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Original article

Method development and validation for the determination of potassium (K_2O) in fertilizer samples by flame photometry technique

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ABSTRACT

Background: Fertilizer samples are tested for determining their nutrient contents; however, different methods give varying results. Therefore, the major objective of this study was to develop and validate potassium determination method by flame photometry technique.
Methods: Flame photometry technique for the quantification of potassium concentration in fertilizer samples was validated in Soil and Water Testing Laboratory (ISO/IEC: 17025), Dera Ghazi Khan. The method validation was done for repeatability, reproducibility, limit of detection, limit of quantification, linearity, recovery, selectivity, and bias.
Results: The limits of detection and quantification were 0.87% and 2.88% K_2O, respectively. The repeatability was 0.33%, and reproducibility (T-calculated was 0.69 which was less than T-tabulated, i.e., 2.06). Linear curve was obtained for concentrations ranging from 5 to 25 ppm (K) exhibiting R^2 of 0.99. The recovery for K in fertilizer sample was 98.8%. The potassium contents were identified with complete recovery without interference of other nutrients in the sample. The Z-score of all the results (Magruder USA, Fertilizer sample check program) were in acceptable range. The correlation coefficient (0.999%) depicts strong relationship between actual K value and observed values.
Conclusions: By this relation, we can say that method performance was excellent. Hence, the method can be successfully used for potassium determination in fertilizer samples.

1. Introduction

The analytical methods play important role in various fields, i.e., food products, environmental analysis, pharmaceutical and biomedical analysis etc. To reach the most consistent, accurate, as well as repeatable data, an easiest analysis method is required (Ahmad et al., 2015; Gumustas et al., 2013; Kurbanoglu et al., 2014). Validation is a vital factor in monitoring the reliability of

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the method that is determined by the validation of the obtained results. The specificity, accuracy, precision, detection limit (LOD), quantification limit (LOQ), sensitivity and repeatability are the criteria for determining the validity of a method. The validated method is crucial for acheiving high quality (Aboul-Enein, 2012; Arkaban et al., 2021; Striegel, 2021).

Potassium is one of the major nutrient elements for plant growth and helps in the activation of several soil enzymes. During the infections of various airborne pathogen, the stomata can perform appropriately if enough potassium is available to plants. Thus, sufficient K availability prevents pathogen attack by quick stomatal closure (Farooq et al., 2017; Härdter and Fairhurst, 2003; Härter et al., 2004; Onen et al., 2017; Özaslan et al., 2016). Potassium regulates the opening and closing of stomata during photosynthesis, consequently regulating the uptake of CO_2. It helps in the synthesis of carbohydrates in plants. It imparts immunity in plants against various diseases, strengthens the stem and prevents plants from lodging. It improves the quality of fruit so act as a quality element (Farooq et al., 2018; Härdter and Fairhurst, 2003; Surucu et al., 2020).

As per ISO (2005) standard, the validation of any analytical method aims to assure that it fulfils the suitable criteria. The objective of this study was to validate flame photometry method for the analysis/determination of potassium in fertilizer samples.

2. Materials and methods

The potassium containing potassium chloride (analytical grade) sample was used (Lot: K47133836 603), and potassium standard solutions were supplied by PanReac Appli Chem (Lot # Lot: 0001757911) (Engelbrecht and McCoy, 1956). The chemicals used in the study were of analytical grade. The calibrated glassware were used during the analysis (Johnson et al., 1987). The 2.5 g ground potassic fertilizer materiel was dissolved in 1000 ml distilled water. The solution was then filtered and dilutions were made accordingly. The reading of the filtrate was taken on flame photometer using standard curve procedure (Knudsen et al., 1983; Wiyantoko et al., 2021).

2.1. Standard solutions' preparation

The stock solution (1000 ppm K) was used for working standards preparation of solutions by employing the formula of Bano et al. (2021).

$C_1V_1 = C_2V_2$where, C_1 = Stock solution concentration (ppm), V_1- = volume to be taken from the stock (ml), C_2 = K concentration required (ppm) and V_2 = total volume required (ml).

