Ministry of Higher Education and Scientific Research
AL Mustaqbal university college
Chemical Engineering Department

Petroleum products Properties Laboratory

3rd. Stage.

Exp. No. 6
Sulfur content of Petroleum Products Test

Prepared by

Asst. lect. Ban Ali Hassan

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Purpose of this test:

This test method covers the determination of sulfur in petroleum products, including lubricating oils containing additives، additive concentrates, and lubricating greases that cannot be burned completely in a wick lamp.

Introduction and theory :

This test method is valid in the concentration range of 0.01 to 0.04mass % of sulfur in liquid petroleum products. By using a special sulfate analysis procedure the determination can be extended to as low as 5 mg/kg of sulfur. The direct burning procedure is applicable to gasoline, kerosene, naphtha, and other liquids that can be burned completely in a wick lamp.
The blending procedure is applicable to gas oils, distillate fuel oils, naphthenic acids, alkyl phenols, high sulfur content petroleum products, and other materials that cannot be burned satisfactorily by the direct burning procedure. Phosphorus compounds normally present in commercial gasoline do not interfere. A correction is needed for the small amount of acid resulting from the combustion of lead antiknock fluids in gasolines. Appreciable concentrations of acid or base-forming elements from other sources interfere when the titration procedure is employed, since no correction is provided in these cases.

(A) These methods are suitable for the determination of the total sulphur content of petroleum products such as gases, liquefiable gases, light distillates, kerosines, and gas oils. Gas oils require dilution with a volatile combustible solvent of known low-sulphur content. The method can also be applied to combustible materials not of petroleum origin, but coming within the volatility range covered by the above petroleum products.

(B) The test solutions from the absorber can be examined by volumetric, gravimetric or turbidimetric methods according to need. These are designated A, B and C respectively.

(C) The results obtained by the volumetric method (A) are slightly high, owing to formation of nitric acid during combustion. For petroleum products, the error is usually of the order of 0 01 per

cent, sulphur, but it varies with the nature of the sample and the conditions of burning. The results obtained by the gravimetric method (B) and the turbidimetric method (C) are not affected by nitric acid formation, and are thus more accurate. Method B is suitable for sulphur contents down to 0 001 per cent., and Method C for sulphur contents down to 0 0001 per cent. Method C has a normal upper limit of 0-005 per cent, sulphur, but this can be considerably extended by burning small quantities of sample or by working with aliquot portions of the test solutions.

The method has the following advantages:
i) The lamp specified for use with liquid products enables samples to be burned more rapidly than that prescribed in I.P.—62.
ii) The lamp facilitates the combustion of highly aromatic materials without smoking. In exceptional cases a small amount of aromatic free diluent of known low sulphur content may be required.
iii) Provision is made for carrying out the combustion in purified air.
Sulphur present as carbon disulphide can be estimated satisfactorily.

Significance and Use

Applicable Standard BK-1027 Sulfur lamp method unit conforms to the ASTM D1266 standard Test Methed for Sulfur in Petroleum Froduets (Lamp Method).This apparatus covers the detemination of total sulfur in liquid petroleum producets in concentrations from 0.01 to OA mass%. A spccial sulfate analysis procedure pemits the detemination of sulfur in concentrations as low as 5 mg/ kg.

The instrument is designed and made as per the national standard of People’s Republic of China GB/T380 Standard Test Method for Sulfur Content in Petroleum Products (Lamp Method). It is used to determine the sulfur content in light oils (such as gasoline, kerosene) which the Reid vapor pressure is not higher than 600mmHg as per GB/T380. I.

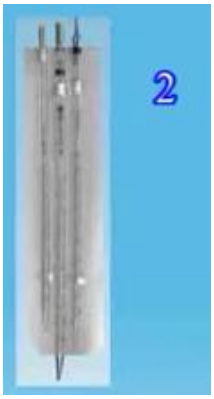
Main technical features

1. Small desktop structure. Can do determination for 5 samples at a time.
2. The lamp is located in a fixed seat. The height and sucking rate for each can be adjusted independently.
3. Stainless steel material. Reasonable design and easy to clean.

Petroleum product sulfur content tester.

