



A Honeywell Company

Free Sulfur in Distillates by Mercury Number

UOP Method 286-13

Scope

This is a semi-quantitative method for determining free (elemental) sulfur or polysulfide sulfur dissolved in peroxide-free petroleum distillates. The lower limit of detection is a mercury number of 1, which is equivalent to approximately 1 mg/kg (mass-ppm).

References

ASTM Method D1298, "Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method," www.astm.org

ASTM Method D4052, "Density, Relative Density, and API Gravity of Liquids by Digital Density Meter," www.astm.org

Outline of Method

The oil sample is shaken with clean mercury and oleic acid. The resulting suspension of mercuric sulfide is visually compared to a set of previously prepared standards containing known amounts of elemental sulfur and the mercury number is calculated. A mercury number of 1 corresponds to a concentration of 1 mg of elemental sulfur per liter of sample.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Balance, readable to 0.0001 g

Bottles, clear oil sample, 118-mL, with cork stopper, Fisher Scientific, Cat. No. 02-911-472

Cylinder, graduated, 10-mL, Fisher Scientific, Cat. No. 80-555A

Flask, volumetric, 1000-mL, Fisher Scientific, Cat. No. 10-212-5L

Pipet, Mohr, 1-mL, 0.1-mL sub-divisions, Fisher Scientific, Cat. No. 13-665-G

Pipets, volumetric, Class A, 2- and 100-mL, Fisher Scientific, Cat. Nos. 13-650-2C and -2U, respectively

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Mercury, triple distilled, Fisher Scientific, Cat. No. 7439-97-6

Oleic acid, Fisher Scientific, Cat. No. A195

Sulfur, precipitated, Fisher Scientific, Cat. No. S595. Flowers of sulfur cannot be used.

Toluene, 99.8% minimum purity, Fisher Scientific, Cat. No. T324

Procedure

The analyst is expected to be familiar with general laboratory practices and the equipment being used. Dispose of used reagents, materials, and samples in an environmentally safe manner according to local regulations.

Preparation of Standards

Prepare a standard sulfur stock solution as follows:

1. Place $1.0 \text{ g} \pm 0.0001 \text{ g}$ of precipitated sulfur in a 1000-mL volumetric flask.
 - Do not use flowers of sulfur.
2. Add toluene to dissolve the sulfur.
3. Fill to the mark with toluene and shake to mix.
 - This standard sulfur stock solution contains 0.001 g of elemental sulfur per mL.
4. By pipet, place 100 mL of toluene and 2 mL of oleic acid into each of 10 clear oil sample bottles.
 - The oleic acid is added as a dispersant for the mercuric sulfide formed.
5. With a 1-mL Mohr pipet, add 0.1 mL of standard sulfur stock solution to the first sample bottle, 0.2 mL to the second sample bottle, 0.3 mL to the third sample bottle, and continue until 1.0 mL is added to the 10th sample bottle. This results in a series of standards containing from 0.1 to 1.0 mg of elemental sulfur per 100 mL in 0.1-mg increments. Label the bottles with the concentration of elemental sulfur.
6. Using a graduated cylinder, add 3 mL of mercury to each bottle, stopper and shake for 3 minutes.
 - CAUTION: Take suitable precautions to avoid contact with mercury or mercury vapors
 - Prepare new standards at monthly intervals.

Determination of Free Sulfur

1. By pipet, add 100 mL of the oil sample and 2 mL of oleic acid to a sample bottle.
2. Add 3 mL of mercury, stopper and shake vigorously for 3 minutes.
3. Compare the color of the suspension in the unknown sample to the set of freshly shaken standards using any source of transmitted light.
4. If the sample is darker than the standard containing 1.0 mg sulfur per 100-mL, repeat the determination on an aliquot of the sample diluted to 100 mL with toluene to obtain a match within the range of standards.

5. Record the volume of sample used and the mg of sulfur per 100 mL in the standard that was matched.
6. Obtain the density of the sample by ASTM Method D4052, "Density, Relative Density, and API Gravity of Liquids by Digital Density Meter," or D1298, "Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method," if mass-ppm of free sulfur is to be calculated.

Calculations

Calculate the mercury number of the sample using Equation 1:

$$M = \frac{(100 A) 10}{S} \quad (1)$$

where:

M = mercury number
A = sulfur in matching standard, mg/100 mL
S = volume of sample used, mL
10 = factor to convert from mg/100 mL to mg/L
100 = volume of sample or diluted sample analyzed, mL

Calculate the mass-ppm free sulfur in the sample using Equation 2:

$$\text{Free Sulfur, mass-ppm} = \frac{M}{D} \quad (2)$$

where:

D = density of the sample
M = mercury number, from Equation 1

Note

The presence of hydrogen sulfide is not an interference.

Precision

Repeatability

Based on two tests performed by each of two analysts, on each of two days for a total of eight analyses in one laboratory, the within-laboratory estimated standard deviations (esd) could not be calculated for free sulfur in toluene as no variation was found at the 1, 10, and 20 mercury number levels. Two tests performed in the one laboratory by different analysts on different days should not be expected to differ.

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

Time for Analysis

The elapsed time and labor required, not including the preparation of standards, are identical, 0.3 hour.

Suggested Supplier

Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219-4785, USA (412-490-8300)
www.fishersci.com