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Verification of the Test Method for Determination of Boron in NPK Blending Fertilizer Using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

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Abstract. Verification of the test method for determining boron (B) in NPK blending fertilizer using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) at PT. Pupuk Kalimantan Timur has been conducted. Determination of B in NPK fertilizer refers to the Testing Methods for Fertilizers Incorporated Administrative Agency Food and Agricultural Materials Inspection Center (FAMIC 2016). The optimum parameters of the analytical performance method were performed. The results of the research are good, showed with the linearity value (R^2) were 0.9997, the limit of detection (LOD) was 0.83 mg/L and the limit of quantification (LOQ) was 0.88 mg/L. The precision is shown as percentage of coefficient of variance (% CV) < 2/3 CV Horwitz values and the accuracy of this method is shown by a recovery percentage was 97-105%. Based on the results of research it can be concluded that the verification of the test method (FAMIC 2016) has been verified and can be used for routine analysis at the Quality Testing Laboratory of PT. Pupuk Kalimantan Timur.

INTRODUCTION

Fertilizer is one of the materials added to address the nutrients that plants need to produce properly. Fertilizers frequently applied to plants of organic fertilizer and inorganic fertilizer. Organic fertilizer is typically a product of both plants and animals. Inorganic fertilizer is a fertilizer produced by a manufacturer using modern tools [1]-[2]. Artificial fertilizers are fertilizers made to meet plant nutrients. Artificial fertilizers that are often used are nitrogen, potassium, sulfur and phosphate fertilizers or mixed fertilizers such as NPK fertilizer [3]-[4].Boron (B) can be found in NPK fertilizer and is a micronutrient such as S, Ca, Mg, B, Mo, Cu, Fe, and other organic materials, which are added to the process of making NPK fertilizer [5]-[6]. Boron in NPK fertilizer comes from boric acid (H_3BO_3), because during the urea fertilizer production process, boric acid was used as a distillate, which functions to capture NH_3 gas which is alkaline. Boron is found in NPK fertilizers, even though in small amounts [7]-[8].

Method verification is usually done to prove that the laboratory is capable of carrying out the method according to the procedure used because each instrument has different capabilities. Verification is a process in order to obtain important information in assessing the strengths and weaknesses of the method being developed. Valid or not the results of the method analysis test carried out usually by verifying the method first before developing it in the method validation test [9]-[10]. Where the parameters conducted are usually more complete, when compared to verification methods, including linearity, accuracy, precision, detection and quantitation limits [11]-[12].

Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) is a measuring instrument capable of identifying and measuring all elements simultaneously and different metals. ICP-OES is very suitable for measuring various concentrations of elements from ultra-trace to main components. This is because the detection limit is generally low in the mg/L range [13].

Several verification methods for determining an analyte using ICP-OES tools such as determination of lead (Pb) in ambient air samples [11], analysis of Pb, Cd, Cr, Cu, Ni, Co, Fe, Mn and Ba metals in water [14], validation

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method verification of test methods of Fe ion activated Methyl Diethanol Amine (aMDEA) [15], measurement of Mn in lubrication oil [16], and verification of Pb in NPK fertilizers using AAS [3], all of which demonstrate the effectiveness and accuracy of measurements.

Based on the description above, verification of the test method for determining Boron (B) in NPK blending fertilizer using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) at PT. Pupuk Kalimantan Timur, with measurement the Boron (B) content in NPK blending fertilizer, with determining several verification parameters such as linearity, precision, accuracy, limit of detection (LOD), and limit of quantitation (LOQ).

EXPERIMENTAL

Instruments and Materials

The instruments used in this research was the ICP-OES variant 270 ES, digital analytical balance, volume pipette, volumetric flask, glass beaker, burette, spatula, stirring rod, funnel, spray bottle, blender, bulb, magnetic stirer, and hotplate.The materials used in this study were 12-12-17-2 NPK fertilizer samples, 1:5 HCl solution, 1% HNO_3, standard liquor ICP multielement V 2.0 mg/L, chromate solution, Whatman 40 paper filter, and aquabidest.

