

NITROGEN DIOXIDE IN WORKPLACE ATMOSPHERES
(ION CHROMATOGRAPHY)



Method Number: ID-182 (This method supersedes ID-109)

Matrix: Air

OSHA Permissible Exposure Limits
Final Rule Limit: 1 ppm Short-Term Exposure Limit (STEL)
Transitional Limit: 5 ppm Ceiling

Collection Device: Each sample is collected using a sampling tube containing triethanolamine-impregnated molecular sieve (TEA-IMS) and a calibrated sampling pump.

Recommended Sampling Rate: 0.20 L/min

Recommended Air Volume: 3.0 L (0.20 L/min for 15 min)

Analytical Procedure: The sample is desorbed from the solid sorbent using a 1.5% triethanolamine (TEA) solution. Analysis is performed as nitrite (NO_2^-) by ion chromatography.

Detection Limit
Qualitative: 0.07 ppm (3-L air sample)
Quantitative: 0.19 ppm (3-L air sample)

Precision and Accuracy
Validation Range: 2.64 to 9.45 ppm
 CV_T : 0.034
Bias: +0.13
Overall Error: $\pm 19.8\%$

Method Classification: Validated Method

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Date (Date Revised): December 1987 (May, 1991)

Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Similar products from other sources can be substituted.

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1. Introduction

This method describes the collection and analysis of airborne nitrogen dioxide (NO₂). Samples are taken in the breathing zone of workplace personnel and analysis is performed by ion chromatography (IC).

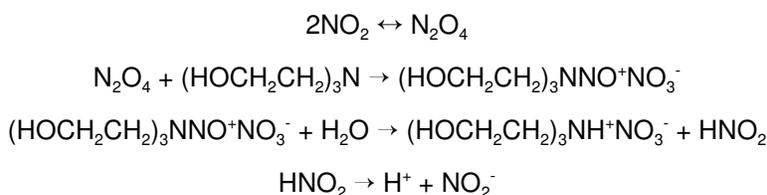
1.1 History

Previous methods of analysis for NO₂ involved collection of nitrogen dioxide in bubblers of triethanolamine (TEA) solution or a triethanolamine-impregnated molecular sieve (TEA-IMS) solid sorbent and TEA extraction (8.1). Nitrogen dioxide exposure was determined colorimetrically by the Griess-Saltzman reaction (8.1-8.3). This method, like most colorimetric procedures, can have significant interferences. A differential pulse polarographic (DPP) method (8.4) was later developed to improve sensitivity and decrease the potential for interferences. The sensitivity of the DPP method was adequate for measuring workplace concentrations of nitrogen dioxide; however, the nitrite ion is unstable at the pH range (pH 1-2) used during analysis (8.5).

Method no. ID-182 uses the collection principle of the TEA-IMS tube. The samples are analyzed by IC to determine NO₂ exposure.

1.2 Principle

A known volume of air is drawn through a sampling tube containing TEA-IMS. Nitrogen dioxide is trapped and converted to nitrite in the presence of TEA and water. Samples are desorbed using an aqueous TEA solution and analyzed as nitrite. The conversion mechanism of NO₂ gas to nitrite ion has been proposed by Gold (8.6). The following is Gold's proposal for the reaction of equivalent amounts of NO₂ and TEA in an aqueous solution:



Nitrogen dioxide disproportionates to nitrite and nitrate ions in the presence of TEA. The nitrite ion (NO₂⁻) formed from the above reaction can be analyzed via conventional analytical methods (8.1-8.5) including IC (8.7). The high background levels of nitrate found in commercial TEA-IMS sorbents ruled out further research to assess this NO₂-TEA disproportionation product by IC.

This reaction path requires a stoichiometric factor of 0.5 for the conversion of gaseous NO₂ to NO₂⁻. Experiments indicate the proposed factor of 0.5 is seen only when NO₂ concentrations are greater than 10 ppm (8.6, 8.8-8.9). The conversion factor has been experimentally determined to average approximately 0.6 to 0.7 when concentrations are below 10 ppm (8.1-8.4, 8.6-8.9). The deviation from ideal stoichiometry is believed to be due to other competing reactions; however, evidence to support this has not been found (8.6).

