TEST METHOD

Effective: 28 March 2011 DOWM 102557-E11A
Supersedes: New

Benzyl Alcohol in Low-Density Polyethylene Pellets

1. Scope

This method is applicable to the analysis of benzyl alcohol in low density polyethylene pellets over the range of 0.02 – 25.6 ppm (μg/g).

2. Principle

Polyethylene pellets are soaked in water at 70 °C for one hour. The water is cooled to room temperature and an aliquot of the liquid is analyzed by liquid chromatography where the benzyl alcohol is separated and detected by a UV-Vis absorbance detector. Quantitation is by external standard based on peak area.

3. Safety

Each analyst must be acquainted with the potential hazards of the equipment, reagents, products, solvents and procedures before beginning laboratory work. SOURCES OF INFORMATION INCLUDE: OPERATION MANUALS, MATERIAL SAFETY DATA SHEETS, LITERATURE AND OTHER RELATED DATA. Safety information should be requested from the supplier. Disposal of waste materials, reagents, reactants and solvents must be in compliance with applicable governmental requirements.

4. Interferences

4.1 Water solutions of benzyl alcohol are not stable for more than one day, and must be prepared fresh daily prior to the analysis.

4.2 No other interferences have been observed in the use of this method. If results are suspect based on the analytical history of the product, the data should be confirmed by an alternate technique or method

5. Apparatus

5.1 Liquid Chromatography (LC) System, consisting of the following components: Agilent 1260 Quaternary Pump, Agilent 1260 Autosampler, Agilent 1260 Column Heating Compartment, Agilent 1260 VWD UV/VIS Absorbance Detector and Agilent Chemstation, available from Agilent Technologies, 2850 Centerville Road, Wilmington, DE 19808 (www.agilent.com), or equivalent.

5.2 Liquid chromatographic column: Zorbax SB-C8, 3.5 µm particle, 4.6 x 75 mm, available from Agilent Technologies, or equivalent.

5.3 Autosampler vials and caps for the Agilent 1260 autosampler, available from Agilent Technologies, or equivalent.

5.4 Analytical balance: capable of weighing to the nearest 0.0001-g, Mettler model AE163, available from Mettler-Toledo Inc., P.O. Box 71, 69 Princeton-Hightstown Road, Hightstown, NJ 08520, or equivalent.

5.5 Electronic Digital Pipette capable of accurately delivering 50µL: E3-100, available from Rainin Inc., Box 4026, Woburn MA 01888-4026 (www.rainin.com), or equivalent.

5.6 Volumetric flask: 50 mL, available from Fisher Scientific, or equivalent.

5.7 Volumetric flask: 100 mL, available from Fisher Scientific, or equivalent.

5.8 Beaker: 250 mL, available from Fisher Scientific, or equivalent.

5.9 Aluminum foil, available from Fisher Scientific, or equivalent.

5.10 Oven, capable of maintaining 70C ± 5C, available from Fisher Scientific, or equivalent.

5.11 Transfer pipets, available from Fisher Scientific, or equivalent.

6. **Reagents**

6.1 Benzyl alcohol [CAS# 100-51-6]: available from Sigma-Aldrich, PO Box 14508, St. Louis, MO 63178 (www.sigmaaldrich.com), or equivalent.

Note: The purity of this standard should either be provided by the supplier or determined by the analyst for use in Section 7.3.

6.2 Acetonitrile: HPLC grade, or equivalent, available from Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA 15219, or equivalent.

6.3 High purity water: with 18 megaOhm resistance, prepared by filtering deionized water through any reliable laboratory water purification system, available from Barnstead/Thermolyne Corp., Subs. of Sybron Corp., 2555 Kerper Boulevard, Dubuque, IA 52001, or equivalent.

Note: this is Type I water per ASTM D1193.

7. Reagent Solutions

7.1 Calibration stock solution 1 (100 ppm benzyl alcohol in water):

7.1.1 Weigh (and record to the nearest 0.0001-g) 0.010 ± 0.001 g of benzyl alcohol (Section 6.1) into a 100-mL volumetric flask (Section 5.7).

7.1.2 Dilute the contents of the flask in Section 7.1.1 to volume with purified water (Section 6.3), cap and mix well.

7.2 Calibration stock solution 2 (0.1ppm benzyl alcohol in water):

7.2.1 Transfer 50 µL of Calibration stock solution 1 (Section 7.1.2) using an electronic pipette (Section 5.5) into a 50-mL volumetric flask (Section 5.6).

