

KES

Method for Metal Component of
Rubber Product by Ash Method

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Additional Rule



1. Scope of Application

This standard shall specify a quantitative analysis method of metal component from the rubber product by ash method.

2. Silicon dioxide (SiO₂) and Insoluble component

- (1) Dissolve ash specimen in 10cc of hydrochloric acid, dilute with 100cc of distilled water, and evaporate and dry.
- (2) Fry at 110 °C for 1 hour.
- (3) Wet with 100cc of the hydrochloric acid, drop 3 drops of nitric acid, and assimilate in steam bath for 15 minutes.
- (4) Add 100 ml of the distilled water, filter out, and wash out with hot water.
- (5) Dry the settlements in chinaware crucible and make to ash.
- (6) Keep the remaining solution and use for an analysis of lead dioxide (PbO₂).
- (7) Measure weight of silicon dioxides (SiO₂) and insoluble components.
- (8) Move ash components to platinum crucible, add 2 ~ 3 ml of HF, add 2 ~ 3 drops of sulfuric acid, dry, and make to ash carefully.

Amount disappeared here is SiO₂.

(9) Calculation

$$\text{SiO}_2 + \text{Insoluble component (\%)} = [(A - B) / C] \times 100$$

Here, A : Weight of crucible and ash

B : Weight of crucible

C : Weight of specimen

D : Weight of crucible and ash after treating with hydrofluoric acid

E : Weight of crucible

3. Lead dioxide (PbO₂)

3.1 Reagent

- (1) Ethyl alcohol : 95 % and 50 %
- (2) Hydrogen sulfide (H_2S)

3.2 Method

- (1) Add small amount of ammonium hydroxide to the remaining solution produced in 2. (6), and add 1 ml of hydrochloric acid.
- (2) Pass hydrogen sulfide gas until settlements are created completely.
- (3) Filter out the settlements, wash out with distilled water, which contains hydrogen sulfide.
At this time, keep the remaining solution to analyze aluminum or iron oxidized material.
- (4) Dissolve the settlements in nitric acid (1 + 1), heat up and filter out.
- (5) Cool down the remaining solution, add 10 ml of sulfuric acid, and evaporate and dry.
- (6) Cool down, add 50 ml of water, dilute, add the same amount of ethyl alcohol, and leave for one night.
- (7) Filter out with Gooch crucible, wash out with ethanol, and dry at 105 °C.
- (8) Calculation

$$\text{Lead dioxide (PbO}_2\text{)} = [(A - B) \times 0.736 / C] \times 100$$

Here, A : Weight of Gooch crucible and lead sulfate (gr)

B : Weight of Gooch crucible (gr)

C : Weight of specimen

0.736 : Conversion coefficient

4. Iron and aluminum oxidized material (R_2O_3)

4.1 Reagent

- (1) Ammonium chloride (Solid state)
- (2) Ammonium chloride solution (20gr NH_4Cl / dm^3)

- (3) Methyl red indicator solution
- (4) Potassium Ferricyanide indicator
- (5) Potassium Pyrosulfate ($K_2S_2O_7$)

4.2 Method

- (1) Boil the remaining solution produced in item 3.2. (3)
- (2) Make volume of the solution to be 100 ~ 150 ml, add 3 ~ 4 drops of the nitric acid and boil this solution again.
- (3) Using $K_3Fe(CN)_6$ outer indicator solution, confirm an existence of ferrous iron, and, if it does, add more nitric acid.
- (4) Add ammonium hydroxide solution until color of 5 gr of NH_4Cl and the solution become yellow by the methyl red indicator.

However, it is not allowed to just heat up as it is.
- (5) Heat up until it boils, and heat 5 more minutes.
- (6) Filter out if the settlements are produced, and wash out with the ammonium hydroxide solution.
- (7) Use the remaining solution to analyze calcium oxide, and oxidize the settlements along with the filtered out components at low temperature.

4.3 Calculation

$$R_2O_3 (\%) = [(A - B) / C] \times 100$$

Here, A : Weight of crucible and ($Fe_2O_3 + Al_2O_3$) (gr)

B : Weight of crucible (gr)

C : Weight of specimen (gr)

5. Calcium oxide

5.1 Reagent

- (1) Ammonium oxalate saturation solution