

KES

Analysis of Phosphorous in Lubricating Oil and Additives

KES T – M033

(EST.) 1988 – 06 – 13

Contents

1. Scope of Application
2. Test Method
3. Oxidation of Sample
4. Photometric Method
5. Gravimetric Method
6. Quinoline Method



1. Scope of application

This standard shall specify quantitative analysis method of an addition that is contained in new oil or used oil or phosphorous that is contained in worn powder.

2. Test method

- (1) Photometric method
- (2) Gravimetric method : More than 2% of phosphors content by amount
- (3) Quinoline method : 0.005~10% of phosphors content by amount

3. Oxidation of sample

3.1 Summary

This operation is applied to operation of all treatment when photometric method and gravimetric method is applied.

In addition, this operation is to remove organic materials in a sample and to change phosphors to phosphate ion.

3.2 Method summary

After dissolving organic metallic chemical materials in a sample and changing phosphors to phosphate ion by oxidation with sulfuric acid, nitric acid and hydrogen peroxide.

3.3 Equipment

- (1) Separatory flask: use the one of capacity 300ml as a Kjeldahl flask.
- (2) Kjeldahl flask holder

3.4 Reagent

- (1) Hydrogen peroxide: use the one that phosphors content by amount is less than 0.0002% and concentration is 3%.
- (2) Nitric acid
- (3) Sulfuric acid

3.5 Operation

- (1) After extracting sample by following method, put it in 300 ml Kjeldahl flask. Be careful not to be touched with around neck of flask for a sample.

Extracted sample amount

Photometric method : About 2 g when phosphor content by amount is 0.002 ~ 0.2%

About 0.2 g when phosphor content by amount is 0.2 ~ 2%

About 1 g when phosphor content by amount is 5 ~ 10%

About 0.7 g when phosphor content by amount is 10 ~ 15%

About 0.4 g when phosphor content by amount is 15 ~ 25%

After adding sulfuric acid into a flask and putting a glass bead of diameter 6mm in it, mix them by stirring a mixture in the flask.

At this moment, add sulfuric acid of 3 ml for photometric method and 10 ml for gravimetric method.

- (2) In case of photometric method, use white oil not contained phosphors as test oil by the same method.

- (3) Connect a flask to a holder and heat a sample slowly using a micro-burner in a hood.

At this moment, use air stream as a heater if it exists.

Heat it continuously until white smoke is made and oxidize organic material by adding nitric acid 1ml during boiling.

If white smoke is generated without boiling of nitric acid, add nitric acid 1 ml again.

Then, add nitric acid 1 ml continually and heat it until mixed material is disassembled.

The color is not thicker than rice straw color means oxidation of organic material is nearly finished.

- (4) Cool a flask slowly and add 0.5 ml hydrogen peroxide.

Heat until white smoke is generated and add 1ml nitric acid carefully during boiling.

If white smoke is generated much in the state of non-boiling, add hydrogen peroxide

and nitric acid additionally.

Then, record the amount of used hydrogen peroxide.

(5) When oxidation is completely finished, cool the flask.

At this moment, cool and wash a flask inlet and a neck part by distilled water 5 ml in order to mix with remnants.

Reheat and generate white smoke.

Repeat former operation that mix with water and heat several times.

This operation is to remove remained hydrogen peroxide.

4. Molybdivanado method

4.1 Summary

This is below 2% phosphor quantitative analysis method.

4.2 Summary of method

After oxidizing organic material in a sample, change phosphors into phosphate ion and dilute up to fixed quantity.

Add ammonium vanadate solution and ammonium molybdate solution in order until color becomes appeared. Then, measure degree of light absorption at 400 ~ 470nm.

4.3 Equipment

Spectrophotometer

4.4 Reagent

- (1) Ammonium molybdate solution: After dissolving 50 g ammonium molybdate into warm water and diluting up to 1 l indicating line with distilled water, use by filtration.
- (2) Ammonium vanadate solution: After dissolving ammonium vanadate into 500 ml warm water and adding 20 ml nitric acid, dilute up to indicating line with water.
- (3) Phosphor standard solution(1 ml = 0.1mgP) : Make solution of 1 l by dissolving 0.4393 potassium dihydrogen phosphate into water.
- (4) Sulfuric acid