

KES

**Analysis of Inorganic Material
in Poly Vinyl Chloride Resin**

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1. Scope of application

This standard shall be prescribed to the method of quantitative analysis of chlorine and component element in poly vinyl chloride(It is called as PVC below)

2. Chlorine

Analyze the chlorine in PVC by neutralizing titration with thiocyanic acid ammonium solution.

2.1 Titration of thiocyanic acid ammonium solution

2.1.1 Summary

This method is prescribed to method of chlorine analyzing in vinyl chloride resin varnish, vinyl chloride resin enamel and vinyl chloride resin primer.

2.1.2 Regent

- (1) Acetone
- (2) Benzene
- (3) Ligroin
- (4) Oil ether
- (5) Potassium oxide : Special grade
- (6) Potassium hydroxide
- (7) Ethly ether
- (8) Nitric acid : Special grade
- (9) Silver nitrate solution : 0.1 N
- (10) Ferric sulfate ammonium solution : Dissolve 20 g of ferric sulfate ammonium (Special grade) in 80 ml of water and put 20 ml of sulfuric acid(Special grade) solution (1 : 3) into it.
- (11) Thiocyanic acid ammonium solution(0.1 N) : Solute about 8 g of thiocyanic acid ammonium(Special grade) in water, dilute it with water and make it 1 l.

2.1.3 Extraction of resin part.

- (1) Measure the quantity about 5 g of sample exactly, put it into about 60 ml of centrifugal precipitation tube and mix it well with putting 50 ml of acetone. And cover with a stopper, separate the solution with a centrifugal machine and separate a solid from it. At this moment, set the rounds per minute of the centrifugal machine for 5000 ~ 6000 rpm and the time of rounds for 30 ~ 40 minutes.
- (2) Move the upper fluid in an evaporating dish and concentrate it about 50 ml with heating it in boiling water. After that, move it into a different precipitation tube. After precipitating the resin part with putting 40 ml of benzene(1st grade) in it little by little, cover the precipitation tube with a stopper and separate the resin part from it in the centrifugal machine.
- (3) Remove the upper fluid, put acetone and benzene mixture solution(1 : 1) in it and dissolve the resin part with heat it at 40 ~ 50 °C with stirring it. Precipitate and separate the resin part with the same previous method, putting the ligroin. And remove the upper fluid and repeat dissolving, precipitating, removing once more.
- (4) Wash the resin part with petroleum ether twice, move it in glass dish which has large bottom and break it up into fine soil. And dry it about 2 hours at 80 °C in an oven. After cooling in a desiccator, use it for fixing quantity of chlorine.

2.1.4 Fixing quantity of chlorine

- (1) measure quantity about 0.2 g of the separated resin part exactly, spread it in a 40 ~ 60 ml nickel crucible scattered with about 2 g of potassium oxide, cover the resin part with about 5 g of potassium hydroxide and wet the contents with about 1 cc of ethyl alcohol.
- (2) Make the potassium hydroxide a liquid state with heating the crucible gradually in the heater and boil the contents strongly, preventing the overflowing and breaking of the