

SCAQMD METHOD 306-91**ANALYSIS OF PENTANES IN EXPANDABLE STYRENE POLYMERS****1. Principle**

- 1.1 The pentane isomers in expandable styrene polymers are dissolved in toluene, then analyzed by gas chromatography (GC) with flame ionization detection. The weight percent pentanes in the expandable polystyrene polymer can be determined by this method.

2. Apparatus

- 2.1 Gas chromatograph equipped with a liquid injection system and a flame ionization detector
- 2.2 Electronic integrator
- 2.3 Analytical balance capable of weighing to 0.1 mg
- 2.4 GC column, 8 ft. x 1/8 in. O.D. stainless steel packed with 100/120 mesh Porapak^R Q, is recommended. Other columns that quantitatively resolve the individual pentanes and the internal standards may be used.
- 2.5 Glass syringe, 100 mL (calibrated with water), 25 mL, and 500 uL
- 2.6 Glass micro syringe, 10 uL and 2.0 ul, each with 0.1 uL graduations
- 2.7 Vials, crimp top, clear glass, 30 mL and 120 mL, cleaned according to Section 4.1
- 2.8 Septa, Teflon^R-faced, to fit vials
- 2.9 Seals, tear-away to fit vials
- 2.10 Plastic bags, with seals
- 2.11 Labels
- 2.12 Rubber gloves

3. Reagents

- 3.1 Isopentane, reagent grade
- 3.2 n-Pentane, 99.6%
- 3.3 Cyclopentane, reagent grade
- 3.4 n-Hexane, "Baker Instra - Analyzed", for trace organic analysis by GC.
- 3.5 Toluene, reagent grade
- 3.6 Toluene solution: To 3785 mL toluene, add 12 mL n-hexane and mix well. The n-hexane in this solution is the internal standard.
- 3.7 Acetone, pentane-free

4. Procedure

- 4.1 Preparation of Vials
 - 4.1.1 Use dry, clean rubber gloves to handle vials and samples in the execution of this section, and Section 4.2 in order to minimize contamination.
 - 4.1.2 Rinse vials, seals, and septa three times with pentane-free acetone. Air dry for about one hour under a clean hood.
 - 4.1.3 Transfer septa into a desiccator.
 - 4.1.4 Dry vials and seals in an oven at 105°C for one hour. Allow to cool to room temperature in a desiccator.
 - 4.1.5 Attach a unique identification label to each vial.
 - 4.1.6 Transfer vials and seals to a desiccator and allow to equilibrate for one hour.
 - 4.1.7 Weigh together, a labeled vial, a septum, and a seal (taken from the desiccator). Record weight in laboratory notebook.
 - 4.1.8 Place tared vial, septum, and seal in a plastic bag. Seal the plastic bag.

4.1.9 The vials are ready for use in sampling of expandable polystyrene samples (See Appendix I).

4.2 Preparation of Sample

4.2.1 Samples are collected as in Appendix I.

4.2.2 Allow vial to attain room temperature.

4.2.3 Wipe the outside surfaces of the vial with Kimwipes^R.

4.2.4 Allow to equilibrate in a desiccator for one hour.

4.2.5 For unexpanded beads, weigh 1 gram aliquot of the sample from Section 4.2.4 (record weight of aliquot as W_s) into a clean 30 mL vial with crimp top Teflon^R septum, cap and seal and immediately proceed to Section 4.2.7.

4.2.6 For prepuff and molded part samples:

4.2.6.1 Weigh total sample from Section 4.2.4.

4.2.6.2 Subtract the tare weight obtained in Section 4.1.7 from that obtained in 4.2.6.1. The resulting value is the sample weight (W_s).

4.2.6.3 Proceed to Section 4.2.7.

4.2.7 Add exactly 25 mL of toluene solution (Sec. 3.6) through the septum with the aid of a syringe. Mix to dissolve sample.

4.3. Preparation of Standards

4.3.1 With the aid of a syringe, add exactly 100 mL of toluene solution through the septum of an empty, capped 120 mL vial. Similarly, add exactly:

200 uL isopentane which makes a 0.143% w/w solution (equivalent to 0.124 grams isopentane in the standard).

200 uL n-pentane which makes a 0.144% w/w solution (equivalent to 0.125 grams n-pentane in the standard)

200 uL cyclopentane which makes a 0.173% w/w solution (equivalent to 0.150 grams cyclopentane in the standard)

4.3.2 Calibration standards prepared in 4.3.1 have been determined to be stable for one week.

4.4 Calibration and Analysis

4.4.1 The operating conditions for the gas chromatograph are:

N₂ carrier gas flow rate: 25 mL/min.

Injection port temperature: 150°C.

Detector temperature: 200°C

Oven program:

Hold at 150°C for 16 min

Ramp 10°C/min to 170°C

Hold at 170°C for 12 min

Ramp 20°C/min to 230°C

Hold at 230°C for 15 min

GC range: 2⁴

Attenuation: 2⁴

4.4.2 A 0.5 uL aliquot of the standard solution from 4.3.1 is introduced into the GC system where it is separated into component compounds.

4.4.3 Determine the peak height of each component in the standard.

4.4.4 A 0.5 uL aliquot of the sample from 4.2.7 is introduced into the GC where it is separated into component compounds.

4.4.5 Determine the peak height of each component.

5. Calculations

5.1 The response factor (RF) of each component is calculated using the following formula:

$$RF = \frac{C_{csl} \times H_{isl}}{V_{isl} \times H_{csl}}$$

Where: C_{CS1} = Weight of compound in the standard mix, g (Sec. 4.3.1).
 H_{CS1} = Measured peak height of the compound in the standard mix
 H_{IS1} = Measured peak height of the internal standard in the standard mix.
 V_{IS1} = Volume internal standard in the standard mix, mL.