1.3 Advantages and Disadvantages

- 1.3.1 The analysis is simple, rapid, easily automated, and specific for the nitrite ion.
- 1.3.2 After sample preparation, nitrogen dioxide (as nitrite ion) can also be determined by polarographic or colorimetric analytical techniques (8.1-8.4).
- 1.3.3 Nitric oxide (NO) can also be sampled when using a three-tube sampling device (8.10). Sulfur dioxide may also be screened using the TEA-IMS sampling tube and similar analytical conditions (8.7).
- 1.3.4 A disadvantage is the potential interference from large amounts of soluble chloride salts present in commercial molecular sieve. Prior to TEA impregnation, the molecular sieve should be washed with deionized water to remove any soluble chloride salts.
- 1.3.5 Another disadvantage is the need for a concentration-dependent conversion factor when calculating results.

1.4 Physical Properties (8.11)

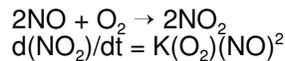
Nitrogen dioxide (CAS No. 10102-44-0), one of several oxides of nitrogen, is a reddish-brown or dark orange gas with a formula weight of 46.01. Its dimer, nitrogen tetroxide (N₂O₄), is colorless. At temperatures between -9.3 and 135 °C, NO₂ and N₂O₄ coexist as a mixture of gases. Below -9.3 °C, a colorless solid consisting of N₂O₄ is formed, while above 135 °C, the gas is mainly composed of NO₂. Physical characteristics of NO₂ are:

| | |
|-----------------------|---|
| Formula weight | 46.01 |
| Specific gravity | 1.448 at 20 °C (liquid) |
| Melting point | -9.3 °C |
| Boiling point | 21.15 °C |
| Vapor pressure | 96 KPa (720 mmHg) at 20 °C |
| Vapor density | 1.58 (air = 1) |
| Other characteristics | strong oxidizer, corrosive, nonflammable |
| Synonyms | dinitrogen tetroxide, nitrogen tetroxide, nitrogen peroxide, liquid dioxide |

1.5 Some sources for potential nitrogen dioxide exposures are:

- agricultural silos
- arc or gas welding (esp. confined space operations)
- electroplating plants
- food and textile bleaching
- jewelry manufacturing
- nitric acid production
- nitrogen fertilizer production
- nitro-explosive production
- pickling plants

Nitrogen dioxide and nitric oxide usually exist together in industrial settings. Nitric oxide is reactive in air and produces NO₂ according to the following equations (8.11):



(K is a temperature dependent constant. At 20 °C, K = 14.8 × 10⁹)

An experimental approximation of the NO/NO₂ distribution found in various industrial operations is shown (8.11).

| Source | % NO ₂ | % NO |
|------------------------------|-------------------|------|
| Carbon arc | 9 | 91 |
| Oxyacetylene torch | 8 | 92 |
| Cellulose nitrate combustion | 19 | 81 |
| Diesel exhaust | 35 | 65 |
| Dynamite blast | 52 | 48 |
| Acid dipping | 78 | 22 |

The potential for exposure to both NO₂ and NO should be considered because NO is easily oxidized to NO₂ and both oxides are likely to coexist in industrial settings.

1.6 Toxicology

Information listed within this section is a synopsis of current knowledge of the physiological effects of nitrogen dioxide and is not intended to be used as a basis for OSHA policy.

1.6.1 Nitrogen dioxide is classified as a respiratory irritant and the route of exposure is mainly inhalation. The term silo-fillers' disease is associated with exposure to nitrogen dioxide as well as other nitrogen oxides.

Unlike the more soluble gases (e.g., chlorine, ammonia) that produce almost immediate upper respiratory tract irritation, symptoms of NO₂ exposure may be delayed for up to 12 hours. The lower solubility of NO₂ provides less warning and increases the potential for physiological damage when exposures occur.

