

DETERMINATION OF NITRITE IN ENGINE COOLANT BY ION CHROMATOGRAPHY

Application

This method is used to determine the concentration of sodium nitrite in automotive antifreeze formulations which also contain sodium benzoate.

Principle

This method is based on the technique of ion chromatography. The separation of nitrite and benzoate anions in the antifreeze is accomplished by the use of an ionic eluent which is pumped through an ion exchange column and a suppressor column. The anions are separated in the ion exchange separator column based on their affinity for the column resin. After separation, the eluent containing the sample anions passes through a membrane suppressor which simultaneously neutralizes the ionic species in the eluent and converts the sample anions to their highly conductive acid form. The nitrite anion is then detected by means of a conductivity detector. Identification and quantitation of nitrite is accomplished by an external standard technique.

Equipment Required

1. Ion Chromatograph - Dionex model DX120, or equivalent, with conductivity detector.
2. Anion Guard/Separator Column - Dionex part # AS-4 Guard, AS-4 Separator or equivalent
3. Anion Suppressor Column - Dionex micromembrane suppressor part # 38019 or equivalent
4. Integrator - Hewlett Packard model 3396C or equivalent.
5. Analytical Balance - + 0.1 mg sensitivity, Mettler model AE160 or equivalent.
6. Pyrex 100 mL Volumetric Flasks - CMS No. 106-377 or equivalent.
7. Pyrex Volumetric Transfer Pipets - (Various capacities, 10, 20 mL), CMS No. 250-816 or equivalent, Class A.
8. Kimax 4000 mL Graduated Beaker - CMS 029-637 or equivalent.
9. pH Meter - Orion model 520A or equivalent with Ross combination pH electrode and temperature compensation probe.

Date	Action	Revisions
2001 01 30	Revised	Editorial - no technical change A. Cockman
2000 04 17	Activated	A. Reaume

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Reagents Required

1. **Water** – deionized - nanopure reagent grade - conductivity <5mohms.
2. **Benzoic Acid (C₆H₅COOH)** - reagent grade.
3. **Sodium Carbonate (Na₂CO₃)** - reagent grade.
4. **Sodium Nitrite (NaNO₂)** - Certified ACS reagent grade (99 + %) or purchased - traceable 1000ppm NO₂ standard solution.
5. **Citric Acid (2-hydroxyl-1,1,2,3-propanetricarboxylic acid)** - reagent grade.
6. **Sulfuric Acid (1.0 N)** - Certified, analytical grade titrant.

Eluent Preparation

The eluent used for this method consists of 0.001 M sodium carbonate and 0.001 M benzoic acid. The procedure for preparation of four liters of eluent is as follows:

1. On an analytical balance, accurately weigh 0.488 grams of benzoic acid and 0.424 grams of sodium carbonate.
2. Add both of the above to a clean four-liter glass beaker.
3. Add deionized water to make up four liters of solution and stir until components are dissolved.

Preparation of the Standards

A 1000 ppm stock solution of nitrite (NO₂⁻) should be prepared as follows:

1. On an analytical balance, accurately weigh 1.4998 grams of sodium nitrite and add to a clean one liter volumetric flask.
2. Dissolve the standard and dilute to the mark with deionized water. Store the stock solutions in one liter plastic containers in the refrigerator with the preparation date labeled.
3. Record all weights, date, and name of technician in the standards log book.
4. A working standard of 20 ppm nitrite is prepared by pipetting 20 mLs of 1000 ppm nitrite stock solution into a clean one liter volumetric flask and diluting to the mark with deionized water. Store the stock solution and working standard in the refrigerator until needed. The working standard should be stable for one month. As the working standard is depleted, make up fresh solution using the stock solution. The stock solution should be stable for three months.