7.2.2 Dilute the contents of the flask in Section 7.2.1 to volume with purified water (Section 6.3), cap, and mix well.

7.3 Determine the concentration (µg/mL) of benzyl alcohol in the calibration stock solution 2 as follows:

$$C_{BA} = \frac{W_{BA}}{100\,mL} \times \frac{P_{BA}}{100\%} \times \frac{0.05\ mL}{50\ mL} \times \frac{10^6\,\mu g}{1\,g}$$

Where:

C_{BA} = the concentration (µg/mL) of benzyl alcohol in calibration stock solution 2
W_{BA} = weight (g) of benzyl alcohol added to calibration stock solution 1 (Section 7.1.1)
P_{BA} = the % purity of benzyl alcohol in its original standard (Section 6.1)

8. Analysis Conditions

Note: The parameters summarized below were used in the validation of the method. Pressures, flow rates, and integrator parameters will depend on each chromatographic system and may differ from those stated below.

8.1 Liquid Chromatographic Conditions

Instrument: Agilent 1260 Quaternary Pump
Mobile Phase: A: Water
B: Acetonitrile
C: off
D: off

Gradient Program:

Time Min	%A	%B	%C	%D	Flowrate mL/min
0.0	85	15	0	0	1.50
1.0	85	15	0	0	1.50
8.0	70	30	0	0	1.50
9.0	70	30	0	0	1.50

Stop Time: 9 minutes
Post Time 5 minutes

Column:	Zorbax SB-C8
Length:	7.5 cm
Diameter:	4.6 mm
Packing Diameter:	3.5 µm
Column Heater:	not controlled
Autosampler:	Agilent 1260 Autosampler
Injection Volume:	50 µL
Detector:	Agilent 1260 VWD UV/Vis Absorbance Detector
Wavelength:	210 nm
Data Acquisition:	Agilent Chemstation

Representative chromatograms are illustrated in Figure 1.

9. Calibration

9.1 Calibration standard:

9.1.1 Transfer 1.0 mL of calibration stock solution 2 (Section 7.2) using a transfer pipet (Section 5.11) to an autosampler vial (Section 5.3).

9.1.2 Inject a 50-µL aliquot of the calibration standard (Section 9.1.1) into the chromatograph and separate according to the chromatographic conditions outlined in Section 8.

9.2 Calibrate the data system according to the instrument manufacturer's operating instructions for an external standard calibration (if manual calculations are used, proceed to Section 9.3).

9.3 If manual calculations are used, calculate the response factors for (analyte) as follows:

$$Rf_{BA} = \frac{C_{BA}}{Area_{BA}}$$

Where:

Rf_{BA} = the response factor for benzyl alcohol

$Area_{BA}$ = the peak area of benzyl alcohol obtained from the analysis of the calibration standard (Section 9.1.2)

C_{BA} = the concentration (µg/mL) of benzyl alcohol in the calibration standard solution (Section 7.3)

10. Procedure

10.1 **Sample Soaking**

10.1.1 Tare a glass beaker (Section 5.8) on the balance.

10.1.2 Add approximately 10.0 grams of the sample pellets into the beaker and record the weight to the nearest 0.01-g.

10.1.3 Add 10.0 grams of purified water (Section 6.3). Swirl the contents of the beaker.

10.1.4 Loosely cover the beaker with foil (Section 5.9) and place in a 70 °C oven (Section 5.10) for 60 minutes.

10.1.5 Remove the beaker from the oven and allow the beaker to cool to room temperature. An ice-water cooling bath may be used, if necessary.

10.1.6 Transfer an aliquot of the liquid from the beaker to an autosampler vial (Section 5.3) and place on the autosampler (Section 5.1) for analysis.

11. Calculation

If manual calculations are used, calculate the concentration (µg/mL) of benzyl alcohol in the original sample as follows:

$$S_{BA} = Rf_{BA} \times Area_{BA}$$

Where:

S_{BA} = concentration (µg/mL) of benzyl alcohol in the sample
Rf_{BA} = the response factor of benzyl alcohol (Section 9.3)
$Area_{BA}$ = the peak area of benzyl alcohol obtained from the analysis of the sample solution (Section 10.1.6)

12. Precision

Precision has been determined from multiple analyses [n = 20] of two synthetic prepared samples of benzyl alcohol in water. The synthetic samples were prepared fresh daily (Section 4.1). The analyses were performed on two separate days at an average benzyl alcohol concentration [$\overline{x}$] of 0.09 ppm (µg/g). The precision data indicate a standard deviation [s] of 0.01 ppm (µg/g), where s = standard deviation of the validation data.

The estimated prediction interval at the 95% confidence level of a future final result determined on a similar sample [$\pm t_{(n-1)} \times s$; where $t_{(n-1)} = 2.093$ = t-value at (n-1) degrees of freedom] is ± 0.01 ppm (µg/g). This assumes a normal distribution of results and equal variability between locations.

