TENNECO CHEMICALS, INC.

TENNECO INTERMEDIATES DIVISION

T.A.M. 4-3

Specific Use: Burlington Compound Plant

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DETERMINATION OF LEAD CONTENT OF DIBASIC LEAD STEARATE, TRIBASIC LEAD SULFATE AND COMPOUNDS CONTAINING THESE STABILIZERS

APPLICATION

This is a gravimetric assay technique for the determination of lead content in dibasic lead stearate, tribasic lead sulfate and compound dry blends containing these stabilizers.

The method is based on the digestion of a sample in 1:1 HNO_3/H_2SO_4 followed by oxidation with H_2O_2, driving off the HNO_3 and precipitating the lead as the sulfate.

APPARATUS

1. Beakers, graduated, 400 ml
2. Hot plate
3. Crucibles, gooch, porcelain (Ace Scientific, Cat. #11-6770, EDP-07)
4. Asbestos, long fiber (acid-washed)(Matheson, Cat. #AX1760)
5. Glass beads
6. Flask, filter, 500 ml
7. Wash bottles, polyethylene
8. Speedyvap watch glasses
9. Analytical balance with readout to 0.1 mg
10. Furnace, muffle
11. Desiccator
12. Glass stirring rod
13. Thongs
14. Vacuum pump
15. Rubber gloves

REAGENTS

1. Sulfuric acid, reagent grade
2. Nitric acid, reagent grade
3. 10% sulfuric acid solution (carefully transfer 100 ml of H_2SO_4 into 400 ml of water - cool and make up to 1,000 ml, mix well)
4. Isopropyl alcohol, reagent grade
5. Demineralized water
6. 30% hydrogen peroxide solution

PROCEDURE

1. Weigh 0.5 - 0.8 gm duplicate samples in 400 ml beakers and record to the nearest 0.1 mg (for compounds 2.0 gm).
2. Add 50 ml of HNO_3 and 25 ml of H_2SO_4 and a few glass beads. Place watch glasses on top of the beakers.
3. Boil the solution until the solution stops boiling (Note 1.).
4. Remove beakers from the hot plate. Add 30% H_2O_2 from a wash bottle until the oxidation of HNO_3 is complete (Note 2.).
5. Add enough demineralized water to bring the volume of the solution to 125 ml and boil for 15 minutes (Note 3.).
6. Remove from heat, cool and add 50 ml of isopropyl alcohol from a wash bottle. Refrigerate the solution.
7. After cooling, filter the solution through a previously-tared gooch crucible fitted with asbestos filters. Wash the filtrate with a 10% H_2SO_4 solution.
8. Place the crucibles on a hot plate (80°C, covered with an asbestos plate) for one hour.
9. Place the crucibles in a desiccator and allow to cool.
10. Re-weigh the crucibles, determine the precipitate weight and calculate the lead content.

CALCULATION

$$\% \text{ Pb} = \frac{A \times 0.683}{B} \times 100$$

A = Precipitate Weight (gm)

B = Sample Weight (gm)

NOTE

1. When solution begins to boil, it will emit nitric oxide fumes. These fumes are brownish in color. At a certain point in the boiling, the nitric oxide will stop being emitted and the solution will stop boiling. When analyzing compounds, make sure sample is completely digested by adding small increments of HNO_3.
2. Be sure to add only small amounts of the 30% H_2O_2 at a time from a wash bottle. The oxidation of HNO_3 is very violent. Heat generated from this reaction will cause the mixture to boil over and a loss of the precipitate will result.
3. Swirl beaker by hand once in a while, until the mixture begins to boil.

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