1.6.2 The symptoms from mild exposures (<50 ppm) are generalized below (8.12-8.14):

| | |
|------------------------------------|---------------|
| mucoïd or frothy sputum production | cough |
| painful breathing | fever |
| chest pains | tachycardia |
| increased breathing rate | lymphocytosis |

Exposures usually result in an increased susceptibility to respiratory infections. Changes in pulmonary function are evident when healthy subjects are exposed to 2 to 3 ppm NO₂ and can occur at far lower concentrations in asthmatic subjects.

More severe exposures (>50 ppm) are characterized by pulmonary edema, cyanosis, bronchiolitis obliterans, respiratory failure and death.

1.6.3 The LC₅₀ (Lethal Concentration 50) for a 4-hour exposure is approximately 90 ppm NO₂.

2. Range, Detection Limit and Sensitivity (8.8)

2.1 This method was evaluated over the concentration range of 2.64 to 9.45 ppm. An air volume of 3 L and a flow rate of 0.2 L/min were used. Samples were taken for 15 min. Sample results were calculated using an average conversion relationship of:

$$1 \mu\text{g NO}_2 = 0.63 \mu\text{g NO}_2^-$$

At NO₂ concentrations above 10 ppm, the conversion factor has been shown to decrease, approaching a value of 0.5 (8.6, 8.8-8.9).

2.2 The qualitative detection limit was 0.08 µg/mL or 0.24 µg (as NO₂⁻) when using a 3-mL solution volume. This corresponds to 0.07 ppm NO₂ for a 3-L air volume.

2.3 The quantitative detection limit was 0.23 µg/mL or 0.69 µg (as NO₂⁻) when using a 3-mL solution volume. This corresponds to 0.19 ppm NO₂ for a 3-L air volume. A 50-µL sample loop and a detector setting of 3 microsiemens were used for both detection limit determinations.

2.4 The sensitivity of the analytical method was calculated from the slope of a linear working range curve (1 to 20 µg/mL nitrite). The sensitivity for this curve was 222,720 area units per 1 µg/mL (a Hewlett-Packard 3357 data reduction system was used, and 1 area unit = 0.25 microvolt-second).

3. Method Performance (8.8)

3.1 The pooled coefficient of variation (CV_T) for samples taken in the range of 2.64 to 9.45 ppm was 0.034. The method exhibited positive bias (+0.13); however, overall error is within acceptable limits at ±19.8%.

3.2 The collection efficiency at approximately 2 times the PEL was 97.3%. Samples were collected at a generation concentration of 9.45 ppm NO₂ for 15 min. Sample generation conditions were 50% RH and 25 °C.

3.3 Breakthrough tests were performed at 30% RH and a concentration of 21 ppm. Samples were collected for 15 min at a flow rate of 0.18 L/min. Breakthrough of NO₂ into a second sorbent tube at these parameters was 1.6% NO₂. This is within an acceptable limit of <5% breakthrough.

3.4 Samples can be stored at ambient (20 to 25 °C) laboratory conditions for a period of at least 29 days. Storage stability results show the mean of samples analyzed after 29 days was within ±5% of the mean of samples analyzed after one day of storage. Samples were stored on a laboratory bench.

4. Interferences

4.1 When other compounds are known or suspected to be present in the sampled air, such information should be transmitted to the laboratory with the sample.

4.2 Any compound having the same retention time as nitrite, when using the operating conditions described, is an interference.

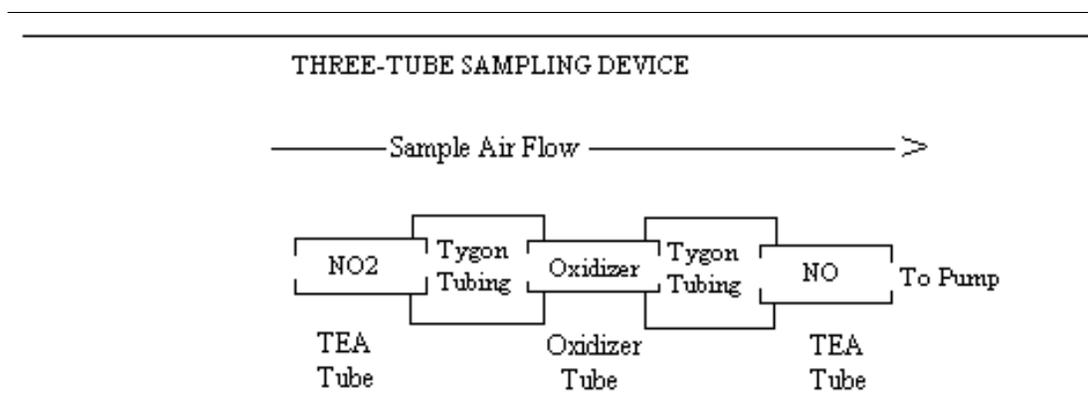
4.3 Interferences may be minimized by changing the eluent concentration, and/or pump flow rate.

