



A Honeywell Company

Impurities in, and Purity of, High Purity *p*-Xylene by GC

UOP Method 720-08

Scope

This method is for determining trace hydrocarbon impurities in high purity *p*-xylene. The lower limit of detection for any single component is 0.002 mass-%. The method can also be used for determining the total purity of the *p*-xylene. Mixed xylene isomers and/or diethylbenzenes in other process streams should be determined using UOP Method 744, "Aromatics in Hydrocarbons by Gas Chromatography."

References

- ASTM Practice D4307, "Preparation of Liquid Blends for Use as Analytical Standards,"
www.astm.org
- UOP Method 744, "Aromatics in Hydrocarbons by Gas Chromatography," www.astm.org
- UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

The sample to be analyzed is injected into a gas chromatograph (GC) that is equipped with a fused silica capillary column, a capillary injection port, and a flame ionization detector (FID). The concentrations of individual or group impurities are determined by the external standard method of quantitation, wherein peak areas of the sample components are compared to the peak areas of a calibration blend analyzed under identical conditions and injection volumes. The concentration of the major component is then determined by subtracting the total impurities from 100%. See *Note 1* for alternative means of calibration and calculation.

Apparatus

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

Balance, readability 0.1-mg

Bottle, 120-mL, clear, round, glass, with TFE lined closure, VWR, Cat. No. 16151-286

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Chromatographic column, 60 m of 0.32-mm ID fused silica capillary, internally coated to a film thickness of 0.50 μm with cross-linked Carbowax 20M, Supelco, Cat. No. 2-4085 (Supelcowax 10). See *Note 2*.

Gas chromatograph, temperature programmable, built for capillary column chromatography, utilizing a split injection system having a glass injection port insert and equipped with an FID that will give a minimum peak height response of five times the background noise for 0.002 mass-% *o*-xylene when operated under the recommended conditions, Agilent Technologies, Model 7890

Data system, electronic, for obtaining peak areas. This device must integrate areas at a sufficiently fast rate so that narrow peaks typically resulting from use of a capillary column can be accurately measured. Agilent Technologies, ChemStation

Refrigerator/Freezer, explosion-proof or flammable storage, VWR, Cat. No. 55700-340

Regulator, air, two-stage, high purity, delivery pressure range 30-700 kPa (4-100 psi), Matheson Tri-Gas, Model 3122-590

Regulator, hydrogen, two-stage, high purity, delivery pressure range 30-700 kPa (4-100 psi), Matheson Tri-Gas, Model 3122-350

Regulator, nitrogen, two-stage, high purity, delivery pressure range 30-700 kPa (4-100 psi), Matheson Tri-Gas, Model 3122-580

Sample injector, any syringe or injector capable of injecting a repeatable 0.5- μL volume of sample. The use of an automatic injection device is required to achieve necessary repeatable injection volumes. See *Appendix*. Agilent Technologies, Model 7683.

Reagents and Materials

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

Air, total hydrocarbons less than 2.0 ppm as methane (zero gas)

Carbon disulfide, low organic impurity, VWR, Cat. No. AA40910-AP

p-Diethylbenzene, 99.9% minimum purity, Chemsampco, Cat. No. 1135.72-1. See *Note 3*.

Ethylbenzene, 99.9% minimum purity, Chemsampco, Cat. No. 2379.03-1

Gas purifier, for hydrogen, to remove oxygen and moisture from carrier gas, VICI Mat/Sen, Cat. No. P200-1

Hydrogen, 99.95% minimum purity, total hydrocarbons less than 0.5 ppm as methane (zero gas)

Nitrogen, 99.99% minimum purity, total hydrocarbons less than 0.5 ppm as methane (zero gas)

n-Nonane, 99.9% minimum purity, Chemsampco, Cat. No. 6780.00-1

Pipet bulbs, VWR, Cat. No. 15001-362

Pipets, disposable, Pasteur, VWR, Cat. No. 14673-043

Syringe, replacement, for recommended sample injector, 5- μL , Agilent Technologies, Cat. No. 5181-1273

Vials, autosampler, for recommended sample injector, Agilent Technologies, Cat. No. 5182-0864

m-Xylene, 99.9% minimum purity, Chemsampco, Cat. No. 1240.12-1

o-Xylene, 99.9% minimum purity, Chemsampco, Cat. No. 1240.02-1

p-Xylene, 99% minimum purity, Chemsampco, Cat. No. 1240.21. See *Purification of p-Xylene*.

Calibration

Purification of *p*-Xylene

p-Xylene containing no other measurable C₈ aromatic impurities must be prepared to be used in a calibration blend. Prepare the purified *p*-xylene as follows.

1. Cool approximately 500 mL of commercial *p*-xylene in an explosion-proof freezer at $-10 \pm 5^\circ\text{C}$ until approximately 1/2 to 3/4 of the *p*-xylene crystallizes.
 - This requires about 5 hours.
2. Remove the *p*-xylene from the freezer and decant and discard the liquid portion.
3. Allow the frozen *p*-xylene to thaw, and then repeat the above crystallization step on the remaining *p*-xylene several times until no ethylbenzene, *m*- and *o*-xylene are detected in the *p*-xylene, as indicated by gas chromatographic analysis (refer to *Chromatographic Technique*). See *Note 3*.
 - Typically 4 or 5 recrystallizations are required.

Preparation of Calibration Blend

Quantitative results are based on the injection of repeatable volumes of both sample and calibration blend. Absolute response factors, derived from the calibration blend, are used to relate the peak areas of each known component to mass-ppm.

1. Prepare a stock solution as described in ASTM Method D4307 to contain approximately 1.5 mass-% each of *n*-nonane, ethylbenzene, *m*-xylene, *o*-xylene, and *p*-diethylbenzene in the purified *p*-xylene (see *Purification of p-Xylene*). Thoroughly mix the solution by shaking. Record all weights to the nearest 0.1 mg.
 - This blend will be used as the stock solution in the preparation of the actual calibration blend. Label this mixture as the stock solution.
2. Prepare the calibration blend to contain approximately 1.5 mass-% of the stock solution in purified *p*-xylene. Thoroughly mix the calibration blend by shaking. Record all weights to the nearest 0.1 mg.
 - If refrigerated, the stock solution and calibration blend should remain stable for several months.
3. Calculate the concentration of *n*-nonane, ethylbenzene, *m*-xylene, *o*-xylene, and *p*-diethylbenzene in the calibration blend to the nearest 0.0001 mass-% using Equation 1. Using the above percentages, the resulting calibration blend should contain approximately 0.023 mass-% of each added component.

$$M = \frac{100 AB}{CD} \quad (1)$$

where:

A = mass of *n*-nonane, ethylbenzene, *m*-xylene, *o*-xylene, or *p*-diethylbenzene in the stock solution, g

B = mass of base blend in the calibration blend, g

C = total mass of the stock solution, g

D = total mass of the calibration blend, g