2.2. Determination of potassium (K)

The instrument flame photometer PFP7 (Jenway) was used with flow rate of 2–6 ml/minute. Continuous supply of air between 14 and 30 psi (approximately 1–2 kg/cm^2) at 6 L/minute was maintained.

2.3. Method validation

Method validation was accomplished through the assessment of numerous analytical figures of merit as per International Conference on Harmonization, including repeatability, reproducibility, method working range, precision, the detection limit (LOD) and quantification limit (LOQ), recovery, selectivity, and bias (Guideline, 2007; Sahoo et al., 2018). This method validation study was carried out at Soil and Water Testing Laboratory for Research, Dera Ghazi Khan, Pakistan. The 1000 ppm standard of K Lot No. Lot: 0,001,757,911 Pan Reac Appli Chem was used for the preparation of working standards. The PFP7 Jenvay, flame photometer was used for standards and for potassium in fertilizer sample analysis. The flow rate of sample was maintained between 2 and 6 ml per minute.

2.4. Accuracy

Accuracy is defined as "nearness of the results to the true value". For determination of method accuracy, the data of repeatability of two different analysts was applied. As per CIPAC (1999) a best validated method is one which has accuracy > 85%. The accuracy was determined by the formula given by Desta and Amare (2017) and Sinshaw et al. (2019).

Accuracy = 100 – error.

$$\text{Error}\,(\%) = \frac{\text{Observed value}\,(x) - \textit{True value}}{\textit{True value}} \times 100$$

2.5. Precision

Precision is defined as "agreement among set of replicate measurements without knowledge of true value". For the calculation of precision, the results of repeatability and reproducibility were used. For repeatability analyst-1 10 samples were prepared having same concentration of potassium and its contents were measured. However, in reproducibility analyst-2 the samples of same concentration of potassium were prepared and run on PFP 7 Flame-photometer taking 10 repeats (Barnawal et al., 2016).

2.6. Linearity and range

For linearity calculation, potassium standards, i.e., 5, 10, 15, 20 and 25 ppm were prepared from 100 ppm sub stock solution and run on flame photometer (Addo et al., 2019; Narsimha and Sudarshan, 2018).

2.7. Limit of quantification

The limit of detection (LOD) is the lowest concentration of any substance which can be detected and obviously differentiated from zero; however, not necessarily quantified (González et al., 2018; McDowall, 2005). One the other hand limit of quantification (LOQ) is the lowest concentration of any substance that can be measured quantitatively with an acceptable level in terms of precision as well as accuracy (González et al., 2018; González and Herrador, 2007; Markley et al., 1998).

2.8. Measurement of uncertainty

For the calculation of uncertainty, the Eurachem Guide was used. The uncertainty in the results might be due of many factors, e.g., personal, method of analysis, environment and chemicals used, and equipment. Combined uncertainty is the combination of all these factors. The budget of uncertainty includes all the uncertainties due to above mentioned factors (Cortez, 1995; Örnemark, 2004). This uncertainty is calculated at 68% level of confidence, as for as ISO/IEC 17,025 is concerned the testing laboratories must represent their uncertainties with defined confidence level which is called as expanded uncertainty (Aslam et al., 2021; Nazir et al., 2020; van der Veen and Cox, 2021).

$$\text{Combined uncertainty} = \sqrt{(U_{(x1)})^{2+}\,(U_{(x2)})^{2+}\,(U_{(x3)})^{2+}\,(U_{(x4)})^{2}}.$$

Expanded uncertainty = Combined uncertainty × confidence level.

3. Results and discussions

Several parameters were estimated for validation of method for analysis of potassium in fertilizer samples, including repeatability, reproducibility, detection limit, quantification limit, calibration curve and linearity. A method working range, measurement uncertainty, recovery, correlation coefficient, selectivity, and bias were also determined.

3.1. Precision

Generally, the measurement of precision was done through repeatability and reproducibility as the relative standard deviation of data (potasium concentration %). The repeatability of potassium (K) was performed under similar environment (i.e., same operator, glassware, laboratory and within short time interval). The measurement of repeatability was calculated as the relative standard deviation quantified as repeatability relative standard deviation (RSD) which was 0.331 %. The results of repeatability are given in Table 1.