- 4.4 If there is reason to suspect an unresolvable interference, alternate polarographic or colorimetric methods can be used (8.1-8.4).
- 4.5 Contaminant anions normally found in molecular sieve, such as NO_3^- , SO_4^{2-} , and PO_4^{3-} , do not interfere. Large amounts (greater than 4 to 5 $\mu\text{g/mL}$) of Cl^- can interfere.

5. Sampling

5.1 Equipment

- 5.1.1 Personal sampling pumps capable of sampling within $\pm 5\%$ of the recommended flow rate of 0.2 L/min are used.
- 5.1.2 Two types of sampling tubes are commercially available (All molecular sieve used for tube packing should be washed with deionized water before impregnation with TEA).
- One type is a two-section tube packed with a 400-mg TEA-IMS front and a 200-mg back-up section (NO_2 sampling tube, Cat. No. 226-40-02-special order, water-washed, SKC, Eighty Four, PA).
 - The other type, a three-tube sampling device (NO/NO_2 sampling tubes, Cat. No. 226-40-special order, water-washed, SKC, Eighty Four, PA) can be used to sample NO_2 and NO simultaneously or individually. The device consists of three flame-sealed glass tubes. Nitrogen dioxide is collected in the first tube which contains 400 mg TEA-IMS. Two other tubes, an oxidizer tube and another 400 mg TEA-IMS packed tube, are also included. The dimensions of each TEA-IMS tube are 7-mm o.d., 5-mm i.d., and 70-mm long. A 3-mm portion of silylated glass wool is placed in the front and rear of each tube. An oxidizer tube containing approximately 1 g of a chromate compound is used to convert NO to NO_2 . The dimensions of the oxidizer tube are 7-mm o.d., 5-mm i.d., and 110-mm long. When the three tubes are connected in series as shown below, NO_2 and NO can be collected simultaneously.



For further information regarding sampling for NO , see reference 8.10.

- 5.1.3 A stopwatch and bubble tube or meter are used to calibrate pumps. A sampling tube or device is placed in-line during flow rate calibration.
- 5.1.4 Various lengths of Tygon tubing are used to connect sampling tubes to pumps.

5.2 Sampling Procedure

Note: If sampling for both NO₂ and NO is necessary, two separate pumps and sampling devices should be used. The differences in OSHA Final Rule PELs (NO₂ is a STEL and NO is a TWA PEL) and flow rates dictates a need for a singular assessment of NO₂. Nitric oxide is collected at a flow rate **not to exceed** 0.025 L/min (8.9-8.10) and a three-tube device must be used. Nitrogen dioxide can be collected at this flow rate; however, a longer sampling time will be necessary to collect a detectable amount of NO₂ than for a short-term measurement. Also, NO₂ concentrations may vary widely during sampling periods as long as 4 hours for NO. The three-tube sampling device will not reflect the varying concentration. Therefore, it is recommended to sample at 0.2 L/min for 15-min intervals using a single or two section tube **for NO₂**. A separate three-tube device and pump is then used **for NO** sampling. The front tube of the device can be submitted for NO₂ analysis; however, results from this front section may not represent short-term exposures.

