

Nitrogen, nitrite, low ionic-strength water, colorimetry,  
diazotization, automated-segmented flow

Parameter and Code:

Nitrogen, nitrite, dissolved, I-2542-89 (mg/L as N): 00613

## 1. Application

This method is used to analyze samples of precipitation or natural water containing from 0.001 to 0.20 mg/L of nitrite-nitrogen. Concentrations greater than 0.20 mg/L must be diluted. This method was implemented in the National Water Quality Laboratory in March 1986 and modified in May 1989.

## 2. Summary of method

Nitrite ion reacts with sulfanilamide under acidic conditions to form a diazo compound which then couples with N-1-naphthylethylenediamine dihydrochloride to form a red compound, the absorbance of which is measured colorimetrically (Shinn, 1941; Bendschneider and Robinson, 1952; Fox, 1979, 1985; Pai and others, 1990).

## 3. Interferences

Concentrations of potentially interfering substances generally are negligible. For specific details of inorganic and organic compounds that interfere with the reaction, see Norwitz and Keliher (1985, 1986) as well as more general information from the American Society for Testing and Materials (1991).

## 4. Apparatus

4.1 *Alpkem rapid flow analyzer (RFA)*, consisting of sampler, peristaltic pump, analytical cartridge, colorimeter, data station, and printer.

4.2 With this equipment, the following operating conditions are satisfactory for the range from 0.001 to 0.20 mg/L N:

Flow cell .....	10 mm
Wavelength .....	540 nm
Sampling rate .....	64 per hour
Sample time .....	24 seconds
Wash time .....	32 seconds
Pecking .....	OFF
Damp (RC) .....	1 second

## 5. Reagents

5.1 *Color reagent*: Add 200 mL of concentrated phosphoric acid (sp gr 1.69) and 20 g sulfanilamide to about 1,500 mL demineralized water. Dissolve completely (warm if necessary). Add 1.0 g N-1-naphthylethylenediamine dihydrochloride and dissolve completely. Dilute to 2 L with demineralized water. Add 1 mL Brij-35 solution. Store in an amber bottle and refrigerate. The reagent is stable for about 1 month.

5.2 *Nitrite-nitrogen standard solution I*, 1.00 mL = 0.100 mg  $\text{NO}_2\text{-N}$ : Dissolve 0.6076 g  $\text{KNO}_2$ , dried overnight over sulfuric acid, in demineralized water and dilute to 1,000 mL. This solution and the following nitrite standard solutions (paragraphs 5.3 and 5.4) are not stable indefinitely; their concentrations need to be checked frequently.

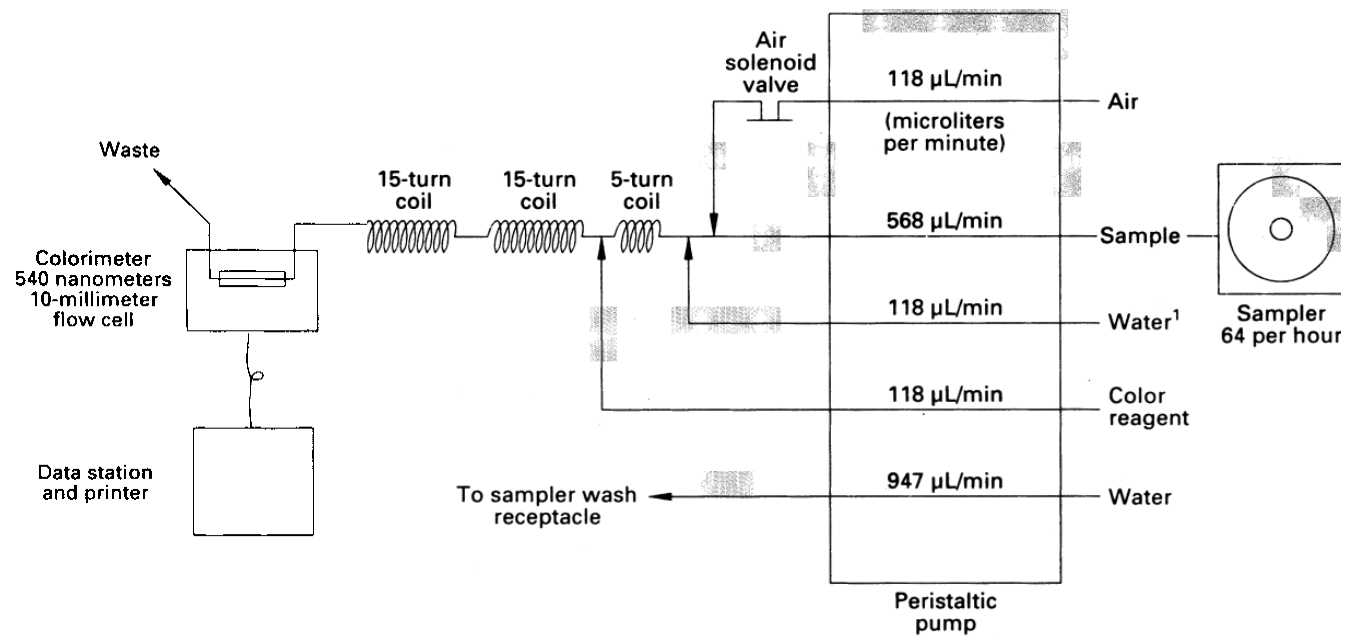
5.3 *Nitrite-nitrogen standard solution II*, 1.00 mL = 0.001 mg  $\text{NO}_2\text{-N}$ : Dilute 10.0 mL nitrite-nitrogen standard solution I to 1,000 mL with demineralized water.

