

NAVISTAR, INC.		
CEMS (CORPORATE ENGINEERING MATERIAL SPECIFICATION)		NUMBER: CEMS BT-43
TITLE: Barium, Calcium, Magnesium and Zinc In Oil		CURRENT ISSUE DATE: August 2009
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<i>This specification may involve hazardous materials, equipment, and procedures. This specification does not purport to address all of the safety issues associated with its use. The user is responsible to consult appropriate safety and health practices and to determine the applicability of regulatory limits prior to use.</i>		
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1.0 SCOPE

This method covers a rapid and accurate procedure for determining the amounts of barium, calcium, magnesium and zinc in unused lubricating oils.

2.0 APPARATUS

- 2.1 Magnetic stirrer.
- 2.2 100 ml porcelain evaporating dish.
- 2.3 Hot plate or bath capable of maintaining solutions 70 to 80°C.
- 2.4 Muffle furnace capable of maintaining $700 \pm 25^\circ\text{C}$.
- 2.5 250 ml beaker.
- 2.6 Funnel.
- 2.7 Titration burette.
- 2.8 Analytical balance.
- 2.9 Desiccator.

3.0 MATERIAL

- 3.1 Sulfuric Acid solution, 1:1 (H_2SO_4)
- 3.2 Disodium Ethylenediaminetetra-acetate, dihydrate, reagent grade.
- 3.3 Magnesium Sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), reagent grade.
- 3.4 Eriochrome Black T, reagent grade.
- 3.5 Triethanolamine, reagent grade.
- 3.6 Ethyl alcohol.
- 3.7 Ammonium Chloride (NH_4Cl), reagent grade.
- 3.8 Ammonium Hydroxide (NH_4OH), reagent grade.
- 3.9 Methyl Red, reagent grade.
- 3.10 Ammonium Chloride, 20% solution (NH_4Cl).
- 3.11 Ammonium Hydroxide solution, 5:95 (NH_4OH).
- 3.12 Ammonium Oxalate, reagent grade [$(\text{NH}_4)_2\text{C}_2\text{O}_4$].
- 3.13 Ammonium Phosphate, Dibasic, 20% solution [$(\text{NH}_4)_2\text{HPO}_4$]
- 3.14 Potassium Cyanide, 20% solution (KCN)
- 3.15 Formaldehyde, 4% solution (HCHO).
- 3.16 Hydrochloric Acid solution, 1:50 (HCl).
- 3.17 Hydrochloric Acid solution, 1:1 (HCl).

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- 3.18 Sodium Hydroxide, 30% (NaOH).
- 3.19 Distilled water.
- 3.20 ph papers, ranges 1 to 6 and 6 to 12.
- 3.21 Filter paper, medium porosity, ashless.

4.0 SOLUTION PREPARATION

4.1 EDTA - 0.01 Molar

Dissolve 3.723 g of disodium ethylenediaminetetra-acetate, dihydrate, in distilled water and dilute to 1 liter.

4.2 Magnesium Sulfate - 0.01 Molar

Dissolve 2.465 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in distilled water and dilute to 1 liter. Standardize against EDTA solution.

4.3 Eriochrome Black T Indicator

Dissolve 0.2 g Eriochrome Black T in 5 ml of ethyl alcohol and mix with 15 ml of triethanolamine.

4.4 Methyl Red Indicator

Dissolve 0.25 g Methyl Red in 15 ml of ethyl alcohol. Dilute to 250 ml with distilled water.

4.5 Buffer Solution - pH 10

Dissolve 35 g NH_4Cl in 150 ml distilled water. Add 285 ml concentrated NH_4OH and dilute to 500 ml with distilled water.

5.0 PROCEDURE

BARIUM

5.1 Approximately 10 g of sample, weighed to the nearest mg, is placed in a 100 ml porcelain evaporating dish.

5.1.1 Burn off organic matter on a hot plate and then in a muffle furnace at 600°C for approximately 10 minutes.

5.1.2 After it has cooled, wet it completely with H_2SO_4 1:1 and heat on hot plate until all the H_2SO_4 fumes are expelled.

5.1.3 Place in muffle furnace maintained at 700°C for a period required to burn off all the carbon.

5.1.4 Cool, add 20 ml HCl 1:1 and evaporate to moist dryness--avoid spattering.

5.1.5 Transfer residue to 250 ml beaker using hot HCl 1:50. Final volume of solution should not exceed 100 ml.

5.1.6 If a precipitate forms in this dilute HCl solution, add 2 ml of H_2SO_4 1:1 and boil for a few minutes. Let stand on hot plate (70 to 80°C) until completely settled out. If no precipitate forms, barium is not present. Proceed directly to Step 5.2.

5.1.7 To separate the barium sulfate precipitate, filter through a medium porosity, ashless filter paper and wash with hot water adding the wash water to the filtrate. Final volume of the solution should not exceed 150 ml.

5.1.8 Place the filter paper containing the barium sulfate precipitate in a tared porcelain crucible, dry on a hot plate and burn off in a muffle furnace at 700°C.

5.1.9 Cool the crucible in a desiccator and weigh to the nearest mg.

5.1.10 Calculation = $\text{g BaSO}_4 = [\text{weight of crucible and residue}] - [\text{weight of crucible (tare)}]$

$$\% \text{ Ba} = \frac{\text{g BaSO}_4 \times 0.5884 \times 100}{\text{Sample weight}}$$

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