Test Procedure SA-011
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Total Heavy Metals

Applicable Products: Carbopol®* Polymers and Pemulen™* Polymeric Emulsifiers

Scope:

This procedure describes a method to determine that the total heavy metals precipitated as sulfide ions from a sample of Carbopol® polymers or Pemulen™ polymeric emulsifiers do not exceed specifications.

Abstract:

A sample of the product is charred, ignited in a muffle furnace to remove all carbon, and digested. A standard preparation containing the specification limit as lead is prepared. The heavy metals in both the sample and standard are precipitated as sulfides. If the color formed for the sample is lighter than the color formation of the standard, the heavy metal content for the material tested is less than the specification limit.

Safety Precautions:

1. Wear safety goggles and gloves and follow good laboratory practices.
2. Polymer dust is irritating to the respiratory passages and inhalation should be avoided.
3. Three different concentrated acids are required in the digestion step. Extreme care should be exercised when handling nitric, sulfuric and hydrochloric acids. Protective clothing, including apron, chemical resistant gloves and goggles should be worn.
4. See all Material Safety Data Sheets (MSDS) for additional safety and handling information.

Interferences:

The result of the test is based on a comparison of color formation of the sample preparation and a standard preparation. Anything that would produce or suppress color in either preparation could influence the result. No interferences are recognized.

This method does not recover mercury.

Apparatus:

1. Volumetric flask, 1000 mL.
2. Volumetric flask, 100 mL.
3. Pipette, 10 mL.
4. Pipette, 5 mL.
5. Pipette, 1 mL.
6. Pipette bulb.
7. pH meter with glass-calomel electrode.
8. 50 mL color comparison tubes.
9. Crucible, 50 to 100 mL capacity, of suitable material.
10. Crucible cover.
11. Bunsen burner or hot plate.
12. Steam bath.
13. Ring stand and 3” ring to support bath.
14. Clay triangle.
15. Litmus paper.
16. pH indicator paper, narrow range (color change between pH 3.0 and 4.0).
17. Funnel, 3” glass, porcelain or plastic.
18. Ashless filter paper.

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19. Analytical balance capable of ±0.001 g accuracy.
20. Liquid dropper.
21. Muffle furnace, capable of 500°C (see Note 2).
22. Desiccator with silica gel desiccant.
23. Boiling water bath.
24. Graduated cylinder, 100 mL.

Reagents:

1. Deionized water.
2. Lead nitrate.
3. Nitric acid.
4. pH 3.5 acetate buffer (see Special Instruction 1).
5. 6 N hydrochloric acid (see Special Instruction 2).
6. 6 N ammonium hydroxide (see Special Instruction 3).
7. 1 N acetic acid (see Special Instruction 4).
8. Concentrated sulfuric acid.
9. Thioacetamide glycerin base TS (see Special Instruction 5).
10. Hydrochloric acid.

Procedure:

STANDARD PREPARATION:

Lead Nitrate Stock Solution

1. To a 1000 mL volumetric flask, add 100 mL deionized water.
2. Add 1 mL nitric acid.
3. Weigh 159.8 mg of lead nitrate and dissolve in the water.
4. Dilute to the mark with deionized water.
5. Store this primary standard (100 µg Pb/mL) in a glass container.

Standard Lead Solution:

The standard lead solution is to be prepared on the day of use.

1. To a 100 mL volumetric flask pipette 10 mL of the lead nitrate stock solution (10 µg Pb/mL).
2. Dilute to the mark with deionized water.

Standard Preparation:

1. To a 50 mL color comparison tube, pipette 2 mL of Standard Lead Solution (20 µg Pb).
2. Dilute with water to 25 mL.
3. Using a pH meter, adjust with 1 N acetic acid (see Special Instruction 4) or 6 N ammonium hydroxide (see Special Instruction 3) to a pH between 3.0 and 4.0.
4. Dilulte with water to 40 mL and mix.