5.4 *Nitrite-nitrogen working solutions*: Prepare a blank and 200 mL of a series of working solutions by appropriate dilution of nitrite-nitrogen standard solution II or working solution No. 3, as listed in the following table. If the samples to be analyzed are preserved, the nitrite-nitrogen working solutions need to contain an equivalent concentration of the same preservative.

<u>Working solution No.</u>	<u>Solution added (m L)</u>	<u>Solution used</u>	<u>Nitrite-nitrogen concentration (mg/L)</u>
1	40	Standard solution 11	0.200
2	20	Standard solution II	0.100
3	10	Standard solution II	0.050
4	2	Standard solution 11	0.010
5	20	Working solution No. 3	0.005
6	8	Working solution No. 3	0.002
7	4	Working solution No. 3	0.001

## 6. Procedure

6.1 Set up manifold (fig. 1).



<sup>1</sup> Contains 0.5-milliliter Brij solution per liter.

Figure 1. - Nitrogen, nitrite, low ionic strength water, diazotization manifold.

6.2 Allow colorimeter recorder to warm for at least 10 minutes,

6.3 After all reagents are on line, adjust the sample output of the photometer to 5 V. Then switch the photometer to "absorbance" mode and use the reference detector "fine gain" control to adjust the baseline absorbance to about 0.2 V. See operation manuals for complete details (Alpkem Corp., 1986)

6.4 Place the most concentrated working solution in two cups before analysis. As the peaks appear on the recorder, adjust the STD CAL control until the peak obtains 95 percent of full scale.

6.5 When the system is clear of all working solutions, determine a dwell time using the most concentrated working solution.

6.6 Place a complete set of working solutions and a blank in the first positions of the sample tray beginning with the most concentrated working solution. Place individual working solutions of differing concentrations in approximately every eighth position on the tray following the accepted protocol. Fill the remainder of each tray with unknown samples.

6.7 Begin analysis.

## **7. Calculations**

7.1 Prepare an analytical curve by plotting the voltage of each working solution peak in relation to its respective nitrite-nitrogen concentration, or by using the RFA Softpac data reduction package. See operation manuals for complete details (Alpkem Corp., 1986).

7.2 Compute the concentration of dissolved nitrogen in each sample either by comparing its voltage to the analytical curve or by using the software. Any baseline drift needs to be accounted for when computing the voltage of a sample or working solution peak; the RFA software automatically corrects for baseline drift.

## **8. Report**

Report concentrations of nitrite-nitrogen, dissolved (00613), as follows: less than 0.10 mg/L, three decimals; 0.10 mg/L and greater, two significant figures.

## 9. Precision

Single operator precision for nitrite-nitrogen, as determined for natural water samples, expressed as standard deviation and percentage relative standard deviation, is as follows:

<u>Number of determinations</u>	<u>Mean (mg/L)</u>	<u>Standard deviation (mg/L)</u>	<u>Relative standard deviation (percent)</u>
22	0.011	0.001	9.1
22	0.063	0.002	3.2
20	0.144	0.003	2.1

### References

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