Preparation of Standard Solution ICP Multielement V 0.05 - 1 ppm

The equipment to be used was washed and soaked first using a chromate solution, before making the ICP standard solution and rinsed using aquabidest. The standard solution used was the standard liquor ICP multielement V 2.0 mg/L. ICP standard solution was inserted into the burette, and put into a 250 mL volumetric flask with volume variations of 5; 12.5; 25; 62.5; 125 mL, diluted using 1% HNO_3 solution. The concentration of the solution was tested using ICP-OES variant 270 ES.

Preparation of Sample Boron in NPK Fertilizer

In this study, sample testing refers to the Testing Methods for Fertilizers Incorporated Administrative Agency regarding the method of testing fertilizer samples using ICP-OES. The NPK 12-12-17-2 fertilizer sample was mashed first using a blender. The refined samples were then sieved using mesh sieves no. 40 and put into the plastic sample and labeled, then weighed ± 0.15 g and put into glass beaker and added with ± 50 mL of aquabidest. Homogenized with a stirrer (± 1 hour), and put into a 100 mL volumetric flask. Diluted with demineralized water and filtered using Whatman 40 paper. Then pipette ± 2 mL into a 100 mL volumetric flask and added 25 mL of 1:5 HCl solutions, diluted with aquabidest. The solution was tested using ICP-OES variant 270 ES.

Linearity

Linearity determination was carried out using a blank solution and standard series solution of ICP multielement V 0.05-1 ppm and measured using ICP-OES variant 270 ES. Measurements are made by comparing the sample concentration with the absorbance of the measured sample. From the data obtained, a standard curve is made, and the value of the slope (a), intercept (b), and the coefficient of correlation (r) was obtained.

Precision

The precision determination was carried out using a sample solution that had been prepared and added with 1:5 HCl solutions, measured using the ICP-OES 270 ES variant. Measurements were made 10 times the sample repetition. From the data obtained, the average value, standard deviation (SD), and relative standard deviation (RSD) were calculated and compared with the acceptance requirements are 2/3 CV Horwitz.

Accuracy

Determination of accuracy was carried out using a sample solution added with 5 mL standard solution of ICP multielement V 0.1 ppm and 1:5 HCl solutions. Repeat measurements were performed 10 times using ICP-OES

variant 270 ES. From the data obtained, the recovery value can be calculated by comparing the spiking concentration after dilution to the standard solution concentration, and it is obtained as a percentage recovery value.

Limit Detection and Limit Quantitation

Determination of the limit of detection (LOD) and limit of quantitation (LOQ) was carried out using a sample blank that did not contain boron and added with 1:5 HCl solution and measured 10 repetitions using ICP-OES variant 270 ES. From the data obtained, it can be calculated the value of the Limit of Detection (LOD) from the standard deviation of the blanks with the lowest concentration. The LOD is usually expressed as 3 Sa/b and the LOQ as 10Sa/b, where Sa is the standard deviation and b is the slope.

RESULTS AND DISCUSSION

Linearity

Determination of linearity was used to determine the proportional relationship between the concentration of standard analytes and the response of the detector system. Linearity is the measurement variable results from the standard concentration with measured absorbance and is reported in the correlation coefficient (r) obtained from the results of statistical calculations. The correlation coefficient between the analyte concentration and the detector response of each metal is presented in the form of a measurement calibration curve [17]. The calibration curve of the linearity measurement results for the determination of boron (B) levels in NPK fertilizer using ICP-OES can be seen in Figure 1.

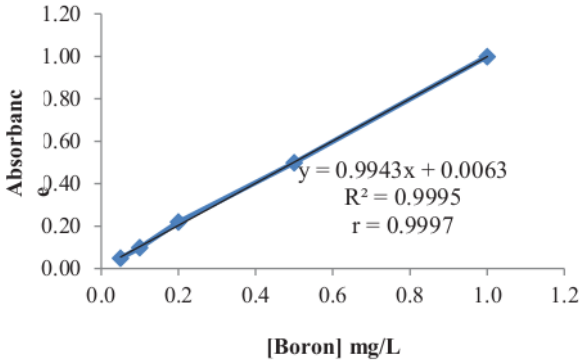

FIGURE 1. Calibration Curve of Boron

The results of linearity measurements in the analysis method for determination of boron (B) levels in NPK fertilizer show the curve between the standard boron concentration and the measured absorbance is directly proportional to the concentration. The slope value of 0.9943 indicates that for each increase in one concentration, the absorbance will increase by 0.9943 times. A positive slope value indicates a positive relationship between concentration and absorbance, meaning that the higher the X value, the higher the y value, and testing give a high sensitivity value [9],[18]. The intercept value shows the sensitivity of the detector system used when the absorbance amount was measured when the standard concentration is zero (0). The measured intercept value is 0.0063. The intercept value also shows the amount of tool noise. According to Riyanto [9], the ideal intercept is zero (0). The smaller the intercept value obtained, the better the measurement, indicating the smaller of noise measurement.

