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Development and Verification of Potassium Determination Method in Solid NPK Fertilizer by Atomic Absorption Spectrophotometry

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Abstract

The measurement of potassium levels in solid NPK fertilizer using the K_2O digestion method still requires repeatability to ensure the validity of the measurement results. This study aims to develop and validate a method for determining potassium levels using the P_2O_5 digestion method in accordance with SNI 2803:2012, employing a pragmatic approach based on SNI 7763:2018. Potassium analysis was performed using an Atomic Absorption Spectrophotometer (AAS) with several validation parameters. The potassium levels measured using both the K_2O and P_2O_5 digestion methods were 14%. The method demonstrated linearity, with a correlation coefficient (r^2) of 0.9986. The precision values for the K_2O and P_2O_5 digestion methods, based on Relative Standard Deviation (%RSD) < 2/3 CV Horwitz, were 1.6% < 1.8% and 1.1% < 1.8%, respectively, while t-calculated (0.646) < t-tabulated (2.179). Accuracy, as indicated by the percentage recovery for the K_2O and P_2O_5 digestion methods, was 108% and 101%, respectively. The P_2O_5 digestion method is categorized as an accurate, precise, and efficient technique for determining potassium levels in solid NPK fertilizer samples, particularly in large quantities.

Keywords: accuracy, digestion, P_2O_5, pragmatic approach, validation.

INTRODUCTION

In laboratory settings, method validation is crucial since each laboratory operates under different conditions, equipment capabilities, and personnel competencies. The application of methods in laboratories requires validation to ensure that the generated data is valid, with a level of accuracy and precision that can be justified (Miguel et al., 2021).

NPK fertilizer is a synthetic granular fertilizer containing various essential nutrients, including nitrogen (N), phosphorus (P), and potassium (K), in both macro and micro forms (Nadarajan & Sukumaran, 2021). The use of NPK fertilizer plays a significant role in improving crop productivity. Quality control of NPK fertilizers, particularly in terms of potassium content accuracy, is essential as it directly affects plant growth and yield.

Potassium in plants plays a role in regulating stomatal opening, protecting against oxidative stress, facilitating photosynthesis, enhancing nutrient uptake, and maintaining leaf inclination (Sardans & Peñuelas, 2021). In solid NPK fertilizers, the minimum potassium content threshold mandated by SNI 2803:2012 is 6%, while different industries may have varying quality requirements. Manufacturers of solid NPK fertilizers must conduct routine quality testing to ensure that the potassium content in their products meets established standards.

This study compares two digestion methods based on SNI 7763:2018, where potassium determination in solid organic fertilizers can be performed using a similar approach. Through a pragmatic approach, a new method was developed to analyze potassium in solid NPK fertilizers. This approach not only considers fundamental concepts derived from SNI 7763:2018 but also emphasizes practical implementation in testing laboratories, as it has been tested and successfully applied in various contexts (Foster & Godbole, 2022).

The standard method for determining potassium content in solid NPK fertilizer is the K_2O digestion method based on SNI 2803:2012. The K_2O digestion method requires multiple repetitions to obtain valid results. It uses similar reagents and processes to the P_2O_5 digestion method, with the primary difference being the volume of reagents used, namely 65% HNO_3. Both methods employ 10 mL of 70–72% $HClO_4$ with the addition of 65% HNO_3; however, the K_2O method uses 10 mL, while the P_2O_5 method uses 6 mL. The larger volume of $HClO_4$-HNO_3 in the P_2O_5 digestion method results in a more complete digestion process and more accurate results. This is because the $HClO_4$-HNO_3 mixture is a strong oxidizing agent (Hu et al., 2023).

The P_2O_5 digestion method is more effective for analyzing a large number of samples with valid results without the need for time-consuming repeat testing. Another advantage of this method is that it is simpler, easier to perform, efficient, and sensitive to samples, providing accurate results that meet the minimum potassium content threshold for solid NPK fertilizers established by the industry.

The P_2O_5 digestion method applied in this study is a modification of an existing standard method. Validation is necessary to ensure that the developed testing method meets the requirements set by ISO/IEC 17025:2017. According to Trishch et al. (2019), in compliance with ISO/IEC 17025:2017, laboratories are required to validate non-standard methods. This step is essential to obtain valid results with objective and reliable data that meet regulatory standards (Miguel et al., 2021). In this study, validation was conducted on the development of a potassium determination method for solid NPK fertilizers

using a pragmatic approach. The validation parameters included linearity, precision, and accuracy. An unpaired t-test, which is part of the precision parameter, was also performed to assess the differences between the K_2O and P_2O_5 digestion methods. If the obtained data meet the established standards, the method can be considered valid.

