System research of soil and water quality sub-group,
Agricultural chemistry group,
Department of Agriculture

PROFICIENCY TEST FOR NUTRIENT LEVEL OF CHEMICAL FERTILIZER IN THAILAND

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AGENDA

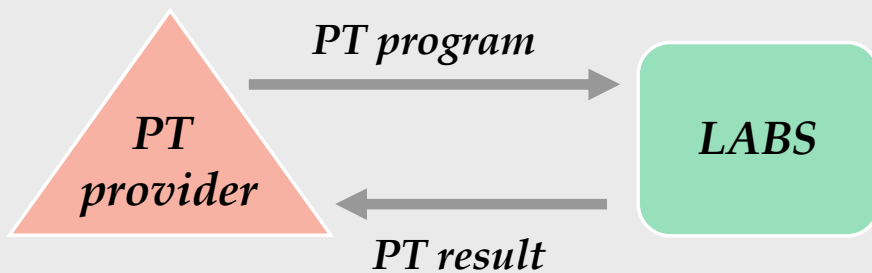1 Introduction

2 Experiment

3 Results and discussions

4 Conclusions

Introduction

Proficiency Testing

Proficiency testing is an external quality assessment to evaluate the laboratory analytical performance for assuring the valid and reliable of analytical results.

Proficiency Testing and ISO/IEC 17025 : 2017

Section 7.7 Ensuring the Validity of Results

7.7.2 The laboratory shall monitor its performance by comparison with results of other laboratories, where available and appropriate. This monitoring shall be planned and reviewed and shall include, but not be limited to, either or both of the following:

a) participation in proficiency testing;
b) participation in interlaboratory comparisons other than proficiency testing.

Introduction

Notification of the Department of Agriculture

ประกาศกรมวิชาการเกษตร

เรื่อง การกำหนดห้องปฏิบัติการวิเคราะห์ปุ๋ย ตามมาตรา ๓๖ (๑๑) และมาตรา ๓๖/๒ (๑๐)

แห่งพระราชบัญญัติปุ๋ย พ.ศ. ๒๕๑๘ ซึ่งแก้ไขเพิ่มเติมโดยพระราชบัญญัติปุ๋ย

(ฉบับที่ ๒) พ.ศ. ๒๕๕๐

พ.ศ. ๒๕๕๔

หมวด 3

ข้อ 6.3 ต้องจัดให้ห้องปฏิบัติการวิเคราะห์ปุ๋ยเข้าร่วมการทดสอบความชำนาญหรือเปรียบเทียบผลการวิเคราะห์ระหว่างห้องปฏิบัติการ หรือทดสอบตัวอย่างเทียบผล (Check sample) กับกรมวิชาการเกษตร อย่างน้อยปีละ 1 ครั้ง

Section 3

6.3 The fertilizer analysis laboratory shall participate in proficiency testing program, interlaboratory comparison, or check sample testing with department of agriculture at least once per year.

Introduction

Chemical Fertilizer Proficiency Testing Schemes

The chemical fertilizer proficiency testing schemes was initially organized in Thailand by the System research of soil and water quality sub-group, Agricultural chemistry group, Department of Agriculture following ISO/IEC 17043.

Experiment

Chemical Fertilizer Proficiency Testing Schemes

Sample:

- **4 Chemical fertilizer samples**

Parameters:

1) pH
2) Ammonium nitrogen
3) Total nitrogen
   Total phosphorus (as P_2O_5)
   Water soluble potassium (as K_2O)
4) Calcium oxide
   Magnesium oxide
   Total sulfur

Experiment

Proficiency Testing Sample Preparation

PT samples were distributed to 57 registered participants.

The test material from this scheme can be used as a reference material.

Chemical Fertilizer Proficiency Testing Schemes

PT samples were randomly selected to statistically evaluate the homogeneity and stability criterions.

Nutrient levels of the chemical fertilizer, as $x*$ and $s*$, target SD or predicted SD, were assigned in line with ISO 13528

Experiment

Sieve (particle size < 40 mesh or 420 µm)

Mix well

Proficiency Testing Sample Preparation

Contain samples in plastic bottle

Mill 5 kg of chemical fertilizers

2

3

1

4

8

Experiment

Proficiency Testing Sample Preparation

PT samples were distributed to 57 registered participants.

The test material from this scheme can be used as a reference material.

