



A Honeywell Company

Doctor Test for Petroleum Distillates

UOP Method 41-07

Scope

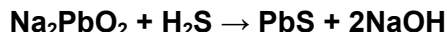
This is a qualitative test for the presence of hydrogen sulfide, or mercaptans in the absence of hydrogen sulfide, in gasoline, jet fuel, kerosine and similar petroleum products. ASTM Test Method D 4952, "Qualitative Analysis for Active Sulfur Species in Fuels and Solvents (Doctor Test)" is similar.

Reference

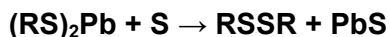
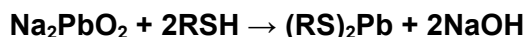
ASTM Method D 4952, "Qualitative Analysis for Active Sulfur Species in Fuels and Solvents (Doctor Test)," www.astm.org

Outline of Method

The sample is shaken with a sodium plumbite solution in a test tube. If hydrogen sulfide is present the following reaction occurs:



The lead sulfide is black and readily visible. If this reaction does not appear, sulfur is added to the test tube and the mixture shaken again. If mercaptans are present, on shaking they undergo a series of reactions, coloring the hydrocarbon layer first orange, then red and brown, and finally a black precipitate of lead sulfide appears. The overall reaction may be written:



The sodium plumbite (Doctor) solution may be prepared or purchased.

Apparatus

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

Balance, readable to 0.01 g, for preparation of Doctor Solution

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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Flask, Erlenmeyer, with cap, 1000-mL, VWR, Cat. No. 10-041-6C, for preparation of Doctor Solution

Funnel, filter, polypropylene, 150-mm top I.D., VWR, Cat. No. 30256-006

Pen, marking, resistant to water and hydrocarbons, VWR, Cat. No. 54848-150, for placing calibration marks on the test tube. Alternatively, a scribe may be used, VWR, Cat. No. 82024-590

Pipets, 5- and 10-mL, VWR, Cat. Nos. 89003-348 and -350, respectively

Pipet filler, VWR, Cat. No. 53497-053

Spatula, micro-spoon, stainless steel, glazed finish, VWR, Cat. No. 57952-311

Stopper, rubber, sulfur free, Size 5, VWR, Cat. No. 59589-212

Test tube, 25 x 150 mm, VWR Cat. No. 89000-490. Place permanent calibration marks at 5 mL and 15 mL with marking ink. Place the marks by measuring the depth of 5 mL of water from a pipet followed by 10 mL.

Timer, 60-minute, GraLab, VWR, Cat. No. 62371-001

Reagents and Materials

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

Doctor solution, VWR, Cat. No. VW3547-1. The Doctor Solution may be purchased commercially, or it may be prepared as described under *Procedure, Preparation of Doctor (Sodium Plumbite) Solution*

Filter paper, medium, Whatman Grade 40, 240-mm diameter, VWR, Cat. No. 28475-140

Lead (II) oxide (litharge), VWR, Cat. No. AA12220-36, for preparation of Doctor Solution

Sodium hydroxide, VWR, Cat. No. EM-SX0590-1, for preparation of Doctor Solution

Sulfur, VWR, Cat. No. EM-SX1225-1

Water, distilled

Procedure

The analyst is expected to be familiar with general laboratory practices and with the equipment being used.

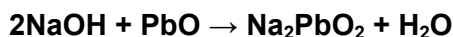
Calibration of Test Tube

Place permanent calibration marks on the test tube at 5 mL and 15 mL.

1. Pipet 5-mL of water into the test tube. Mark the level with marking ink or the scribe.
2. Pipet an additional 10-mL of water into the test tube. Mark the level with marking ink or the scribe.

Preparation of Doctor (Sodium Plumbite) Solution

Doctor Solution may be purchased already prepared, or it may be prepared as described below according to the following reaction:



1. Dissolve 125 g of sodium hydroxide in 1 L of distilled water.
2. Add 60 g of lead (II) oxide (litharge) and shake vigorously for 15 minutes. Let stand overnight.
3. Decant or siphon off the clear liquid. If the liquid does not settle clear, filter it through filter paper.
4. Keep the solution in a tightly-stoppered bottle away from sunlight. Refilter if it becomes cloudy.

Sample Analysis

1. Fill the test tube to the first mark with Doctor Solution.
2. Fill the test tube to the second mark with sample.
3. Stopper and shake vigorously for 15 seconds.
4. Observe any appearance of black or brown lead sulfide precipitate. If this occurs, hydrogen sulfide is present. Stop the test. Report "Hydrogen sulfide present."
5. In the absence of a brown or black precipitate, use a micro-spoon spatula to add a small amount of sulfur to the mixture in the test tube, not more than will just cover the interface between the hydrocarbon and Doctor Solution.
 - Use only sufficient sulfur to form a thin film floating on the interface between the sample and the doctor solution. Approximately 40 to 100 mg of sulfur is appropriate.
6. Shake vigorously for 15 seconds and allow any precipitate and the sulfur to settle for one minute.
7. Observe again and report as indicated in the next section.
8. Dispose of the solution, containing lead, in an environmentally safe manner according to local regulations.

Report

a) "Hydrogen sulfide present" (from *Sample Analysis*, Step 4).

b) Sample sour.

If a brown or black precipitate forms after the addition of sulfur, the sample contains a relatively high concentration of mercaptans and is reported "sour".

c) Sample borderline or sweet.

If the mercaptan content of the sample is low, observe the sulfur layer and judge as follows:

Discoloration of Floating Sulfur **(from its original yellow color)**

Definitely discolored
Barely discolored
Not discolored

Report

"sour"
borderline
"sweet"

Precision

Since this is a qualitative test, no numerical precision is determined.

Time for Analysis

The elapsed time and labor requirement for one analysis are identical, 0.5 hours.

Suggested Suppliers

VWR International, 1310 Goshen Parkway, West Chester, PA 19380 (610-431-1700) www.vwr.com