
International Standard



8176

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Butadiene for industrial use — Determination of active *tert*-butyl-catechol (TBC) [4-(1,1-dimethylethyl)-1,2-benzenediol] — High performance liquid chromatographic method

Butadiène à usage industriel — Dosage du tert-butyl-catéchol (TBC) actif [(diméthyléthyl-1,1)-4-benzènediol-1,2] — Méthode par chromatographie liquide à haute performance

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Foreword

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Butadiene for industrial use — Determination of active *tert*-butyl-catechol (TBC) [4-(1,1-dimethylethyl)-1,2-benzenediol] — High performance liquid chromatographic method

WARNING — [4-(1,1-Dimethylethyl)-1,2-benzenediol] is irritating to skin, particularly when molten or in concentrated solution. It is also toxic if swallowed or in contact with skin.

For all handling of butadiene, work in a well-ventilated hood away from flames and sparks. It is advisable to use proper personal protection, such as gloves and goggles.

1 Scope and field of application

This International Standard specifies a high performance liquid chromatographic method for the determination of [4-(1,1-dimethylethyl)-1,2-benzenediol] (*tert*-butyl catechol or TBC) in buta-1,3-diene for industrial use.

The polymerization inhibitor TBC can also be determined by a spectrometric method (ISO 6684) but that method is not able to differentiate between the active inhibitor and its oxidized, inactive form. The present method specifically measures the concentration of the active form of TBC. The method is applicable to butadiene having TBC contents in the range 0 to 250 mg/kg.

2 References

ISO 653, *Long solid-stem thermometers for precision use.*

ISO 6684, *Butadiene, for industrial use — Determination of tert-butyl catechol (TBC)[4-(1,1-dimethylethyl)-1,2-benzenediol] — Spectrometric method.*

ISO 8563, *Propylene and butadiene in liquid phase — Sampling.*¹⁾

3 Principle

Extraction of the active TBC in a test portion by mixing with a solution containing *m*-nitrophenol (as the internal standard) and evaporating the butadiene. Separation of the TBC and *m*-nitrophenol by high-performance liquid chromatography and detection by UV. Measurement of the peak areas or peak heights and determination of the TBC content using a calibration graph.

4 Reagents and materials

During the analysis, use reagents of HPLC grade or recognized analytical grade.

4.1 Methanol.

4.2 Acetic acid.

4.3 Chloroform.

4.4 Distilled or deionized water.

4.5 Standards.

4.5.1 TBC [4-(1,1 dimethylethyl)-1,2-benzenediol], 25 g/l solution in chloroform.

4.5.2 *m*-Nitrophenol internal standard, 25 mg/l aqueous solution.

5 Apparatus

Ordinary laboratory apparatus and

5.1 **Syringes for liquids**, of capacity 10, 25, 50 and 100 μ l, for the preparation of calibration solutions.

5.2 **Syringe for HPLC**, of capacity 50 μ l or more, for filling the injection loop.

5.3 Chromatograph.

Use a high-performance liquid chromatograph complying with the requirements specified below and which yields a peak

1) At present at the stage of draft.