Chemical Fertilizer Proficiency Testing Schemes

PT samples were randomly selected to statistically evaluate the homogeneity and stability criterions.

Nutrient levels of the chemical fertilizer, as $x*$ and $s*$, target SD or predicted SD, were assigned in line with ISO 13528

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Experiment

Homogeneity check of PT sample

Randomly select

Analyze by the system research of fertilizer quality sub-group

Checking for between-sample standard deviation; s_s with the standard deviation for proficiency assessment; σ_{pt} (ISO 13528 : 2015)

$$s_s \leq 0.3\, \sigma_{pt}$$

Adequately homogeneous

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Experiment

Stability check of PT sample

Compare the general average of the measurement obtained in the check prior to distribution with the general average of the results obtained in the stability check (ISO 13528 : 2015)

$$|\overline{y_1} - \overline{y_2}| \leq 0.3\,\sigma_{pt}$$

Adequately homogeneous

Experiment

Chemical Fertilizer Proficiency Testing Schemes

Proficiency Testing Sample Preparation

PT samples were randomly selected to statistically evaluate the homogeneity and stability criterions.

PT samples were distributed to 57 registered participants.

Nutrient levels of the chemical fertilizer, as $x*$ and $s*$, target SD or predicted SD, were assigned in line with ISO 13528

The test material from this scheme can be used as a reference material.

Experiment

Chemical Fertilizer Proficiency Testing Schemes

Proficiency Testing Sample Preparation

PT samples were randomly selected to statistically evaluate the homogeneity and stability criterions.

PT samples were distributed to 57 registered participants.

Nutrient levels of the chemical fertilizer, as $x*$ and $s*$, target SD or predicted SD, were assigned in line with ISO 13528

The test material from this scheme can be used as a reference material.

Results and discussions

Parameters	x_{pt}	σ_{pt}
pH	Results reported by participants in this round	Reproducibility standard deviation from the reference method
Ammonium nitrogen	Reference laboratory	Results reported by participants in this round
Total nitrogen	Results reported by participants in this round	Results reported by participants in this round
Total phosphorus (as P_2O_5)	Results reported by participants in this round	Tolerances in the Notification of the Ministry of Agriculture Cooperatives
Water soluble potassium (as K_2O)	Results reported by participants in this round	Tolerances in the Notification of the Ministry of Agriculture Cooperatives
Magnesium oxide	Results reported by participants in this round	Experience with previous round and Expert judgment
Calcium oxide	Results reported by participants in this round	Predicted standard deviation
Total sulfur	Results reported by participants in this round	Predicted standard deviation

Results and discussions

Assigned value and standard deviation for proficiency assessment of fertilizer sample

Parameters	x_{pt}	σ_{pt}
pH	7.11	0.10
Ammonium nitrogen (%)	8.03	0.23
Total nitrogen (%)	15.02	0.26
Total phosphorus (as P_2O_5; %)	14.94	0.30
Water soluble potassium (as K_2O; %)	15.31	0.40
Calcium oxide (%)	4.31	0.14
Magnesium oxide (%)	2.03	0.20
Total sulfur (%)	8.94	0.27

Experiment

Calculation of performance statistics

z scores (ISO 13528 : 2015)

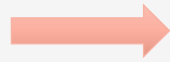$$z = \frac{(x_i - x_{pt})}{\sigma_{pt}}$$