- 5.2.1 Calibrate the sampling pumps at either recommended flow rate listed in Section 5.2.4.
- 5.2.2 Connect the sampling tube or device to the pump. The different sampling schemes are listed below:
 - a) **Sampling for NO₂ only:** A single TEA-IMS tube taken from the three-tube sampling device (Section 5.1.2, part b) or the two-section tube (Section 5.1.2, part a) can be used. If the two-section tube is used, sampled air should enter the 400 mg section first.
 - b) **Sampling for both NO and NO₂:** The three-tube device (Section 5.1.2, part b) is used. Label the first tube "NO₂". The tube following the oxidizer section is labeled "NO". Also consult reference 8.10.
- 5.2.3 Place the sampling tube or device in the breathing zone of the employee.
- 5.2.4 Sample with pre-calibrated pumps at the listed flow rates and sampling times:
 - a) **For NO₂ only:** 0.2 L/min for at least 15 min per sample.
 - b) **For both NO and NO₂:** 0.025 L/min for 4 h per sample. Also consult reference 8.10.

Nitrogen dioxide results from extended sampling times (>15 min) may not reflect short-term exposures.

- 5.2.5 The minimum recommended total air volume for collecting NO₂ is 3 L.

6. Analysis

6.1 Precautions

- 6.1.1 Refer to instrument and standard operating procedure (SOP) (8.15) manuals for proper operation.
- 6.1.2 Observe laboratory safety regulations and practices.
- 6.1.3 Sulfuric acid (H₂SO₄) can cause severe burns. Wear protective gloves and eyewear when using concentrated H₂SO₄.

6.2 Equipment

- 6.2.1 Ion chromatograph (Model 2010 or 4000, Dionex, Sunnyvale, CA) equipped with a conductivity detector.
- 6.2.2 Automatic sampler (Model AS-1, Dionex) and sample vials (0.5 mL).
- 6.2.3 Data processing system: Ion chromatograph interfaced to a data reduction and control system (Autolon 400 or 450 System, Dionex).
- 6.2.4 Printer.
- 6.2.5 Separator and guard columns, anion (Model HPIC-AS4A and AG4A, Dionex).
- 6.2.6 Micromembrane suppressor, anion (Model AMMS-1, Dionex).

6.2.7 Disposable syringes (1 mL) and pre-filters.

Note: Some syringe pre-filters are not cation- or anion-free. Tests should be done with blank solutions first to determine suitability for the analyte being determined.

6.2.8 Erlenmeyer flasks, 25-mL, or scintillation vials, 20-mL.

6.2.9 Miscellaneous volumetric glassware: Micropipettes, volumetric flasks, graduated cylinders, and beakers.

6.2.10 Analytical balance (0.01 mg).

6.3 Reagents - All chemicals should be at least reagent grade.

6.3.1 Deionized water (DI H₂O) with a specific conductance of less than 10 microsiemens.

6.3.2 Triethanolamine [(HOCH₂CH₂)₃N]
Sodium carbonate (Na₂CO₃)
Sodium bicarbonate (NaHCO₃)
Sulfuric acid (H₂SO₄, concentrated 95 to 98%)
Sodium nitrite (NaNO₂)

6.3.3 Liquid desorber (1.5% TEA):

Dissolve 15 g TEA in a 1-L volumetric flask which contains approximately 500 mL DI H₂O. Add 0.5 mL n-butanol and then dilute to volume with DI H₂O.

6.3.4 Eluent (2.0 mM Na₂CO₃/1.0 mM NaHCO₃):

Dissolve 0.848 g Na₂CO₃ and 0.336 g NaHCO₃ in 4.0 L of DI H₂O.

6.3.5 Regeneration solution (0.02 N H₂SO₄):

Place 1.14 mL concentrated H₂SO₄ into a 2-L volumetric flask which contains about 500 mL DI H₂O. Dilute to volume with DI H₂O.

6.3.6 Nitrite stock standard (1,000 µg/mL):

Dissolve 1.5000 g NaNO₂ and dilute to the mark in a 1-L volumetric flask with DI H₂O. Prepare every 3 months.

6.3.7 Nitrite standard (100 µg/mL):

Dilute 10 mL of the 1,000 µg/mL nitrite stock standard to 100 mL with liquid desorber. Prepare monthly.

