

**FERTILIZER
METHODS**

Chapter

POTASH ANALYSIS

Subject

Potash~AA

SCOPE: This is an automated analytical procedure for the determination of potash in mixed or pure material fertilizer samples.

PRINCIPLE: This method outlines the procedure for determining potash in liquid and dry fertilizer samples. Samples are digested in deionized water and analyzed by atomic absorption (AA) spectroscopy. By measuring the amount of light absorbed at the specific potassium (K) wavelength, a quantitative determination of the amount of potassium present can be made. The atomic absorption spectrometer is calibrated to read K₂O ppm.
 Note: The sources for potash include potassium chloride (muriate) and sulfate of potash magnesia. Although these compounds do not present any interferences in the potash analysis, the relationship between the different elements does affect the chlorine/Potash ratio.

SAFETY: Each laboratory is responsible for maintaining a current awareness file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

APPARATUS & EQUIPMENT:

- Perkin-Elmer AAnalyst 100 Atomic Absorption Spectrometer (or equivalent)
- Hamilton Digital Diluter (optional) or equivalent pipets
- Volumetric flasks (Class AA@) 250 mL, 500 mL and 1 L
- Plastic funnel
- Culture tubes (16 x 125 mm or equivalent)
- Vortex shaker
- Seraclear Filters (Westco) or equivalent filter paper

REAGENTS & CHEMICALS:

- Deionized Water
- Potassium Chloride (KCl) Standard (National Institute of Standards and Technology (NIST)-Certified 99.99%) - dried at 100° C for four hours.
 - A) 500 ppm K₂O Stock Standard
 - Weigh 0.791 g of dried KCl (NIST Standard) into a 1 L volumetric flask. Bring to volume with deionized water and mix solution.
 - B) 5, 10, 12.5, 15 ppm K₂O Working Standards (AA) Calibration
 - 1. 5 ppm K₂O standard, - make a 1/100 dilution of the 500 ppm K₂O stock standard using the Hamilton Digital Diluter; the diluter settings are 99/10. (See syringe sizes below)
 - 2. 10 ppm K₂O standard, - make a 1/50 dilution of the 500 ppm K₂O stock standard using the Hamilton Digital Diluter; the diluter settings are 98/20. (See syringe sizes below)
 - 1. 12.5 ppm K₂O standard, - make a 1/40 dilution of the 500 ppm K₂O stock standard; the diluter settings are 78/20. (See syringe sizes below)
 - 2. 15 ppm K₂O standard, - make a 1/33.33 dilution of the 500 ppm K₂O stock standard; the diluter settings are 71/22. (See syringe sizes below)

NOTE: The Hamilton Digital Diluter Syringe sizes used are 10 and 1 mL respectively.

Standard (K ₂ O) Concentration	Hamilton Diluter Setting 10 mL/1 mL	Dilution
5.0 ppm	99/10	1/100
10.0 ppm	98/20	1/50
12.5 ppm	78/20	1/40
15.0 ppm	71/22	1/33.33

- Boiling stones
- Antifoam B Emulsion Solution
 - Dilute 100 mL of the concentrated Antifoam B Emulsion to 500 mL total volume with deionized water.
- Cesium Chloride Certified grade or better - 0.1% solution – (Optional; See Sample Handling Note)
 - Weigh 0.1 g cesium chloride into a 100 mL flask and bring to volume with

deionized water.