The primary objective of this study is to develop and validate a modified analytical method that is easy to implement and has a shorter analysis duration. By developing and validating this method, it is expected to increase consumer confidence, particularly in the effective analysis of potassium in solid NPK fertilizers. This study adapts SNI 7763:2018 using a pragmatic approach, thereby implementing the K_2O and P_2O_5 digestion methods from SNI 2803:2012 to determine potassium content in solid NPK fertilizers.

METHODS

Sample Collection

The solid NPK fertilizer samples were obtained from an industry utilizing potassium content testing services at the Testing Laboratory, Balai Besar Standardisasi dan Pelayanan Jasa Pencegahan Pencemaran Industri (BBSPJPPI). The potassium content in the sample, coded LS.464, must meet the quality requirements of the industry, which specify a maximum of ≤ 14%, as well as comply with SNI 2803:2012, which sets a minimum potassium content of ≥ 6%.

Sample Preparation

The NPK fertilizer samples were prepared by placing them into a 250 mL beaker glass and labeling them according to the sample code.

Validation of Potassium Testing Using Pragmatic Approach

In this study, a method development design was employed to establish and validate the analytical procedure. The workflow is illustrated in Figure 1, which outlines the development and validation process of a new method for potassium analysis. This process was designed to produce a reliable testing method and ensure accuracy in potassium measurement.

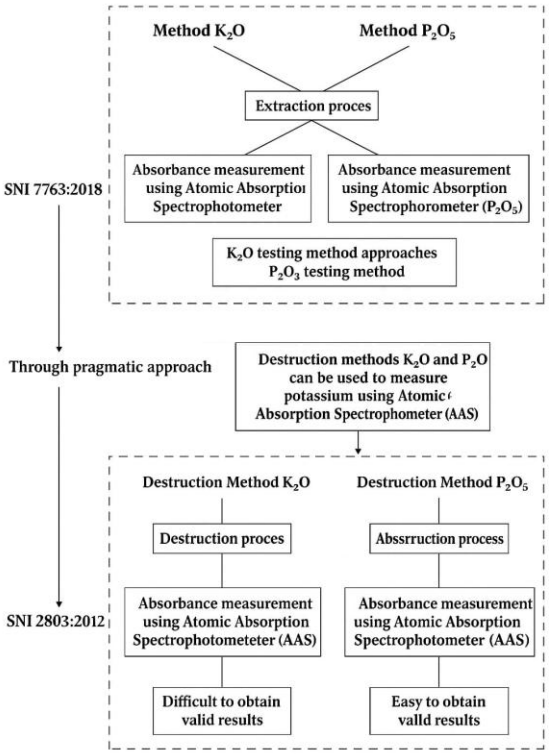

Figure 1. Validation Scheme for Potassium Determination in Solid NPK Fertilizer

The study design, as shown in Figure 1, refers to SNI 7763:2018, which enables potassium measurement in solid organic fertilizer using two digestion methods: the K_2O and P_2O_5 methods.. These two digestion methods serve as references for application to solid NPK fertilizer in accordance with SNI 2803:2012. In SNI 7763:2018, potassium analysis is conducted through an extraction process, whereas SNI 2803:2012 employs a digestion method. Additionally, in SNI 7763:2018,

different instruments are used for sample measurement between the K_2O and P_2O_5 methods. The complexity of developing a digestion method based on this framework makes it impractical to directly adapt the abstract principles of SNI 7763:2018. However, this discrepancy is not an issue, as a pragmatic approach was adopted. This approach focuses on practical applicability and ensures that the method can be effectively implemented in real laboratory settings without concern for theoretical abstraction.

K_2O Digestion Method

A 0.5 g sample of solid NPK fertilizer was weighed and placed into a 250 mL beaker glass. Subsequently, 10 mL of 70-72% $HClO_4$ and 6 mL of 65% HNO_3 were added. The mixture was gently heated to boiling for 5 minutes until it became colorless and emitted white fumes. The solution was then cooled and transferred into a 250 mL volumetric flask. The mixture was diluted to the mark with distilled water and shaken until homogeneous. The solution was filtered using ash-free filter paper No. 42 and transferred into a clean Erlenmeyer flask. Absorbance was measured at a wavelength (λ) of 766,9 nm using Atomic Absorption Spectrophotometry (AAS), and the potassium content in the sample was calculated.

