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# International Standard



# 5275

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## Aromatic hydrocarbons — Test for presence of mercaptans (thiols) — Doctor test

*Hydrocarbures aromatiques — Essai de détection des mercaptans (thiols) — «Doctor test»*

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**Descriptors** : aromatic hydrocarbons, chemical tests, detection, thiols.

## FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5275 was developed by Technical Committee ISO/TC 78, *Aromatic hydrocarbons*, and was circulated to the member bodies in October 1977.

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It has been approved by the member bodies of the following countries:

Australia	Hungary	Portugal
Austria	India	Romania
Brazil	Israel	South Africa, Rep. of
Bulgaria	Korea, Rep. of	Turkey
Chile	Mexico	United Kingdom
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France	Philippines	Yugoslavia
Germany, F. R.	Poland	

No member body expressed disapproval of the document.

# Aromatic hydrocarbons — Test for presence of mercaptans (thiols) — Doctor test

**WARNING** — Aromatic hydrocarbons are generally toxic by inhalation, ingestion or skin absorption. Volatile aromatic hydrocarbons are also highly flammable.

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the "Doctor" test for the presence of mercaptans (thiols) in aromatic hydrocarbons.

Hydrogen sulphide may also be detected by the test.

The method is inapplicable in the presence of more than traces of peroxides.

## 2 PRINCIPLE

Shaking of a test portion with sodium plumbite solution and observation of the mixture. From its appearance, the presence or absence of mercaptans (thiols), hydrogen sulphide, peroxides or elemental sulphur may be deduced, and may be confirmed by the addition of sulphur, shaking and observation of the appearance of the final mixture.

## 3 REAGENTS

During the test, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

### 3.1 Sulphur, sublimed ("flowers of sulphur"), dry.

Store in a closed container.

### 3.2 Sodium plumbite solution (Doctor solution).

Dissolve 25 g of lead acetate trihydrate  $[(CH_3COO)_2Pb \cdot 3H_2O]$  crystals in 200 ml of water, filter and add to a solution of 60 g of sodium hydroxide in 100 ml of water. Heat the mixture on a boiling water bath for 30 min, cool and dilute to 1 000 ml with water.

Store the solution in a tightly closed, rubber-stoppered bottle and filter before use if it is not clear.

### 3.3 Cadmium chloride solution, containing 100 g of cadmium chloride and 10 ml of hydrochloric acid, about 36 % (m/m) or approximately 11 M.

3.4 Potassium iodide, 100 g/l solution freshly prepared.

3.5 Acetic acid, 100 g/l solution.

3.6 Starch, 5 g/l solution, freshly prepared.

## 4 APPARATUS

4.1 Stoppered cylinder, made of glass, of 50 ml capacity, and several unstoppered glass cylinders.

## 5 SAMPLING<sup>1)</sup>

Take a representative sample of not less than 1 000 ml from the bulk of the material.

## 6 PROCEDURE

### 6.1 Preliminary test

Place 10 ml of the sample and 5 ml of the sodium plumbite solution (3.2) in the stoppered cylinder (4.1) and shake together vigorously for 15 s. Observe the appearance of the mixture and continue the procedure as indicated in the table.

TABLE — Variations of "Doctor test"

Observation	Inference	Continue test as described in
Black precipitate forms immediately	Hydrogen sulphide present	6.2
Brown precipitate forms slowly	Peroxides probably present	6.3
During the shaking period the solution becomes opalescent and then darkens in colour	Mercaptans and elementary sulphur present	—
No change occurs or yellow colour is produced		6.4

1) The sampling of aromatic hydrocarbons will form the subject of ISO 1995.

**6.2 If hydrogen sulphide is present**

Shake a fresh quantity of the sample with 5 % of its volume of the cadmium chloride solution (3.3) to remove the hydrogen sulphide. Separate the treated sample and carry out a further preliminary test (see 6.1). If this second test does not produce a black precipitate, continue as specified in 6.4. If the second test does produce a black precipitate, repeat the washing with a fresh portion of the cadmium chloride solution (3.3) and then continue as specified in 6.4.

**6.3 If peroxides are probably present**

To confirm the presence of peroxides in sufficient concentrations to invalidate the test, shake a further portion of the sample with 20 % of its volume of the potassium iodide solution (3.4), a few drops of the acetic acid solution (3.5) and a few drops of the starch solution (3.6). A blue colour in the aqueous layer confirms the presence of peroxides.

**6.4 Final test**

To the mixture obtained as specified in 6.1 or 6.2, add a small quantity of the sulphur (3.1) (not more than will just cover the interface between the sample and the sodium plumbite solution), shake the mixture for 15 s, and allow to settle for 1 min.

Observe the contents of the cylinder. If a brown or black precipitate is formed on the addition of sulphur, mercaptans are present.

**7 EXPRESSION OF RESULTS**

If an opalescent colour is produced during shaking with the sodium plumbite solution, and a brown or black precipitate

is formed after adding elementary sulphur, report the result of the test as "mercaptans (thiols) present".

If, after removal of hydrogen sulphide, a brown or black precipitate is formed after adding elementary sulphur, report the result of the test as "mercaptans (thiols) present".

If no precipitate is formed after the addition of elementary sulphur, report the result of the test as "mercaptans (thiols) absent".

If peroxides are present, the result of the test is invalid.

NOTE — Carbon disulphide, particularly in concentrations exceeding 0,4 % (m/m) of sulphur, present as carbon disulphide, causes a darkening of the aqueous layer on standing. The test is unreliable, therefore, for samples containing high amounts of carbon disulphide. Caution is always necessary to avoid confusing this darkening with the immediate blackening due to hydrogen sulphide or that produced by mercaptans. Similarly, certain phenolic substances (which may have been introduced as inhibitors) cause coloration of the aqueous layer, and, if their presence is suspected, a blank test with sodium hydroxide solution in place of the sodium plumbite solution should be carried out first for the purpose of comparison.

**8 TEST REPORT**

The test report shall include at least the following information

- a) the type and identification of the product tested;
- b) a reference to this International Standard;
- c) any deviation, by agreement or otherwise, from the procedure specified;
- d) the result of the test;
- e) the date of the test.