x_i is the result from participant i,
x_{pt} is the assigned value, and
σ_{pt} is the standard deviation for proficiency assessment.
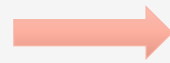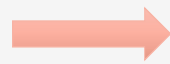
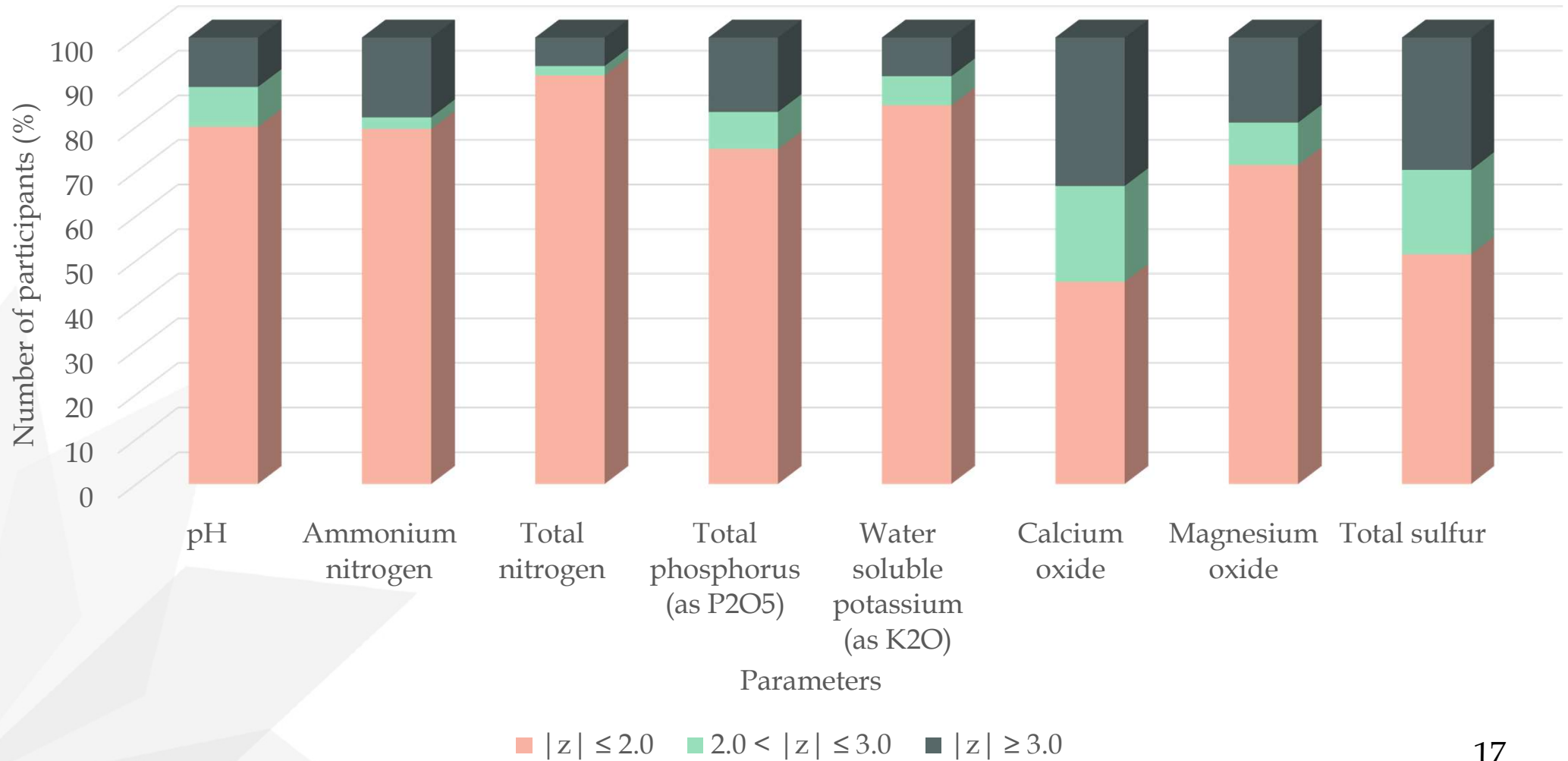$|z| \leq 2.0$ ⟶ Acceptable

$2.0 < |z| \leq 3.0$ ⟶ Warning

$|z| \geq 3.0$ ⟶ Unacceptable

Results and discussions

Conclusions

- Nutrient levels of the chemical fertilizer, as robust mean ($x*$) and robust standard deviation ($s*$), target SD or predicted SD, were assigned in line with ISO 13528
    - pH 7.1±0.10
    - Ammonium nitrogen 8.03±0.23%
    - Total nitrogen 15.02±0.26%
    - Total phosphorus (as P_2O_5) 14.94±0.30%
    - water soluble potassium (as K_2O) 15.31±0.40%
    - Calcium oxide 4.31±0.14%
    - Magnesium oxide 2.03±0.20%
    - Total sulfur 8.94±0.27%
- Participants achieving satisfactory performance ($z \leq 2$) ranged from 51.4 – 91.5%.
- Participants whose results were identified as questionable or unsatisfactory results were possibly caused by the unsuitable of analytical methods and/or instrument.
- Proficiency testing programs have allowed building up and strengthening the quality of chemical fertilizer laboratories in Thailand.

THANK YOU