P_2O_5 Digestion Method

A 0.5 g sample of solid NPK fertilizer was weighed and placed into a 250 mL beaker glass. Then, 20 mL of 65% HNO_3 and 10 mL of 70-72% $HClO_4$ were added. The mixture was slowly heated to boiling for 30-45 minutes until it became colorless and emitted white fumes. The solution was then cooled and transferred into a 250 mL volumetric flask. The mixture was diluted to the mark with distilled water and shaken until homogeneous. The solution was filtered using ash-free filter paper No. 40 and transferred into a clean Erlenmeyer flask. Absorbance was measured at a wavelength of 766,9 nm using Atomic Absorption Spectrophotometry (AAS), and the potassium content in the sample was calculated.

Method Validation Process

Linearity Test

Linearity occurs when the analyte concentration is directly proportional to the absorbance and the concentration of the unknown solution within a specific range (Attimarad et al., 2020). A calibration curve was constructed by measuring the absorbance of seven potassium standard solutions with concentrations of 0,0; 0,1; 0,2; 0,7; 0,8; 0,9, and 1,0 mg/L at a wavelength (λ) of 766,9 nm using Atomic Absorption Spectrophotometry (AAS). The calibration curve was used to illustrate the correlation between absorbance and concentration, resulting in a correlation coefficient (r^2) and a linear equation in the form of $y = ax + b$.

Precision Test

Precision refers to the accuracy of analysis, which is determined by repeatability and reproducibility values (Kowalska et al., 2022). Repeatability is the agreement of multiple measurements performed under nearly identical conditions (Schmeel et al., 2019). In this study, precision was measured through repeatability and reproducibility values, repeated seven times using the same analytical method, conducted on the same day (intraday) under identical laboratory conditions with a time interval of less than one hour. Repeatability is expressed in terms of Relative Standard Deviation (%RSD) (Alquadeib, 2019), where the accepted RSD value must meet the condition %RSD ≤ 2/3 Horwitz CV (Koesmawati et al., 2021). The %RSD value can

be calculated using Equation (1). where: SD is standard deviation, $\bar{x}$= mean potassium content (mg/kg).

$$RSD = \frac{SD \times 100\%}{\bar{X}} \quad (1)$$

Intra-laboratory reproducibility represents the accuracy of a method under varying operational conditions. It refers to the consistency of measurement results when validating a method (Raposo & Ibelli-Bianco, 2020). Consistency is assessed through reproducibility, as this method is performed by different analysts using different techniques.

Accuracy Test

Accuracy is assessed to determine the correctness of the method based on the average concentration obtained by comparing the measured value with the theoretical value. The results are expressed as the percentage of recovery (%recovery) (Rambo et al., 2019). Accuracy indicates the %recovery of an analyte when a spike is added to a measurement. Accuracy can be evaluated through spike recovery values (Balçık et al., 2020); if the obtained value meets the requirements, the digestion method used is considered accurate. The acceptable %recovery range is 85% to 115% (Spitteler et al., 2019).

Independent Sample t-Test (Independent Sample t-Test)

Hypothesis testing in the independent sample t-test is conducted by comparing the calculated t-value (t-calculated) with the reference t-value (t-table) (Mishra et al., 2019). If the t-calculated value exceeds the t-table value, it indicates that the mean of method 1 ($\bar{x_1}$) differs significantly from the mean of method 2 ($\bar{x_2}$). Conversely, if the t-calculated value is smaller than the t-table value, there is no significant difference between the means of method 1 ($\bar{x_1}$) and method 2 ($\bar{x_2}$).

RESULTS AND DISCUSSION

Analysis of Potassium Metal in Solid NPK Fertilizer

Potassium is one of the essential nutrients that play a crucial role in plant growth by activating various enzymes, regulating stomata to facilitate efficient photosynthesis, enhancing carbohydrate formation, and improving plant resistance to diseases (Ullah et al., 2022).

In the initial test using the K_2O destruction method, the measured potassium content in the solid NPK fertilizer sample was below 14% (Table 1). However, after reanalysis, the measurement results indicated that the potassium concentration in the sample reached 14% (Table 2). This concentration met the minimum threshold set by SNI 2803:2012 as well as the requirements of industrial users of testing services, although no maximum limit was specified. Furthermore, based on Table 2, the analysis results after reanalysis were compared with the P_2O_5 destruction method and showed equivalent potassium levels. This finding indicates that both destruction methods have comparable validity, despite some differences in the testing procedures.

Sample Destruction According to SNI 2803:2012 Through a Pragmatic Approach

A pragmatic approach was adopted to develop the P_2O_5 destruction method into a novel method for potassium testing in solid NPK fertilizers after undergoing validation. This approach employs a comprehensive framework, despite differences between the abstract principles of SNI 7763:2018 and the P_2O_5 destruction method of SNI 2803:2012, which was developed in this study.

