	6

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 food testing 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 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. A PT report is provided to assist laboratories in identifying areas of improvement within their quality system.

The current study protocol has been designed to support routine analytical laboratories testing nutritional and toxic elements in agricultural and food commodities. The analysis of elements in cocoa has gained increased attention over the last few years, due to presence of toxic elements such as cadmium that may have an impact on human health. The presence of these toxic elements can also affect inter- and intra-regional trade as specific requirements need to be met for import such as those summarised in the EU Commission Regulation (EC) No 1881/2006 as amended in 2015 and the Standard for cocoa powders (cocoas) and dry mixtures of cocoa and sugars (CXS 105 – 1981) CODEX Alimentarius.

Scheme provider

National Metrology Institute of South Africa
CSIR Scientia Campus
Pretoria
Meiring Naude Road
Brummeria
0183

Private Bag X34
Lynnwood Ridge
Pretoria
0040
South Africa

Scheme Co-ordinator

Dr Laura Quinn
Principal Scientist
Organic Analysis Section
Phone + 27 12 841 4431
E-mail: pt@nmisa.org

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 one 50 g portion of test sample. 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.

Statistical analysis

Participant performance will be evaluated against the assigned value for each parameter. The assigned value for the PTS will be obtained through ICP analysis at the NMISA Inorganic Analysis Laboratory

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.

Scheme details

Test samples

The PT scheme consists of one 50 g sample of raw milled and homogenised cocoa powder. The elements listed in Table 1, are those which are currently tested either for nutritional content labelling or as possible toxic elements in food. As the elements within the sample are naturally occurred, only a limited selection from those listed in Table 1 may be present at naturally occurred levels in the sample. The moisture content of the material should also be reported, while all elemental results should be dry mass corrected.

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: Nutritional and toxic elements in cocoa PT scheme list of analytes

Nutritional elements	Toxic elements
Calcium	Arsenic (Total)
Copper	Lead
Iron	Cadmium
Magnesium	
Manganese	
Phosphorus	
Potassium	
Sodium	
Zinc	

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. An electronic result submission form will be sent to participants when samples are delivered/collected. Participants are expected to submit two result for each element tested per sample and are encouraged to include an uncertainty estimate for each result obtained. Where applicable, participants must report whether a recovery correction was applied.

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:

- Sample size analysed
- Instrumentation specification

- Analytical method information
- Any corrections applied, e.g. recovery

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 2 Nutritional and toxic elements in cocoa powder PT scheme sample distribution dates

Matrix	Sample Distribution	Reporting	Samples
Cocoa powder	November 2020	December 2020	1 x 50 g

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)				