6.3.8 Nitrite standard (10 µg/mL):

Dilute 10 mL of the 100 µg/mL nitrite stock standard to 100 mL with liquid desorber. Prepare weekly.

6.3.9 Nitrite standard (1 µg/mL):

Dilute 10 mL of the 10 µg/mL nitrite stock standard to 100 mL with liquid desorber. Prepare daily.

6.4 Working Standard Preparation

6.4.1 Nitrite working standards (10-mL final volumes) may be prepared in the ranges specified below:

| Working Std µg/mL | Standard Solution, µg/mL | Aliquot mL |
|----------------------|-----------------------------|---------------|
| 0.5 | 1 | 5 |
| 1 | 1 | * |
| 3 | 10 | 3 |
| 6 | 10 | 6 |
| 10 | 10 | * |
| 20 | 100 | 2 |

* Already prepared in Section 6.3

- 6.4.2 Pipette appropriate aliquots of standard solutions (prepared in Section 6.3) into 10-mL volumetric flasks and dilute to volume with liquid desorber.
- 6.4.3 Pipette a 0.5- to 0.6-mL portion of each standard solution into separate automatic sampler vials. Place a 0.5-mL filter cap into each vial. The large exposed filter portion of the cap should face the standard solution.
- 6.4.4 Prepare a reagent blank from the liquid desorber solution.

6.5 Sample Preparation

Note: For NO sample analysis and result calculations, see reference 8.10.

- 6.5.1 Clean the 25-mL Erlenmeyer flasks or scintillation vials by rinsing with DI H₂O.
- 6.5.2 Carefully remove the glass wool plugs from the sample tubes, making sure that no sorbent is lost in the process. If the two-section tube was used for sampling, transfer each TEA-IMS section to individual 25-mL Erlenmeyer flasks or scintillation vials. Analyze these two sections separately. If a single section tube was used, transfer that section to an individual 25-mL Erlenmeyer flask or scintillation vial.
- 6.5.3 Add 3 mL of liquid desorber to each flask or vial, shake vigorously for about 30 s and allow the solution to settle for at least 1 h.
- 6.5.4 If the sample solutions contain suspended particulate, remove the particles using a pre-filter and syringe. Fill the 0.5-mL automatic sampler vials with sample solutions and push a 0.5-mL filtercap into each vial. Label each vial.
- 6.5.5 Load the automatic sampler with labeled samples, standards and blanks.

6.6 Analytical Procedure

Set up the ion chromatograph and analyze the samples in accordance with the SOP (8.15). Typical operating conditions for equipment mentioned in Section 6.2 are listed below.

Ion chromatograph

Eluent: 2.0 mM Na₂CO₃/1.0 mM NaHCO₃
 Column temperature: ambient
 Sample injection loop: 50 µL

Pump

Pump pressure: approximately 1,000 psi
 Flow rate: 2 mL/min

Chromatogram

Run time: 6 min
 Average retention time: approximately 2 min

7. Calculations

- 7.1 Obtain hard copies of chromatograms from a printer. A typical chromatogram is shown in Figure 1.
- 7.2 Prepare a concentration-response curve by plotting the concentration of the standards in µg/mL (or µg/sample if the same solution volumes are used for samples and standards) versus peak areas or peak heights.

- 7.3 Blank correct the samples by subtracting the $\mu\text{g}/\text{mL NO}_2^-$ found in the blank from the $\mu\text{g}/\text{mL NO}_2^-$ found in the samples. If a different solution volume was used for blanks and samples, use total micrograms NO_2^- to blank correct.
- 7.4 Calculate the concentration of nitrogen dioxide in each air sample in ppm. A concentration-dependent conversion factor is used. The equation is:

$$\text{ppm NO}_2 = \frac{\text{Molar volume} \times \mu\text{g} / \text{mL NO}_2^- \times \text{Solution volume} \times \text{Conversion}}{\text{Formula weight} \times \text{Air volume}}$$

Where:

Molar volume = 24.45 (25 °C and 760 mmHg)

