

KES

Analysis of Sulfur in Petroleum Products

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1. Scope of Application

This standard shall specify the method to measure lubricating oils containing an annex (additive) and all sulfur powder of lubricating oil annex at high temperature. This test method shall apply to the product with temperature of more than 177 °C and specimen with more than 0.06 %. If the concentration of chlorine is less than 1 %, it is not an obstacle to the test. In addition to these obstacles to the test, the degree of obstructing test is different with respect to the form of nitrogen compounds. Zinc, phosphorus, lead, alkali, metals and alkali-plated metals are not obstacles to the test.

2. Summary

The specimen is burnt long enough in the oxygen stream of high temperature for more than 97 % of sulfur powder to be sulfur dioxide. A concentration coefficient is applied to obtain accurate results. The product is passed through the absorber, in which iodine acid potassium and acid solution of starch reagent are inserted.

If the standard iodine acid potassium solution is added to the solution in the absorber, light blue color will appear. The blue will disappear as the combustion reaction is processing. At this moment, iodine acid potassium solution is added more. The quantity of sulfur inserted in the specimen is affected by the standard iodine acid potassium solution consumed in the combustion reaction.

3. Apparatus

3.1 Combustion apparatus

Following 2 types are applied and the difference of the two is the method to obtain the necessary high temperature.

(1) Induction furnace

Electric high frequency induction apparatus is applied for heating. The combustion temperature of specimen on the condition 7 obtained at least 1482 °C with this apparatus. The top of the combustion region is composed of induction coil. ⁽¹⁾

Note⁽¹⁾ There is a danger of electric high frequency combustion or high-pressure shock on this type of furnace. Therefore, there should be a safety preparation to these. When electric trouble repair or adjust is needed, it should always be

possible to cut the power line to the furnace.

(2) Resistant silicon carbide resistor

Capable of keeping the temperature of 1316°C

3.2 Absorber

It follows the specification of KS M 2054 [Test method of lamp type sulfur powder for petroleum products (Volume method)]. ⁽²⁾

Note⁽²⁾ Automatic optimal method is able to be applied for both types of furnace and it should especially be designed to perform the iodine optimization. Therefore, absorbing function and optimizing function to expect the terminal point can be linked together.

3.3 Burette

Standard burette (25 mL) or automatic burette by manufacturer of special combustion apparatus is applied.

3.4 Other apparatus

For the special combustion apparatus, vessel, combustion boat, cap of vessel, bar inserted in the bottom of boat, several magnetic pieces, combustion pipe, oxygen flow meter and oxygen desiccator are required.

Supplementary apparatus differs by the types of furnace. All equipments, which are used in the induction furnace prior to the test, deformed plastically the low sulfur content specimen, which is explained in 1, on the muffler of 1315 °C at least more than 4 hours and used.

4. Reagent and Material

4.1 Oxidized aluminum (Al_2O_3) or oxidized magnesium (MgO) reagent grade.

4.2 Hydrochloric acid (HCl 3 : 197) specific gravity 1.19

Dissolve reagent grade hydrochloric acid 30 mL in the water (H_2O) and make it 2L.

4.3 Perchloric acid magnesium (or oxidized phosphorus) reagent grade.

4.4 The purity of oxygen should be at least more than 99.5% and sulfur powder should not appear on the ground test.

4.5 Iodine acid potassium standard solution (0.06238 N, 1 ml = 1 mg S) Iodine acid potassium solution (0.06238 N, 1 ml = 1 mg S) Accurately take 100 ml to 1 l of mass flask. Pour in the water and make it 1L of well-mixed solution.

4.6 Iodine acid potassium standard solution (0.0124 N, 1 ml = 0.2 mg S) Iodine acid potassium solution (0.06238 N, 1 ml = 1 mg S) Accurately take 200 ml to 1 l of mass flask. Pour in the water and make it 1L of well-mixed solution.

4.7 Iodine acid potassium standard solution (0.06238 N, 1 ml = 0.2 mg S) 2.225 g of the dried iodine potassium is dissolved in the water at 180 °C until it desolates and make it 1 l of well mixed solution.

4.8 Askalite powder is 8 ~ 30 meshes.

4.9 Special materials, which is applied to the induction furnace.

4.9.1 Tartar : Powder is 20 ~ 20 meshes

4.9.2 Hastener : Sulfur content is 0.005. The inserted sample should not exceed the weight (%).

4.10 Potassium alum [$\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$] an alum for standard test is applied.

4.11 Iodine potassium starch solution

Dissolve 9g of soluble starch in the 15 ml of water. Then, this is inserted in the 500 ml of boiling water with stirring. This compounds is cooled down and add 15 g of iodine potassium (KI). Then, make it 1 l of solution by adding water.

4.12 Sulfuric acid

Strong sulfuric acid (H_2SO_4) reagent grade Sulfuric acid (Specific gravity 1.84) is applied.

4.13 Vanadium anhydrous powder V_2O_5 Reagent grade anhydrous powder is