5.2 The concentration of each component in percent by weight is calculated by the following:

$$\text{Concentration, (\% w/w)} = \text{RF} \times \frac{H_{CS2} \times V_{IS2}}{W_S \times H_{IS2}} \times 100$$

Where:

RF = Response factor of component (Sec. 5.1)
 H_{CS2} = Measured peak height of component in the sample
 H_{IS2} = Measured peak height of internal standard in the sample
 W_S = Weight of sample, g (Sec 4.2.5 or 4.2.6.2)
 V_{IS2} = Volume of internal standard in the sample, mL

6. Quality Control

6.1 Prepare a 5-point calibration curve to determine linearity monthly.

6.1.1 Use prepared standard solution from Section 4.3.1 for one of the calibration points and as the stock solution for the preparation of other calibration standards.

6.1.2 Prepare other calibration standards as follows:

6.1.2.1 Mix one volume of stock standard with 19 volumes of toluene solution.

6.1.2.2 Mix one volume of stock standard with 9 volumes of toluene solution.

6.1.2.3 Mix one volume of stock standard with 3 volumes of toluene solution.

- 6.1.2.4 Mix one volume of stock standard with 1 volume of toluene solution.
- 6.1.3 Inject 0.5 uL aliquots of each calibration standard.
- 6.1.4 Determine the peak height of each component.
- 6.1.5 Determine the linear range of the method.
- 6.2 Prepare calibration standards weekly.
- 6.3 Determine initial average response factor for each standard.
- 6.4 The GC system is checked daily with a calibration standard. Prepared calibration standard 6.1.2.2 is recommended.
- 6.5 The calibration check is repeated if the response of the instrument has changed by more than 10% from those obtained in 6.3.
- 6.6 If the response is still out of tolerance, remedial action is initiated, and new calibration factors calculated.
- 6.7 System blanks are analyzed daily after the calibration is completed, and whenever necessary, between samples to check for contamination and/or carry over from previous injection. Diluent treated as a sample constitutes a system blank. The linearity check standard is analyzed after the system blank.
- 6.8 Duplicate runs are performed on all samples until enough data are gathered to construct a control chart (30 samples). This control chart will be used to determine if duplicate runs are in control and if remedial action is necessary.
- 6.9 For manual injection, if the duplicate runs vary by more than $\pm 10\%$, another duplicate run is performed. If the variation is still more than 10%, remedial action is initiated. For laboratories with auto-samplers, if duplicate runs vary by more than $\pm 5\%$, remedial action is initiated.
- 6.10 If the drift of retention times results in peak misidentification, all instrument parameters are checked and reset in order to obtain original retention times. Retention time criteria are established during the initial calibration.

- 6.11 Every tenth unexpanded bead sample taken should be prepared in triplicate and analyzed as follows:
 - 6.11.1 One preparation is analyzed as a regular sample.
 - 6.11.2 A second preparation is analyzed as a duplicate
 - 6.11.3 The third preparation is spiked with standard and analyzed as the spike sample. The amount of spike recovered should be within 10% of the theoretical value.
- 6.12 To obtain an instrument limit of detection, use prepared standard (6.1.2.1) or a standard which is within a factor of 20 of the estimated limit of detection. Limit of detection is three times the standard deviation of a minimum of twenty runs.

Appendix I Method for the Sampling of Expandable Polystyrene for Pentane Analysis

1. Introduction

- 1.1 This procedure describes a method of sampling expandable polystyrene for pentane analysis.
- 1.2 This procedure is a modification of Method No. 1 submitted to the District by the Society of the Plastics Industry, Inc.

2. Sample Collection

2.1 Equipment

- 2.1.1 Hand crimper
- 2.1.2 Tared 30 mL vials with septa and seals

2.2 Procedure

- 2.2.1 Remove vial from the plastic bag, add sample as follows:
For unexpanded beads, prepuff and molded part samples, fill the vial to the top with sample. Bead samples must be taken within 5 minutes after opening a carton and from at least 6 inches beneath the surface of the beads.
- Select representative section of the molded part for the sample. Avoid edges and sections of poor fusion. Do not take sample from edges that have been hot wire cut.
- 2.2.2 Immediately set a septum over the top of the vial with the Teflon^R side toward the samples, place a seal over it and crimp tightly.
- 2.2.3 Label the sample properly.
- 2.2.4 Keep sample in a container at <4°C (40°F) whenever feasible, for transport to the laboratory.
- 2.2.5 Return sample to the laboratory for analysis.

SCAQMD METHOD 306-91**ANALYSIS OF PENTANES IN EXPANDABLE STYRENE POLYMERS**

This is a method for analyzing isopentane, n-pentane, and cyclopentane in expandable styrene polymers. It is applicable to samples regulated by rules under Regulation XI.

CONTENTS

	<u>Page</u>
1. Principle	1
2. Apparatus	1
3. Reagents	2
4. Procedure	2
4.1 Preparation of Vials	2
4.2 Preparation of Sample	3
4.3 Preparation of Standards	3
4.4 Calibration and Analysis	4
5. Calculations	4
6. Quality Control	5
7. Appendix I: Method for the Sampling of Expandable Polystyrene for Pentane Analysis	8

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

APPLIED SCIENCE & TECHNOLOGY DIVISION

LABORATORY SERVICES BRANCH

SCAQMD METHOD 306-91

ANALYSIS OF PENTANES IN EXPANDABLE STYRENE POLYMERS

Approved June 1, 1991
Revised February 1993