# Potassium, atomic absorption spectrometric, direct-EPA

Parameter and Code:
Potassium, total recoverable, I-3631-85 (mg/L as K): 00937

## 1. Application

1.1 This method may be used to analyze water-suspended sediment. Sample solutions containing from 0.1 to 100 mg/L of potassium may be analyzed without dilution, but those containing more than 100 mg/L need to be diluted.

1.2 For ambient water, analysis may be made on a portion of the acidified water-suspended sediment sample.

1.3 For all other water, including domestic and industrial effluent, the atomic absorption procedure must be preceded by a digestion-solubilization as specified below. In cases where the analyst is uncertain about the type of sample, the digestion-solubilization procedure must be used.

## 2. Summary of method

2.1 Potassium is determined by atomic absorption spectrometry by direct aspiration of the filtered or digested and filtered sample into an air-acetylene flame (Fishman and Downs, 1966).

2.2 Effluent samples must undergo a preliminary nitric acid digestion followed by a hydrochloric acid solubilization.

## 3. Interferences

3.1 Of the substances commonly occurring in water, only sodium has been found to interfere, and its interference is greatly minimized if a reducing flame is used and the burner is raised to approximately 0.05 cm below the optical light path. The following data are indicative of the magnitude of sodium interference under these conditions:

| Potassium added (mg/L) | Sodium added (mg/L) | Potassium found (mg/L) |
|---|---|---|
| 1.00 | 0.0 | 1.00 |
| 1.00 | 100 | 1.03 |
| 1.00 | 500 | 1.05 |
| 1.00 | 1000 | 1.07 |

3.2 Alternatively, an excess of sodium or cesium may be added to all samples and standards. Such additions essentially eliminate the effects of the then comparatively minor contributions of sodium present in the samples.

## 4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

| | |
|---|---|
| Grating | Visible |
| Wavelength | 766.5 nm |
| Source (hollow-cathode lamp) | Potassium |
| Oxidant | Air |
| Fuel | Acetylene |
| Type of flame | Slightly reducing |

4.3 The 50-mm (2-in.), flathead, single-slot burner allows a working range of 0.1 to 10 mg/L. The burner, rotated 90°, extends the range to 100 mg/L. Different burners may be used according to manufacturers' instructions.

## 5. Reagents

5.1 *Hydrochloric acid,* 6*M*: Dilute 500 mL concentrated HCl (sp gr 1.19) to 1 L with demineralized water.

5.2 *Hydrochloric acid,* 0.3*M*: Dilute 25 mL concentrated HCl (sp gr 1.19) to 1 L with demineralized water.

5.3 *Nitric acid,* concentrated, (sp gr 1.41).

5.4 *Potassium standard solution I,* 1.00 mL = 0.100 mg K: Dissolve 0.1907 g KCl, dried at 180°C for 1 h, in demineralized water and dilute to 1,000 ML.

5.5 *Potassium working standards:* Prepare a series of at least six working standards containing from 0.1 to 100 mg/L of potassium by appropriate dilution. of potassium standard solution.

## 6. Procedure

6.1 Transfer the entire sample to a beaker.

6.2 Rinse the sample bottle with 3 mL concentrated $HNO_3$ for each 100 mL of sample and add to the beaker. Prepare a blank using 3 mL concentrated $HNO_3$ per 100 mL demineralized water.

6.3 Evaporate samples and blank to dryness on a hotplate, making sure the samples do not boil.

6.4 Cool and add an additional 3 mL concentrated $HNO_3$ to each beaker. Cover with a watchglass, return to the hotplate, and gently reflux the samples.

6.5 Continue heating, adding additional acid as necessary, until the digestion is complete (indicated by a light-colored residue). Evaporate just to dryness.

6.6 Add 6 mL 6*M* HCl solution per 100 mL original sample and warm the beaker to dissolve the residue.

6.7 Wash the watchglass and beaker with demineralized water and filter the sample (Whatman No. 41 or equivalent), rinsing the filter with hot 0.3*M* HCl. Dilute to the original volume with demineralized water.

6.8 While aspirating the blank use the automatic zero control to set the digital display to read zero concentration. While aspirating standards use the automatic concentration control to set the digital display to read the concentration of standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

## 7. Calculations

Determine the milligrams per liter of total recoverable potassium in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing potassium concentrations that exceed the working range of the method and multiply by the proper dilution factors.

## 8. Report

Report potassium, total-recoverable (00937), concentrations as follows: less than 10 mg/L, one decimal; 10 mg/L and above, two significant figures.

## 9. Precision

It is estimated that the percent relative standard deviation for total recoverable potassium over the range of the method will be greater than 13 percent.

## References

Fishman, M. J., and Downs, S. C., 1966, Method for analysis of selected metals in water by atomic absorption: U.S. Geological Survey Water-Supply Paper 1540-C, p. 36-8.

U.S. Environmental Protection Agency, 1979, Methods for chemical analysis of water and wastes: Cincinnati, p. 258.1-1.