SAMPLE PREPARATION

1. Dry a crucible and loosely fitting cover at 100°C for at least 30 minutes. Allow to cool in a desiccator.
2. Transfer 1.0 ± 0.01 g, accurately weighed, of the product to be tested to the crucible.
3. Using a liquid dropper, add sufficient sulfuric acid to wet the powder.
4. Supporting the crucible on a clay triangle and ring, use a Bunsen burner (see Note 1) to carefully ignite at a low temperature until thoroughly charred. (The crucible may be loosely covered with a suitable cover during the charring).
5. Add 2 ml nitric acid and 5 drops sulfuric acid to the charred powder.
6. Heat cautiously until white flames no longer emit.
7. Ignite in a muffle furnace at 500 to 600°C until the carbon is completely burned off.
8. Allow to cool in a desiccator.
9. Add 4 mL of 6 N hydrochloric acid.
10. Cover the crucible and digest on a steam bath for 15 minutes.
11. Uncover and slowly evaporate to dryness on a steam bath.
12. Moisten the residue with 1 drop hydrochloric acid and add 10 ml hot water.
13. Digest for 2 minutes.

14. Add 6 N ammonium hydroxide dropwise until the solution is just alkaline to litmus paper.
15. Dilute with water to 25 mL and adjust with 1 N acetic acid to a pH between 3.0 and 4.0 using narrow-range pH indicator paper.
16. Filter, if necessary, rinse the crucible and the filter with 10 mL water.
17. Combine the filtrate and rinsing in a 50 mL color comparison tube.
18. Dilute with water to 40 mL and mix.

PROCEDURE:

1. To each of the tubes containing the standard preparation and the sample preparation, add 2 mL of pH 3.5 acetate buffer.
2. Add 1.2 mL of thioacetamide-glycerin base TS.
3. Dilute with water to 50 mL and mix thoroughly. Allow to stand for 2 minutes.
4. In a well lighted area, view the tubes from the top over a white surface. If the color of the test preparation is not darker than the solution from the standard preparation, the total heavy metal content of the product being tested is less than the specification limit of 20 ppm. The result of the test is recorded as “pass”.

CALCULATIONS:

There are no calculations. The result of the test is recorded as “pass” or “fail.”

NOTES:

1. A hot plate capable of reaching the temperature to char the sample could be used in place of a Bunsen burner.
2. The procedure specifies igniting at 500 to 600°C.

SPECIAL INSTRUCTIONS:

Thioacetamide-glycerin base TS must be prepared fresh. All other reagents prepared for this procedure are stable and can be stored.

1. pH 3.5 acetate buffer. Dissolve 25.0 g of ammonium acetate in 25 mL water in a 100 mL volumetric flask. Add 38 mL 6 N hydrochloric acid. Adjust, if necessary, with 6 N ammonium hydroxide or 6 N hydrochloric acid to a pH of 3.5. Dilute with deionized water to 100 mL and mix. Transfer to a reagent bottle.
2. Hydrochloric acid, 6 N. Add 510 mL concentrated hydrochloric acid to 450 mL deionized water in a 1000 mL volumetric flask. Mix and dilute to the mark with deionized water. Mix again and transfer to a reagent bottle.
3. Ammonium hydroxide, 6 N. Add 400 mL concentrated ammonium hydroxide to 550 mL distilled water in a 1000 mL volumetric flask. Mix and dilute to the mark with water. Mix thoroughly and transfer to a reagent bottle.
4. Acetic acid, 1 N. Add 60 mL glacial acetic acid to a 1000 mL volumetric flask partially filled with deionized water. Mix and dilute to the mark with deionized water. Mix thoroughly and transfer to a reagent bottle.
5. Thioacetamide-glycerin base TS. Mix 0.2 mL thioacetamide TS and 1 mL glycerin base TS. Heat in a water bath for 20 seconds. Use immediately. (see Special Instructions 6 and 7).
6. Thioacetamide TS. Dissolve 4 g thioacetamide in 100 mL deionized water.
7. Glycerin base TS. To 200 g glycerin add deionized water to bring the total weight to 235 g. Add 140 mL of 1 N sodium hydroxide and 50 mL deionized water.

REFERENCES:

- *Current edition of the United States Pharmacopeia/National Formulary (USP/NF), <231> Method II.*