$\mu\text{g}/\text{mL NO}_2^-$ = blank corrected sample result

Formula weight (NO_2) = 46.01

Conversion = varies with concentration

The conversion of gaseous NO_2 to NO_2^- is concentration-dependent and should be calculated using one of the equations given below:

Below 10 ppm NO_2

From 0 to 10 ppm, the average relationship has been experimentally determined to be (8.1-8.4, 8.6-8.9):

$$1 \mu\text{g NO}_2 \text{ (gas)} = 0.63 \mu\text{g NO}_2^-$$

or conversely:

$$1 \mu\text{g NO}_2^- = 1.587 \mu\text{g NO}_2 \text{ (gas)}$$

Simplifying the equation and using a 3-mL sample volume gives:

$$\text{ppm nitrogen dioxide} = \frac{\mu\text{g} / \text{mL NO}_2^- \times 3 \text{ mL} \times 0.843}{\text{Air volume (L)}}$$

Above 10 ppm NO_2

Above 10 ppm NO_2 , the expected stoichiometric factor of 0.5 mole of nitrite to 1 mole of nitrogen dioxide gas is seen (8.6, 8.8-8.9). Therefore, the following calculation should be used for sample results above 10 ppm and a 3-mL sample volume:

$$\text{ppm nitrogen dioxide} = \frac{\mu\text{g} / \text{mL NO}_2^- \times 3 \text{ mL} \times 1.0633}{\text{Air volume (L)}}$$

7.5 Reporting Results

Report all results to the industrial hygienist as ppm nitrogen dioxide.

8. References

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- 8.14 American Conference of Governmental Industrial Hygienists: Documentation of the Threshold Limit Values and Biological Exposure Indices. 5th ed. Cincinnati, OH: ACGIH, 1986. pp. 435-436.
- 8.15 Occupational Safety and Health Administration Technical Center: Standard Operating Procedure-Ion Chromatography. Salt Lake City, UT. In progress (unpublished).

Chromatogram of a 10 µg/mL Nitrate Standard in 1.5% TEA Solution

| Peak Num | Ret Time | Peak Name | Area |
|----------|----------|-----------|------------|
| 1 | 0.90 | | 1.778e+004 |
| 2 | 1.15 | | 1.965e+004 |
| 3 | 1.50 | | 2.214e+004 |
| 4 | 1.80 | chloride | 3.476e+003 |
| 5 | 2.13 | nitrite | 8.886e+004 |
| 6 | 4.18 | | 3.600e+003 |

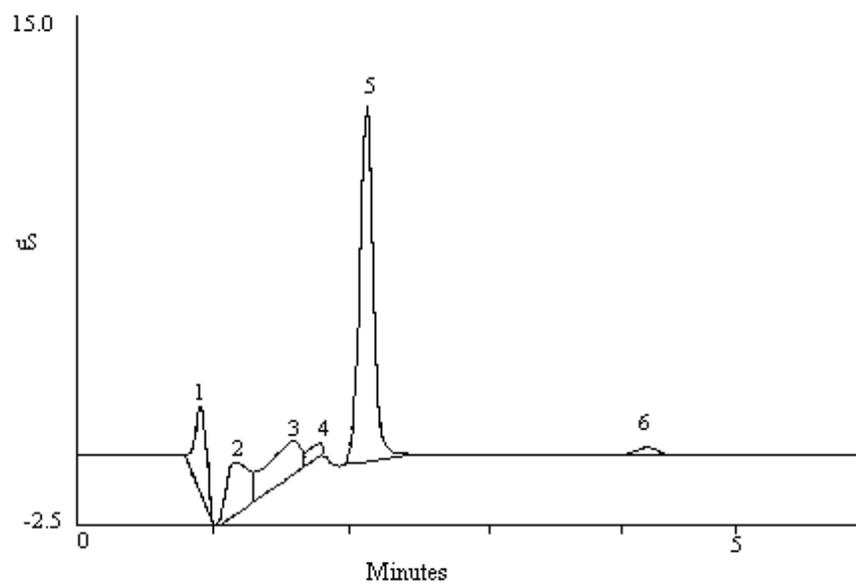


Figure 1