Table 1. Potassium Content (K_2O) Measurement Results Before Replication

Parameter (Destruction Method)	Repetition	Absorbance	Potassium Content (mg/kg)	Average Content (mg/kg)	Potassium Content (%)
K_2O (Analyst-1)	1	0.2300	114885.1149	115704.9	13.94
	2	0.2305	115111.8658		
	3	0.2310	115315.4952		
	4	0.2322	116007.1942		
	5	0.2305	115134.8651		
	6	0.2331	116480.1119		
	7	0.2340	117000.0000		

Table 2. Measurement Results of Potassium Metal Content in Solid NPK Fertilizer Samples

Parameter (Destruction Method)	Repetition	Absorbance	Potassium Content (mg/kg)	Average Content (mg/kg)	Potassium Content (%)
K_2O (Analyst-1)	1	0.2344	117083	117404	14
	2	0.2305	115112		
	3	0.2351	117362		
	4	0.2380	118905		
	5	0.2300	114885		
	6	0.2395	119678		
	7	0.2376	118800		
P_2O_5 (Analyst-2)	1	0.2339	116786	116855	14
	2	0.2367	118161		
	3	0.2375	118560		
	4	0.2346	117136		
	5	0.2310	115315		
	6	0.2336	116613		
	7	0.2312	115415		

The design of the destruction method development in this study is complex and cannot fully adapt the abstract principles of SNI 7763:2018. In this research, the method outlined in SNI 7763:2018 serves only as a reference, indicating that potassium content analysis in organic fertilizers can be conducted through a similar procedural sequence. The potassium content can be determined using two destruction methods: K_2O and P_2O_5. Validation is essential to ensure that the developed testing method complies with the standards established in ISO/IEC 17025:2005.

The findings of this study are supported by research conducted by Kelly and Cordeiro (2020), which stated that a methodology based on a pragmatic approach serves as a framework for investigating how its application strengthens each research phase.

This approach provides guidance within a theoretical knowledge framework that focuses on research stages and practical implementation, ensuring that the study is feasible and actionable. These stages range

from project design and data collection to data analysis, conclusion formulation, and even research publication. The resulting research will be contextually relevant based on theoretical foundations.

Comparison of K_2O and P_2O_5 Destruction Methods

In this study, both the K_2O and P_2O_5 destruction methods fall under the category of wet destruction. This process involves the use of HNO_3 and $HClO_4$ as strong acid reagents to oxidize the sample, producing organic compounds that can be measured. The greater the amount of HNO_3 and $HClO_4$ used, the longer the degradation time required for sample decomposition. The P_2O_5 destruction method requires a higher amount of HNO_3 and $HClO_4$ compared to the K_2O destruction method.

A prolonged sample degradation duration enables complete decomposition of the solid NPK fertilizer sample. In contrast, the K_2O destruction method has a shorter degradation time but requires multiple repetitions, as the obtained potassium content falls below the minimum threshold set by the industry.

Validation of the Method

The validation parameters for the potassium analysis method in this study include linearity, precision, and accuracy. Additionally, an independent t-test was conducted as part of the precision parameter to assess the extent of differences between the K_2O and P_2O_5 destruction methods.

Linearity

Using the standard potassium calibration curve shown in Figure 2, the resulting equation is formulated as y = 0.4103x + 0.0093, with a correlation coefficient (r^2) of 0.9986. This correlation coefficient meets the acceptance criteria for potassium content validation, which requires $r^2 \geq 0.99$. This finding is supported by Yang et al. (2019), who stated that good linearity is indicated by a correlation coefficient (r^2) of ≥ 0.99. This confirms that the Atomic Absorption Spectroscopy (AAS) instrument is in optimal condition for measuring analyte concentrations in solid NPK fertilizer samples.

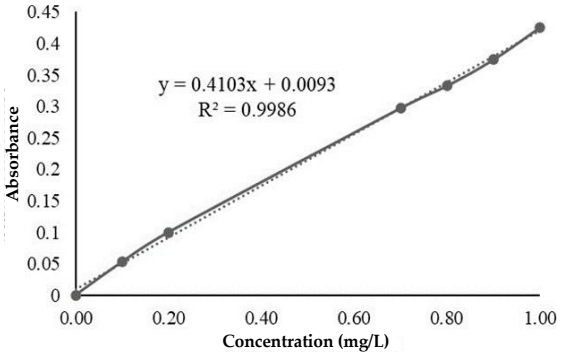

Figure 2. Standard Potassium Calibration Curve

Precision

The precision test results for solid NPK fertilizer samples, expressed as repeatability (%RSD), are presented in Table 3. A %RSD ≤ 2/3 Horwitz CV indicates that both destruction methods meet the testing requirements (Koesmawati et al., 2021), as evidenced by 1.6% < 1.8% (K_2O destruction method by Analyst-1) and 1.1% < 1.8% (P_2O_5 destruction method by Analyst-2). Comparing the %RSD values with 2/3 Horwitz CV helps determine whether the destruction method is acceptable. The repeatability measurements show that the %RSD values for both K_2O and P_2O_5 destruction methods are lower than 2/3 Horwitz CV (Table 3). These results confirm that both methods have good repeatability and meet the acceptance criteria, making them applicable for potassium testing in laboratories.