The correlation coefficient (r) of the determination of boron content in NPK fertilizer is 0.9997. The value of r = 0.09997 states that the linearity result is valid and there is a good relationship between the measured concentration and the resulting absorbance. The correlation between the increase in concentration and the increase in absorbance can be said to be strong because the value is close to 1. The coefficient of determination (R^2) on the determination of boron content in NPK fertilizer is 0.9995. This value shows that the concentration has an affects on 99.95% of the

absorbance. Meanwhile, the value of 0.05% indicates the error or deviation of the regression line that can be caused by the noise signal read by the instrument.

Precision

Precision is the closeness of measuring the results of measurements made repeatedly by the same analyst, under the same conditions, and over the same time frame [9]. Determination of the precision value in determining ~~of~~ boron in NPK fertilizer was carried out by measuring the sample 10 times, and the measurement results can be seen in Table 1.

TABLE 1. Measurement data of the precision determination of Boron in NPK fertilizer by ICP-OES

No.	Weight of sample (mg)	Measured concentration in the instrument (mg/L)	The concentration of B (%)	Concentration of B_2O_3 (%)
1	150.1	0.1881	0.36	1.14
2	150.1	0.1831	0.34	1.08
3	150.5	0.1822	0.34	1.07
4	150.7	0.1852	0.34	1.10
5	150.8	0.1838	0.35	1.09
6	150.3	0.1832	0.34	1.08
7	150.0	0.1840	0.34	1.09
8	150.1	0.1837	0.34	1.09
9	150.8	0.1837	0.34	1.09
10	150.5	0.1832	0.34	1.08
	Average	0.1840	0.34	1.09
		SD		**0.02**
		%RSD		**1.83**
		2/3 CV *Horwitz*		**2.63**

Based on the precision measurement results on the determination of boron levels, the% RSD value for boron levels was 1.83. The results obtained were compared with 2/3 CV Horwitz as a condition of acceptance, showing that the% RSD value was smaller than the 2/3 CV Horwitz value (1.83 < 2.63). The results of this precision test show that the measurement of Boron levels meets the acceptance requirements. The very high accuracy shows that the instrument operating system is stable, the reagents are good, the laboratory conditions are controlled, and the analyst is competent. This is evidenced by the relatively constant response obtained, and the random error that occurs is relatively small [18].

Accuracy

Accuracy was determined to show the closeness of the analysis results to the actual analyte content. Accuracy is expressed as the percentage recovery (% recovery) value of the added analyte [17]. Accuracy measurements were carried out by the spiking method (addition of the ICP multielement V standard) at the standard concentration added to the NPK fertilizer sample of 0.1 mg/L. The calculation of % recovery for the determination of boron levels in NPK fertilizer can be seen in Table 2.

TABLE 2. Measurement of % recovery on the determination of Boron in NPK fertilizer using ICP-OES

No.	Content of B_2O_3 in NPK (%)	Content of B_2O_3 standard (%)	Content of B_2O_3 measured (%)	*% Recovery*
1	1.14	0.95	2.06	98.56
2	1.08	0.95	2.05	100.98
3	1.07	0.95	2.07	102.48
4	1.10	0.95	2.03	99.02
5	1.09	0.95	2.04	100.02
6	1.08	0.95	2.04	100.49
7	1.09	0.95	2.05	100.98
8	1.09	0.95	2.08	100.04
9	1.09	0.95	2.06	100.98
10	1.08	0.95	2.05	100.98
		Average		**100.45**
		Range of % Recovery		**(98-102)**

The accuracy of a method was declared good if the% recovery value is close to 100% [9]. Based on the measurement results, the% recovery value accuracy is obtained in the range (98-102 %). These results inform that the determination of boron levels in NPK fertilizers by ICP-OES has good accuracy. The measurement results are within the conformity level of a measurement range proportional to the true value so that the results can be accepted.

