

Sodium, atomic absorption spectrometric, direct-EPA

Parameter and Code:

Sodium, total recoverable, I-3736-85 (mg/L as Na): 00929

1. Application

1.1 This method may be used to analyze water-suspended sediment containing from 0.1 to 80 mg/L of sodium. If the sodium concentration exceeds 80 mg/L, the sample solution needs to be diluted. The method is very sensitive and can be extended to much lower sodium concentrations.

1.2 For ambient water, analysis may be made on an aliquot 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 of the type of sample, this procedure must be followed.

2. Summary of method

2.1 Sodium 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

None of the substances commonly occurring in natural water interfere with this method.

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 ----- 588.8 nm
Source (hollow-cathode
lamp) ----- Sodium
Oxidant ----- Air
Fuel ----- Acetylene
Type of flame ----- Oxidizing

4.3 The 50-mm (2-in.), flathead, single-slot burner, rotated 90°, allows a range of 0.1 to 80 mg/L. Different burners may be used according to manufacturers' instructions.

5. Reagents

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

5.2 *Hydrochloric acid, 0.3M*: 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 *Sodium standard solution*, 1.00 mL = 1.00 mg Na: Dissolve 2.542 g NaCl in demineralized water and dilute to 1,000 mL.

5.5 *Sodium working standards*: Prepare a series of at least six working standards containing from 0.1 to 80 mg/L sodium by appropriate dilutions of sodium standard solution. The preparation of an intermediate standard solution is desirable when preparing working solutions of extreme dilution.

6. Procedure

6.1 When analyzing samples of ambient waters, begin the analysis at step 6.9.

6.2 Transfer the entire sample to a beaker.

6.3 Rinse the sample bottle with 3 mL concentrated HNO₃ 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.

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

6.5 Cool and add an additional 3 mL concentrated HNO_3 to the beaker. Cover with a watchglass, return to the hotplate, and gently reflux the solution.

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

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

6.8 Filter (Whatman No. 41 or equivalent) the sample and wash the watchglass and beaker with demineralized water. Rinse the filter with hot 0.3M HCl. Dilute to the original volume with demineralized water.

6.9 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 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 sodium in each sample from the digital display or printer while aspirating each sample. Dilute those samples containing sodium concentrations that exceed the working range of the method and multiply by the proper dilution factors.

8. Report

Report sodium, total-recoverable (00929), 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 sodium is greater than 13 percent at 2.76 mg/L and greater than 5 percent at 96.9 mg/L.

References

- Fishman, M. J., and Downs, S.C., 1966, Methods for analysis of selected metals in water by atomic absorption: U.S. Geological Survey Water-Supply Paper 1540-C, p. 38-41.
U.S. Environmental Protection Agency, 1979, Methods for chemical analysis of water and wastes: Cincinnati, p. 273.1-1.