

**SOLVENTS
IDENTIFICATION OF BENZENE
(COLORIMETRIC METHOD)**

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RESTRICTIONS DESCRIBED IN THE NORME

This is a translation, the French original shall be used in all cases of litigation

Date of translation : 26/11/2004

PROHIBITED for NEW DESIGNS and NOT REPLACED**1.OBJECT AND FIELD OF APPLICATION**

The object of this method is to describe a method of operation to allow the identification of benzene which can be found in solvents, or mixtures of solvents used in the automotive industry.

2.PRINCIPLE

A mixture of fuming nitric acid and concentrated sulphuric acid, the benzene and its higher homologous products transform into meta-dinitro derivatives which give a violet-like colour with methyl ethyl ketone in an alkaline medium. If chromic acid is added to the nitrating reagent, the meta-dinitro benzene remains unaltered, while the nitrated derivatives of the higher homologous products are oxidised in their side chain and are transformed into nitrated aromatic acids.

After neutralisation of the medium, a methyl ethyl ketone extraction allows the meta-dinitro benzene derivatives to be isolated since these pass into the ketonic phase, the alkaline salts of the nitrated aromatic acids remaining in solution.

This selective separation enables benzene to be qualitatively detected by using specific colourimetric reaction.

3.REAGENTS**3.1.NITRATING MIXTURE**

consisting of the following:

- Nitric acid fuming ($d = 1,50$) quality: analytical quality purity (PPA).
- Concentrated sulphuric acid ($d = 1,83$) quality : analytical quality purity (PPA).

Slowly pour the sulphuric acid into the fuming nitric acid, previously cooled in ice, in order to reach a 1/1 by volume mixture.

3.2.SATURATED CHROMIC ACID SOLUTION

prepared as follows: in a wide neck flask which ground stopper, introduce 20 ml distilled water and 50 g approximately chromic acid (PPA).

Stir occasionally to facilitate dissolution; an excess of chromic anhydride is deposited at the bottom of the flask.

3.3.SODIUM HYDROXIDE

in aqueous solution ($d = 1,33$).

3.4.PURE METHYL ETHYL KETONE**3.5.PAPER**

pH (range from 5 to 9).

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4.EQUIPMENT

- water bath.
- 50 ml separating funnel.
- 20 ml Pyrex test tube.
- wide neck 250 ml flask which ground stopper.

5.METHOD OF OPERATION

5.1.NITRATION

In a Pyrex test tube, pour 5 ml of the nitrating mixture, cool in ice water and add, drop by drop, 0,5 ml of the solvent to be analysed. Dilute with care using iced distilled water (approximately 5 ml).

5.2.TEST FOR THE PRESENCE OF BENZENE

- Introduce into the test tube, 1 ml of the chromic solution and heat to boiling point in the water bath for 1/4 of an hour. Cool the tube in ice water, neutralise one drop at a time using the sodium hydroxide and check with pH paper (3.5); then make the solution slightly alkaline.
- Transfer into the separating funnel and pour 10 ml of methyl ethyl ketone. Stir energetically and allow decanting.
- In the presence of benzene, the upper ketonic layer will turn pink in colour.

6.REMARKS

6.1 This method is valid for benzene contents greater than 0,2 %.

6.2 This method allows a quantitative determination of the benzene to be made subsequently by absorption spectrophotometry in the visible range. Other methods such as infrared spectrography or gas chromatography may, can also be used according to the circumstances.

7.TEST REPORT

The test report must indicate, as well as the results obtained, the reference to this test method and any specific conditions different to those described and likely to have affected the results.

8.RECORDS AND REFERENCE DOCUMENTS

8.1.RECORDS

8.1.1.CREATION

- OR: 01/03/1980 – CREATION OF THE NORME

8.1.2.SUBJECT OF THE MODIFICATION

- B: 15/03/2002 – PROHIBITED FOR NEW DESIGNS (B. CARRAZE)
- A: 05/03/1997 – INTRODUCTION TO IDEM (French only)

8.2.REFERENCE DOCUMENTS

8.2.1.PSA DOCUMENTS

8.2.1.1.Normes

8.2.1.2.Others

8.2.2.EXTERNAL DOCUMENTS

8.3.EQUIVALENT TO:

8.4.CONFORMS TO:

8.5.KEY-WORDS