Limit of Detection (LOD)

Limit of Detection (LOD) is the smallest number of samples that still provide a measurable response compared to blank response measurements and can still be detected. LOD is a parameter used to indicate the lowest Boron concentration detected by the instrument using the test method being performed [12]. The LOD value obtained in this measurement was 0.0807 mg/L. This value indicates that the measurement of Boron using ICP-OES can be detected up to a limit of 0.0807 mg/L, so when measuring boron in a sample it is recommended to use a concentration above 0.0807 mg/L.

Limit Quantitation (LOQ)

Limit of Quantitation (LOQ) measurement is the reporting value as the smallest concentration of analyte that can be determined by an agreed measurement method in a test laboratory. LOQ is the lowest concentration of Boron which provides a response precision and accuracy that is acceptable in the experimental method specified. The LOQ value obtained from the measurement results was 0.0882 mg/L. This value indicates that the measurement of Boron using ICP-OES can read the quantitation limit reported is above 0.0882 mg/L.

CONCLUSION

Based on the results of the research, it can be concluded that the verification of the test method for determining Boron in NPK fertilizers using ICP-OES with various test parameters has been verified properly. The data on measurement parameters obtained can meet the acceptance requirements, as stipulated in the FAMIC 2016, indicating that the ICP-OES can be used for routine analysis at the Quality Test Laboratory of PT. Pupuk Kalimantan Timur.

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REFERENCES

1. I. M. Adnyana, Ecotrophic **7**, 152–153 (2008).
2. H. D. Julita, Syamsuddin and R. Hayati, J. Floratek. **11**, 10–17 (2016).
3. Friscylia, S. Koesnarpadi, and A. S. Panggabean, Prosiding Seminar Nasional Kimia-UNMUL, 35-39 (2019).
4. R. Rosliani, E. R. Palupi,and Y. Hilman, J. hort. **22**, 242-250 (2012).
5. L. H. Simanjuntak, E. Erwin, and A. S. Panggabean, ALCHEMY Jurnal Penelitian Kimia **16**, 1-11 (2020).
6. H. Sugianto, L. Darsana, and Pardono, J. Penelitian Agronomi. **16** , 29-36 (2014).
7. C. Dordas, G. E. Apostolides, and O. Goundra, J. Agri Sci. **145**, 377–384 (2007).
8. S. E. Seadh, M. I. EL-Abady, A. M.El-Ghamry, and S. Farouk, J. Biological Sci. **9**, 851-858 (2009).
9. Riyanto, *Validasi & Verifikasi Metode Uji Sesuai dengan ISO/IEC 17025 Laboratorium Pengujian dan Kalibrasi*, (Deepublish, Yogyakarta, 2014).
10. M. Taufiq, A. Sabarudin, and A. Mulyasuryani, ALCHEMY Jurnal Penelitian Kimia **5**, 31-37 (2016).
11. Z. Afifah, Kurniyawan, and T. Huda, Indo. J. of Chem. Anal. **2**, 74–79 (2019).
12. A. S. Panggabean, S. P.Pasaribu, and F. Kristiana, Indo. J. Chem. **18**, 279–285 (2018).

13. C. B. Boss, and K. J. Fredeen, *Concepts, Instrumentation, and techniques in ICP-OES*, (In PerkinElmer, USA, 2004).
14. P. Pirdaus, M. Rahman, Rinawati, N. L. G. R. Juliasih, D. Pratama, and A. A. Kiswandono, Analit: Analytical and Environmental Chemistry, **3**, 1–10 (2018).
15. A. S. Panggabean, and A. Rachman, Indo J. of Chem. Res. **3**, 302-307 (2016).
16. R. M. Napitupulu, D. Julia, and A. S. Panggabean, Indo. J. Chem. Res. **6**, 94-100 (2019).
17. A. S. Panggabean, S. P. Pasaribu, Bohari and Nurhasanah, Indo. J. Chem. **14**, 51–56 (2014).
18. Harmita, Majalah Ilmu Kefarmasian **1**, 117–135 (2004).

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