Any future final result obtained on a similar sample (with a true benzyl alcohol concentration [$\overline{x}$] of 0.090 ppm (µg/g)) is expected to range from 0.08 to 0.10 ppm (µg/g) at the 95% confidence level.

The distribution of the results is assumed to be normal. The validity of this assumption has been verified using the Shapiro-Wilk test for normality. The test confirmed that the results could originate from a normal distribution.

13. Accuracy

Analysis of five synthetic mixtures containing approximately 0.1 ppm benzyl alcohol spiked onto pellets gave recoveries that averaged 93% with a range of 73 to 117 % and a standard deviation of 17%. Recoveries were based on the nominal concentration of benzyl alcohol in the spiked samples.

14. Linearity

Detector response was found to be linear over the concentration range of 0.024 – 25.6 ppm (μg/g).

15. Limit of Detection/Limit of Quantitation

The limit of detection (LOD), defined as the lowest detectable concentration, was estimated to be 0.013 ppm (μg/g). The limit of quantitation (LOQ), defined as 3.3 times the LOD, was determined to be 0.046 ppm (μg/g).

16. Reporting

Record results to two decimal places in your laboratory information management system (LIMS). For product release, report Pass or Fail and identify if the result is above or below the upper specification limit.

For example: Pass ≤ 0.1 ppm or Fail > 0.1ppm

17. Notes

17.1. The accuracy of balances and pipettes should be confirmed on a regular basis and documentation of the check should be kept.

17.2. This method was validated using automated injections but could also be performed using manual injections. In either case, it is recommended that the precision, accuracy, linearity, limit of detection and limit of quantitation of the method be verified if another set of equipment is to be used or the method is to be used at another location.

17.3. Because LC systems have different dwell volumes, gradients may need to be adjusted so that the method delivers the same performance as the validated method.

Figure 1. A representative chromatogram of benzyl alcohol obtained using the conditions outlined in Section 8 of this method.

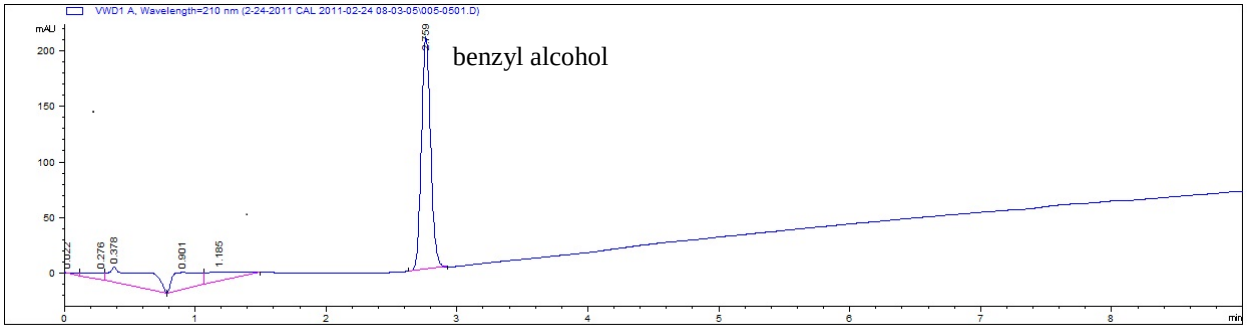
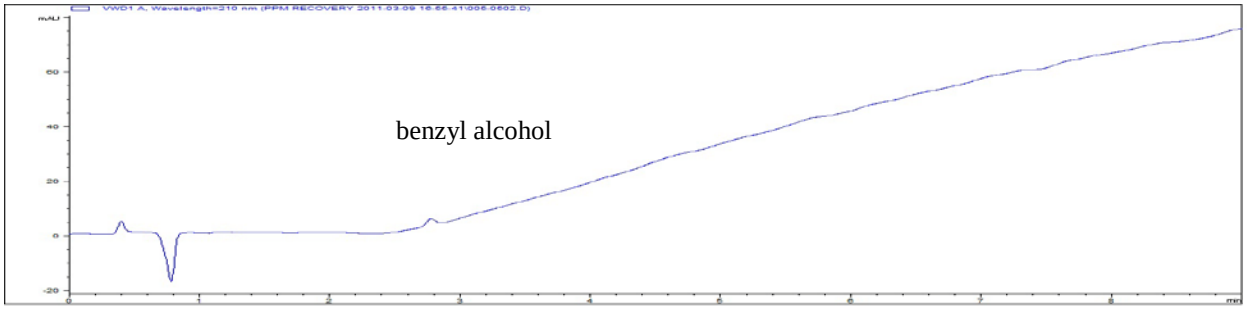


Figure 2. A representative chromatogram of approximately 0.1 ppm benzyl alcohol obtained using the conditions outlined in Section 8 of this method.



Expanded view of benzyl alcohol region at 0.1ppm concentration