While the reproducibility of K was examined if the instrument (flame photometer) reading of K standards was always exactly reproducible (same for various parameters). This was considered only for the error that comes from the system and not towards those errors attributed to handling of sample as well as sample preparation (Eka et al., 2012; Horwitz and Latimer, 2005; Pointner et al., 2014; Ullah et al., 2017).

The reproducibility data of two analysts performing K_2O analysis on flame photometer at different times was calculated by the application of T-test which showed that the T-calculated (0.690) is less than T-tabulated (2.262). Thus, the results were non-significant to each other, and method can furnish reproducible results. One the other hand, duplicating analysis with relative standard deviation of ± 0.331 and ± 0.400 %, respectively performed by the two analysts working independently at different time interval. Reproducibility is considered as successful; hence, parameter is graded as pass. The results of the reproducibility are given in Table 2. According to the maximum values of relative standard deviation (RSD), which are acceptable for the analyst concentration of 1 μg/L is about 16%. Thus, the method can furnish reproducible results. Reproducibility is considered as successful; hence, the parameter is graded as pass (González et al., 2010; González and Herrador, 2007; Uno, 2016).

3.2. Method limit of detection (LOD) and limit of quantification (LOQ)

The sensitivity of flame photometer was evaluated by calculating LOD and LOQ. The LOD is the minimum concentration of any substance which can only be detected and obviously differentiated from the zero; however, not necessarily quantified. The LOQ is defined as the minimum quantity of any substance which can be measured with an acceptable limit in terms of the precision as well as accuracy (González et al., 2010; Renger et al., 2011). The LOD and LOQ in this experiment were 0.87% and 2.88%, respectively for K_2O using flame photometry technique. The data of 10 spiked samples was employed to calculate together the LOD and LOQ parameters (Table 3).

Table 1
Repeatability for analysis results of potassium fertilizer (SOP).

Repeat	K_2O (63.05%)
1	61.9
2	61.8
3	62.0
4	61.8
5	62.1
6	61.5
7	61.9
8	61.7
9	61.6
10	61.5
Average	**61.78**
Standard deviation	**0.2044**
RSD%	**0.331**

Table 2
Reproducibility of potash test result.

Repeat	Analyst-1 K_2O%	Analyst-2 K_2O%
1	61.9	61.8
2	61.8	62.1
3	62.0	61.5
4	61.8	61.9
5	62.1	61.7
6	61.5	61.6
7	61.9	62.0
8	61.7	61.3
9	61.6	61.7
10	61.5	61.5
Average	61.78	61.71
Standard deviation %	0.2044	0.2470
RSD %	0.331	0.400
T-calculated =	0.690	
T-tabulated =	2.26 at 95% CI	

3.3. Recovery

The method accuracy was assayed through the calculation of K recoveries. To check the method accuracy, the recovery study was executed to confirm K losses due to contamination during sample preparation as well as matrix interferences during the analysis. Taverniers et al. (2004) reported that for analyte concentration of 1 μg/ mL, the acceptable range of the recovery is 95% to 105%. In the present study the recorded recovery (98%) was within suggested range of criteria, i.e., ±5% of recovery (Table 4), hence, the method is verified in this respect and is marked as pass.

3.4. The method working range

To accomplish the requirements of ISO 17025, the working range of the method must be calculated. In this study, LOQ was 2.82 % (Table 4) and LOD was 0.87%. Hence, we can safely consider the range as 2.82% to 63.05 % K_2O (potash) starting from limit of quantification (LOQ). If we consider LOD of starting point, then the working range will be 0.87 to 63.05% K_2O.

3.5. Selectivity

Method selectivity of any analytical method refer to the extent to which the specific method can determine the presence of specific analytical parameters in a complex mixture (matrix) without interference from other analytical parameters. To determine K selectivity of method, a sample of nutrients mixture N + P + K was analyzed. The data is given in Table 5. The K contents were identified with complete recovery (98%) without significant interference of other nutrients in product samples containing K within LOQ (i.e., 2.82%) in a mixture as per the formulation, hence parameter is passed.

