PICKERING LABORATORIES

Sensitive and Selective Analysis of Nitrite and Nitrate in Drinking Water by Ion Chromatography (IC) with Post-Column Derivatization and UV/VIS Detection

Nitrite and Nitrate are formed naturally in soil and water when bacteria break down waste and organic material from plants, humans and animals. Nitrate is also one of the main components of chemical fertilizers. Contamination of the drinking water supply with Nitrite and Nitrate occurs due to runoff and seepage into ground water from farms, golf courses, landfields, improperly managed animal feedlots and sewage systems.

Under certain conditions, the human body converts Nitrate to Nitrite, which can react with hemoglobin in blood and decrease its ability to carry oxygen. Nitrite can also form a variety of N-Nitroso compounds, many of which are known carcinogens. Elevated concentrations of Nitrite and Nitrate in water are especially dangerous to infants younger than 6 months old and pregnant women. To prevent harmful health effects from Nitrite and Nitrate contamination of drinking water, the USEPA has established Maximum Contamination Levels (MCLs) at 10 ppm for Nitrate and 1 ppm for Nitrite.

The USFDA method for Nitrite and Nitrate calls for post-column derivatization using a Vanadium (III) Chloride reagent containing HCl. Pickering Laboratories has improved this method by substituting the volatile and corrosive Hydrochloric Acid with Methanesulfonic Acid. When coupled with modern IC systems and columns, the Pickering post-column derivatization systems allow for fast, sensitive and selective analysis of Nitrite and Nitrate in drinking water, and additionally in food matrices*.

Method

Analytical Conditions

IC System: ICS 900 or equivalent IC system (Thermo Scientific)

Analytical Column: IonPac AS9-HC, 4 x 250 mm (Thermo Scientific)

Flow Rate: 1 mL/min

Column Temperature: 30 °C

Mobile Phase: 9.0 mM Sodium Carbonate

Injection Volume: 20 uL

Post-Column Conditions

Post-Column System: Onyx PCX, Pinnacle PCX or Vector PCX

Reactor Volume: 0.5 mL

ReactorTemperature: 100 °C

Reagent Flow Rate: 0.1 mL/min

Detection: UV/VIS, 535 nm

Reagent : Mix 50 mL of (i) and (ii) and 1.25 mL of (iii) in 250 mL volumetric flask. Bring to volume with 20/80 Methanesulfonic Acid / Water.

(i) 1% Vanadium (III) Chloride in 20/80 Methanesulfonic Acid / Water.

(ii) 1% m-Nitro Aniline in 20/80 Methanesulfonic Acid / Water.

(iii) 1% N-(1-Naphthyl)ethylenediamine Dihydrochloride in 20/80 Methanesulfonic Acid / Water.

Table 1. Recoveries Data for Nitrite and Nitrate

Spike Concentration	Nitrite	Nitrate
10 ppm	100.6%	102.4%
0.5 ppm	97.5%	85.5%
0.5 ppm in 100 ppm NaCl	100.1%	87.1%

*Inquire about Pickering Laboratories Method Abstract 123 "Simultaneous Determination of Nitrite and Nitrate in Processed Foods" and Method Abstract 121 "Nitrite and Nitrate Analysis in Baby Food"

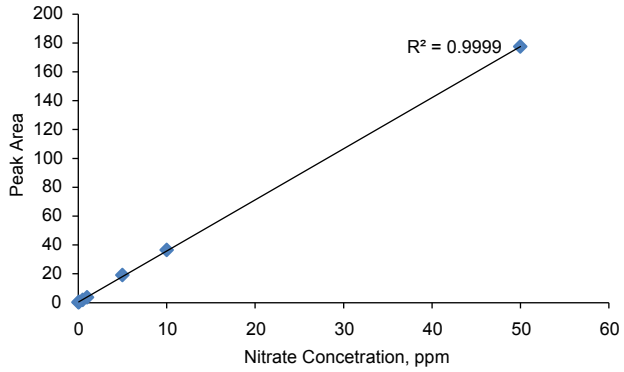

Fig 1. *Calibration Curve for Nitrate 0.05-50 ppm range*

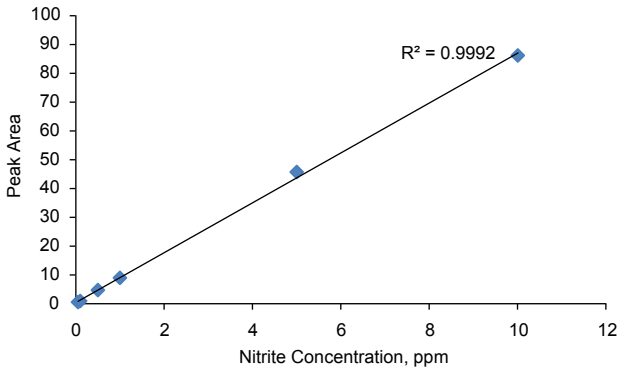

Fig 2. *Calibration Curve for Nitrite 0.05-10 ppm range*

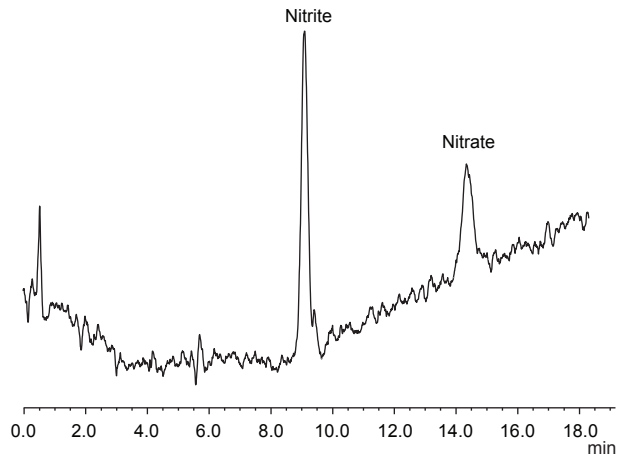

Fig 3. *Chromatogram of 0.05 ppm Nitrite and 0.05 Nitrate solution.*

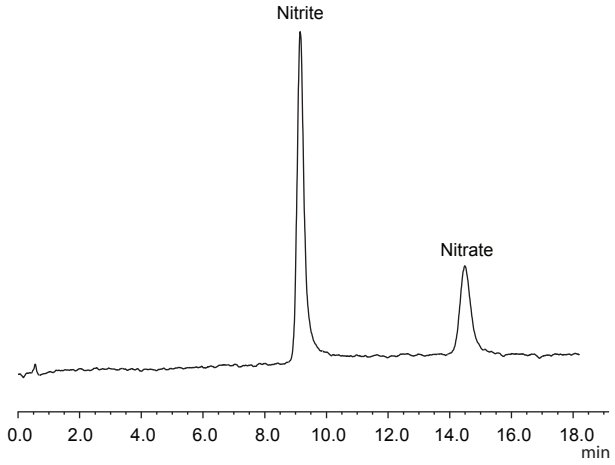

Fig 4. *Chromatogram of 0.5 ppm Nitrite and 0.5 ppm Nitrate solution in presence of 100 ppm NaCl.*

References

1. Official Methods of Analysis of AOAC International (2000) 17th Ed, Section 50.1.11
2. Use of Griess Reagents Containing Vanadium (III) for Post-Column Derivatization and Simultaneous Determination of Nitrite and Nitrate in Baby Food. John A. Casanova, Lois K. Gross, Sarah E. McMullen and Frank Schenk, Food and Drug Administartion, 60 8th Street, Atlanta, GA 30309