

DETERMINATION OF BENZOATE AND TRIAZOLE IN COOLANT BY LIQUID CHROMATOGRAPHY

Application

This method is used to determine the concentrations of sodium benzoate and benzotriazole corrosion inhibitors in formulated automotive coolant. The method is based on the technique of high pressure liquid chromatography. The separation of the corrosion inhibitors is accomplished using a reverse phase C-18 column and an isocratic mobile phase consisting of a volume to volume mixture of 50% sodium acetate buffer/50% methanol at a pressure of 1000 to 2000 psig. The compounds are detected by means of their UV absorbance at 254 nm and quantitation is accomplished by use of external standards.

Equipment Required

1. Hewlett-Packard 1100 Series Liquid Chromatograph or equivalent.
2. Hewlett-Packard 3396C Integrator or equivalent.
3. Phenomenex Selectosil reverse phase C-18 column or equivalent.
4. Analytical balance, Mettler Model AE 160 or equivalent.
5. pH meter, Brinkmann Metrohm Model 744 with combination electrode or equivalent.
6. Vacuum system
7. Gelman # 4200 47 mm magnetic filtration funnel - 300 mL plastic with stainless steel coupling ring and support screen.
8. Kimax 2000 mL filtering flask
9. Metrical 47 mm, .45 µm membrane filters
10. Syringe Filters - 0.45 µm.
11. Kimax 4000 mL graduated beaker
12. Pyrex 1000 mL graduated

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** - HPLC grade
2. **Methanol** - HPLC grade
3. **Sodium Acetate Trihydrate** - HPLC grade
4. **Glacial Acetic Acid** - reagent grade
5. **Sodium Benzoate** - analytical grade, 99%
6. **Benzotriazole** - analytical grade, 99+%
7. **Sodium Hydroxide Solution** - 10-15%

Preparation of the Mobil Phase

The glassware and filtration equipment used in preparing the mobile phase should be very clean. The mobile phase used in this method consists of two parts:

1. **Solvent A** = 0.08 M Sodium Acetate buffer solution (50%)
2. **Solvent B** = HPLC grade methanol (50%)

A. Solvent A Preparation (Sodium Acetate Buffer Solution)

1. Add 44 grams of HPLC grade sodium acetate trihydrate to a lean four-liter glass beaker. Add HPLC (or nanopure) grade water to make up four liters of solution. Stir until sodium acetate is dissolved.
2. While stirring, adjust the solution pH to 6.0 using a pH meter and drop wise addition of reagent grade glacial acetic acid.
3. Filter the completed solvent A through a 0.45 micron membrane filter using the Gelman filtration apparatus. Fill the one liter reservoir marked Solvent A and store the remaining mobile phase in a clean, labeled four liter glass container. Keep refrigerated.

B. Solvent B Preparation (HPLC grade methanol)

1. No preparation required. Fill the one liter reservoir marked Solvent B.