Table 3
Evaluation of method limit of detection (LOD) and quantification (LOQ).

	Repeat	K_2O (%)	Standard deviation	Slope	LOD (%)	LOQ (%)
Potassium Chloride Analytical grade	1	61.9	**0.20**	**0.288**	**0.87**	**2.88**
K_2O (63.05%)	2	61.8				
	3	62.0				
K47133836 603	4	61.8				
	5	62.1				
	6	61.5				
	7	61.9				
	8	61.7				
	9	61.6				
	10	61.5				

Table 4
Evaluation of K_2O recovery.

Standard sample	Expected K_2O (%)	Observed K_2O (%)	Recovery (%)	Range (±5 %)	Remarks
Potassium chloride, AR grade (63.05% K_2O)	63.05	61.78	98%	95–105	Pass

Table 5
Evaluation of method selectivity.

Expected K_2O	Analysis results of mixture (NPK No. 62)	K_2O Recovery (95–105%)
K_2O = 20% w/w	N = 19.6% w/w, P_2O_5 = 19.35% w/w, K_2O = 19.6% w/w	98.00

Table 6
Bias (PT results for potash (K_2O) (Magruder Fertilizer Sample Check Program, USA).

Analyte	SWTL, D. G. Khan	Z score	Number of labs	Remarks
Soluble K_2O (Sample # 200111, Magruder USA, issue date 29.02.2021)	3.996	0.02	93	PT Qualifies
Potassium as (K_2O) (Sample # 210211, Magruder USA, issue date 30.09.2020)	21.18	0.95	82	PT Qualifies
Soluble K_2O (Sample # 210411, Magruder USA, issue date 31.05.2021)	61.68	−0.40	83	PT Qualifies
Soluble K_2O (Sample # 210611, Magruder USA, issue date 07.31.2021)	18.92	0.06	85	PT Qualifies

Table 7
Measurement of uncertainty For Estimation of potassium in fertilizer by flame photometry method.

S/N	Sources of Uncertainty	Uncertainty	Type A/B	K Factor	Uncertainty Contribution	Average or Value	Relative Uncertainty
1	Analyst	0.7540	A	1	0.754	49.437	0.015251
2	Equipment)	0.1000	B	2	0.051020408	100	0.000510
3	Vol. flask 100 ml	1	B	2	0.510204082	100	0.005102
4	Pipett 01 ml	0.01	B	2	0.005102041	1	0.005102
5	Pipett 10 ml	0.01	B	2	0.005102041	10	0.000510
6	Analytical Balance	0.0001	B	2	5.10204E-05	200	2.55102E-07
7	CRM	4	B	2	2.040816327	1000	0.0020408
8	Environment	0.56	A	1	0.56	25.14	0.0222752
	Combined Uncertainty	0.02802766	**@**	**95 % CL**			
	CL (K)	2	2	**2**			
	Expanded Uncertainty	0.05605532	**@**	**2**			

3.6. Bias

The difference among the expected test results with respect to an accepted reference value is bias. The test results (Table 6) are within an acceptable range of Z score. Hence, the parameter is regarded as passed.

3.7. Measurement of uncertainty

According to Sunilkumar et al. (2020), uncertainty is expressed in both negative and positive forms (±). The uncertainty budget comprised of both types of sources. The standard deviation of the repeatability as well as reproducibility was used for the calculation

of type-A uncertainty then it was employed for the measurement of standard uncertainty. Whereas the uncertainty of type-B was obtained from calibration certificates etc. First relative uncertainty was calculated then it was multiplied by the confidence level to calculate the expanded uncertainty. The uncertainty of the method under study was ±0.05605532 at 95% confidence interval (Table 7).

4. Conclusion

It is concluded that the tested method fulfilled all validation parameters of ISO/IEC 17025 standard. Moreover, it is the easiest, simple, precise, and accurate method for the estimation / analysis of potassium in fertilizer.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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