Table 3. Precision with Repeatability (%RSD) for Solid NPK Fertilizer Samples

Parameter (Destruction Method)	Relative Standard Deviation (%RSD)
K_2O (Analyst-1)	1,6
P_2O_5 (Analyst-2)	1,1
Acceptance limit (%)	≤ 1,8

The reproducibility test for the K_2O and P_2O_5 destruction methods was conducted using an independent sample t-test. According to Kang (2021), a t-test is required to compare the mean values of two independent groups. In this study, the t-test analysis was performed to examine whether there was a significant difference between the means of the two destruction methods.

The precision test results for solid NPK fertilizer samples, analyzed using the t-test (Table 4), show that the calculated t-value (0.646) is smaller than the t-table value (2.179). This indicates that there is no significant difference between the K_2O destruction method (Analyst-1) and the P_2O_5 destruction method (Analyst-2). The reproducibility test for both destruction methods is considered successful, as the parameter values meet the required criteria (Wu et al., 2023).

Table 4. Precision with Reproducibility Test Using the t-Test for Solid NPK Fertilizer Samples

Replication	Destruction Method	
	K_2O (Analyst-1)	P_2O_5 (Analyst-2)
1	14,110	14,074
2	13,872	14,240
3	14,144	14,288
4	14,330	14,116
5	13,845	13,897
6	14,423	14,053
7	14,317	13,909
Mean	14,149	14,083
SD	0,226	0,149
n	7	7
sp	0,191	
t- calculated	0,646	
t-table	2,179	

Accuracy

The accuracy test of the K_2O and P_2O_5 destruction methods in this study aims to ensure that both methods exhibit a high level of precision. The %recovery values obtained for the K_2O and P_2O_5 destruction methods are 108% and 101%, respectively as shown in Table 5, . The %recovery values obtained using AAS are excellent for both methods, as they fall within the acceptable validation criteria of 85–115% (Spitteler et al., 2019). The potassium content analysis using both methods produced %recovery values close to 100%, indicating a very high level of accuracy (Nowak et al., 2021). Therefore, both methods are deemed valid.

A pragmatic approach to potassium determination has proven to be essential in measuring potassium content in solid NPK fertilizer samples. Based on the validation of the K_2O and P_2O_5 destruction methods, large sample quantities do not require extensive retesting, allowing for time-efficient testing while maintaining valid and acceptable results. This study concludes that the P_2O_5 destruction method is equivalent to the standard method (K_2O destruction method) and can be used as a routine analysis method for measuring potassium levels in solid NPK fertilizers in testing laboratories. The P_2O_5 destruction method can be applied by laboratory analysts who require a faster and accurate analysis process for solid NPK fertilizer.

Table 5. %Recovery of Potassium Measurement Methods for Solid NPK Fertilizer Samples

Parameter (Destruction Method)	*%recovery*

K_2O (Analyst-1)	108
P_2O_5 (Analyst-2)	101
Acceptance limit (%)	85 ± 115

CONCLUSION

In this study, potassium testing was conducted, resulting in a potassium content of 14% in the solid NPK fertilizer sample. This concentration complies with the requirements of SNI 2803:2012 and the quality standards of the industrial service users. The determination of potassium in the solid NPK fertilizer sample using the K_2O and P_2O_5 destruction methods with Atomic Absorption Spectrophotometry (AAS) met validation requirements. This was demonstrated by several measurement parameters, including linearity (r^2) of 0.9986, which meets the acceptance criterion of ≥ 0.99, accuracy within the acceptable range of 85 ± 115%, and a relative standard deviation (RSD) lower than 2/3 of the Horwitz value. No significant difference was observed between the K_2O and P_2O_5 destruction methods, as evidenced by the t-calculated value being smaller than the t-table value. In this study, the P_2O_5 destruction method, developed through a pragmatic approach, was determined to be a suitable, accurate, and precise testing method. It can be effectively applied for the analysis of potassium content in large quantities of solid NPK fertilizer samples.

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