NOTE: The following materials are used as internal standards

- **Magruder Collaborative Sample**
Weigh an appropriate amount of a suitable Magruder Collaborative Sample into a 500 mL volumetric flask and proceed with Sample Preparation steps 1 through 6. The Magruder Collaborative Sample is diluted and analyzed with each potash set to check the precision and accuracy of the dilution step and instrument performance by monitoring the mean value of the %K₂O in the Magruder Collaborative Sample.
- **Potassium Chloride (Fisher Reagent Grade 99% or equivalent) Internal Standard**
Dry the potassium chloride (KCl) at 100° C for four hours. Weigh 50 g of the dried potassium chloride into a clean, dry beaker. Transfer to a 500 mL volumetric flask. Bring to a final volume of 500 mL with deionized water and mix. This standard is weighed with each set to check method, dilution step, and instrument performance. The standard must be at room temperature before bringing to volume.
- **Fisher Potassium (Certified Atomic Absorption Standard or equivalent) Reference Solution (1000 ppm + or - 1%)**
Weigh 250.0 g of the 1000 ppm potassium standard into a 500 mL volumetric flask. Bring to volume with deionized water and mix. This is the auto-diluter check standard (K₂O theoretical ppm reading with a 1/50 dilution - 12.05).

SAMPLE

HANDLING:

Potash samples containing a mix of sulfate of potash (potassium sulfate), potassium chloride (muriate), ammonium sulfate and polyammonium phosphate may need to be diluted four-fold by weight since these samples are slurries. The solubility of potassium sulfate in water decreases in the presence of potassium chloride and/or ammonium sulfate and is practically insoluble in a saturated solution of the latter. The polyammonium phosphate may also play a role in this insolubility.

Note: If the four-fold dilution does not correct the deficiency, ionization can be controlled by the addition of an alkali salt (0.1% cesium as a chloride) to samples and standards. Strong concentrations of mineral acids may also cause the potassium signal to be depressed.

SAMPLE

PREPARATION:

1. Weigh the appropriate sample weight (See Table 1) into a 250 mL volumetric flask.
2. Rinse neck of flask with deionized water, add a couple of boiling stones if sample contains organic matter.

3. Fill flask approximately half full with deionized water and place on hot plate.
4. Digest for 30 minutes at a slow boil, if sample foams, add 1 to 3 mL of Antifoam B Emulsion Solution to flask. Remove samples from hot plate and allow to cool to room temperature before bringing to volume with deionized water and mixing.
5. Use seraclear filters to filter samples containing free-floating or particulate matter.
6. Deficient samples with a guarantee > 30%: A 2 g sample is reweighed into a 1 L volumetric flask and proceed with Sample Preparation steps 2 through 6.

TABLE 1 - SAMPLE WEIGHT AND DILUTIONS

%K ₂ O Guarantee	Sample Weight +/- .1 g	Hamilton Diluter Setting	Dilution
0-3	4.00	98/20	1/50
4	3.00	98/20	1/50
5	2.50	98/20	1/50
6-7	2.00	98/20	1/50
8-9	1.50	98/20	1/50
10-11	1.25	98/20	1/50
12-14	1.00	98/20	1/50
15-21	0.75	98/20	1/50
22-32	0.50	98/20	1/50
> 32	0.30	99/10	1/100

NOTE: When weighing liquids that have been diluted, multiply the weight to be taken by the dilution factor so the ppm reading will still read around 10 ppm on the atomic absorption spectrometer.

SAMPLE ANALYSIS:

Atomic absorbance settings used:

- Wavelength - 769.9 nm
- Slit Width - 1.4 nm
- Straight Burner Head Alignment
- 10 ppm K₂O Absorbance Reading - 0.230 to 0.240 absorption units

- Fuel (Acetylene) Flow = 1.0 L/minute
- Oxidant (Air) Flow = 10.0 L/minute

CALCULATION:

$$\% K_2O = \frac{(ppm K_2O \text{ reading})(250 ml)}{\text{sample weight}(g) \times 10^6 \text{ mcg/g}} \times \text{dilution} \times 100$$

APPROVAL:

Approved by: Leigh Humphreys Date: 4/3/02
 Signature

Bureau Chief

Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	6/11/98	Replaces K-100	J. Corry
Revision	4/03/02		G. H. Huang

REFERENCE:

Analytical Methods for Atomic Absorption Spectrometry, Perkin Elmer, p 149