Equipment's Used :

1.Base frame of burning lamp is equipped with lift adjusting device
2. Strut and the vacuum manifold adopt stainless steel
3. Vacuum manifold
4.Regulating valve
5.Glasswares support plate is made of organic glass pane
6.Crganie glass support plate
7.Vacuum pump



1. Burette Used for neutralization titration the sulfur dioxide (SO_2) in the collector.
2. Pipette: Used for measuring the volume of the sample and introducing to the lamp
3. Washing bottle: Washing bottle with distillated water is used for cleaning the residues en the collector, chimney and absorber.

4. Balances Used for measuring the weight of the sample

5. Cotton wicking: Used for being instilled in the wick tube in the lamp burning the sample

6. Glass bead: Used for being installed in the tank of the collector as high as the 23 of the collector.

Procedure :

- the cotton wicking is cut to the required lengths ،
- insert the lamp wick into the wick tube , user can use a copper wire to do this .
-plug the lamp with the wick tube , the lower end of the wick is placed at bottom of the lamp .
-before the best , clean the collector , chimney and absorber .
-after washing the lamp and the wick with the petroleum ether , dry them .
-chose the sample weight according to the sulfur content of different sample.
-after the sample soaks the wick , cut the wick out of the tube.
-after the upper limbs of the wick and the wick tube edge becomes flush ,light the lamp .
-make the flame high as 5 to 6 mm, then , lid the lamp with lampshade to extinguish the flame.
-weight the lamp on the balance to the nearest 0.0004 g and accomplish the second sample according to the same way .
-enclose the tank with glass bead washed by distilled water.
- and use the pipette to inject exactly 10ml 0.3% sodium carbonate solution .
-measure 10 ml distilled water into the tank.
-turn on the pump power switch .
-put the lamp in the tray on the pedestal.
-light the lamp, avoid using matchstick .
-adjust the height of the tray to make the top of the wick is at most 8 mm higher than the chimney.
-adjust the valve to make the section of rate keep constant and avoid the black smoke in the flame.
-after the sample burner out .
-inject 1 to 2 ml standard normal heptane or 95% ethanol or gasoline to the lamp to burn it out.
-extinguish the lamp after sample burns out, lid the lamp shade. 3 to 5 min. later , turn off vacuum pump.
-disconnect the vacuum manifold and the upper mouth of the collector.
-wash the collector, chimney and absorber by spraying the distilled water , allow the distilled water to flow into the absorber .

-after injecting the indicator , titrate with 0.05 N hydrochloric solution and air agitation until the solution becomes red.

-After the Test

1. Fill in the test record.
2. Calculate the dosage of the sample and the volume of the hydrochloric solution according to the requirement.
3. Caleulate the test results according the sulfur content form
4. Clean the test glassware subassemblies

Calculation :

Calculate the sulphur content by means of the following formula:

Sulfur per cent. wt. $= \frac{(A-B)\times 13.73}{W}$

Where:

A = weight in g. of barium sulphate obtained from sample،

B = weight in g. of barium sulphate obtained from blank, and

W = weight in g. of sample burned.

Test Summary

A sample is burned in a closed system using a suitable lamp and an artificial atmosphere composed of 70 % carbon dioxide and 30 % oxygen to prevent formation of nitrogen oxides. The oxides of sulfur produced are absorbed and oxidized to sulfuric acid by means of hydrogen peroxide solution which is then flushed with air to remove dissolved carbon dioxide. Sulfur as sulfate in the absorbent is determined acidimetrically by titration with standard NaOH solution, or gravimetrically by precipitation as barium sulfate. Alternatively, the sample may be burned in air and the sulfur as sulfate in the absorbent be determined by precipitating as barium sulfate for weighing. For sulfur content below 0.01 mass %, it is necessary to determine sulfur in the absorber solution turbidimetrically as barium sulfate.

Test Method	Repeatability	Reproducibility
D 1266: 0.01 to 0.04 m % Sulfur	0.005	$0.10+0.25\,X$
D 1266, Annexe Al: Sulfur 5 to 80 ppm	$0.116\,X$	$0.145\,X$
Sulfur over 80 to 280 ppm	$(0.01\,X)+8.5$	$(0.508\,X)-45.4$

Where X is the mean sulfur concentration in mass % for Test Method D 1266 and in mg/kg for Test Method D 1266, Annexe Al.

The bias of these test methods has not been determined.

Discussion:

1. What is the purpose of this experiment ?
2. What are the types of indicator can be used in the experiment ?
3. What are the parts of the device used in the experiment?
4. What are the other sample can be used in this experiment?