TP SA-094
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Residual Benzene Content
Applicable Product: Carbopol®* 934P NF Polymer

Scope:

This procedure is for the analysis of the residual benzene level in Carbopol® 934P NF polymer by head space-gas chromatography.

Note: This method is only applicable to Carbopol® 934P NF polymer. See product specifications for the appropriate method to determine benzene content in other products.

Abstract:

Benzene is released from a sample of the polymer by shaking with a 16.6% v/v dimethyl sulfoxide (DSMO) in water mixture. One mL of the headspace is injected onto a gas chromatograph column and the components separated. The response of the benzene peak as detected by flame ionization is compared to the responses of prepared standards to determine the concentration in the polymer.

Safety Precautions:

1. Wear safety goggles and gloves and follow good laboratory practices.
2. Polymer dust is irritating to the respiratory passages and inhalation should be avoided.
3. Dimethyl sulfoxide (DMSO) is combustible and may be harmful if inhaled or absorbed through the skin.
4. Benzene is a flammable liquid and a known carcinogen.
5. See Material Safety Data Sheet (MSDS) for additional safety and handling information.

Interferences:

Any component eluting at the same retention time as benzene would influence the results. These interferences could be present in the sample or the DMSO used to extract. Isopropyl acetate, iso-octane and cyclohexene elute at similar times as benzene. Demonstrating these chemicals do not co-elute with benzene on the analytical column used under the oven conditions and flow rates employed should be accomplished prior to performing analyses.

Apparatus:

1. Gas chromatograph with capillary column capability and flame ionization detector.
2. Headspace sampler.
3. Data processing station.
4. Restek RTX-1301 column, 30 meter x 0.53 mm with 3 µm film thickness.
5. Mechanical shaker.
6. 22 mL headspace vial.
7. Teflon-lined butyl rubber septum with crimp top for headspace vials.
8. 50 µl syringe.
9. Manual aluminum seal crimper tool.
10. Analytical balance capable of ±0.0001 g accuracy.
11. 2 ml auto-sampler vials with Teflon-lined screw cap.
12. One liter volumetric flask.
13. 10 mL graduated pipette.
14. Pipette bulb.
15. Graduated cylinder.

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Reagents:

1. Dimethyl sulfoxide (DMSO).
2. HPLC water or similar solvent free water.
3. Benzene.

GC Conditions:

Prior to the analysis of samples or standards the following chromatographic conditions should be set:

Detector Temperature	250°C
Injection Port Temperature	140°C
Oven Conditions	
Initial Temperature	40°C
Initial Time	10 minutes
Ramp Rate	30°C/min
Final Temperature	240°C
Final Time	5 minutes
Flow	5 ml/min (35 cm/sec velocity) Programmed in constant pressure mode
Split Ratio	1:1

Headspace Parameters:

Equilibrium Time	45 minutes
Equilibration Temperature	80°C
Transfer Line Temperature	105°C
Valve Oven Temperature	105°C
Vial Pressure	10 psig
Loop Fill Pressure	7 psig
Injection Volume	1 ml
Mixer Option (If equipped)	Off

Calibration:

1. Add 1.8 mL DMSO to a tared 2 ml GC autosampler vial and record the weight. Place the Teflon-lined screw cap on the vial.
2. Using a 50 µl syringe, add 25 µl benzene through the septum. Record the weight of benzene added.
3. Label the vial **Primary Standard**.
4. Add 1 mL DMSO to a tared 2 mL autosampler vial and record the weight. Place the Teflon-lined screw cap on the vial.
5. Tare the vial, add 20 µl of the Primary Standard and record the weight of the Primary Standard added.
6. Label the vial **Secondary Standard**.
7. Prepare three working standards and a blank in 22 mL vials as follows:

   Weigh 0.0500 ± 0.0010 g polymer to be tested in a 22 mL headspace vial. Record the weight.
8. Add 6 ml of 16.6% v/v DMSO in water to one of the vials form step 7 (See Special Instruction 1 for preparation of 16.6% v/v DMSO).
9. Tare the vial. Using a 50 µl syringe, add 10 µl of the Secondary Standard to the vial and record the weight.
10. Place a Teflon-lined butyl rubber septum on the vial and seal with an aluminum crimp cap. Label the vial as **Working Standard 1**.
11. Shake for one hour on a mechanical shaker.
12. Repeat steps 8 through 11, adding 20 µl and 30 µl of the Secondary Standard to a second and third vial from Step 7. Label as **Working Standard 2** and **Working Standard 3**.
13. Prepare the fourth vial from step 7 as in steps 8 through 11 but without the addition of the secondary standard. Label as **Blank**.
14. Shake for one hour on a mechanical shaker.

Working Standard Preparation Summary

	Polymer (g ± 0.0010)	16.6% v/v DMSO in water (ml)	Secondary Standard (µl)
Working Standard 1	0.0500	6	10
Working Standard 2	0.0500	6	20
Working Standard 3	0.0500	6	30
Blank	0.0500	6	0

15. Set instrument up according to operational parameters and analyze the samples on the gas chromatograph.

Sample Procedure:

1. Add 50 mg (0.05 g) of sample to a tared 22 ml headspace vial. Record the weight to the nearest 0.0001 g.
2. Dispense 6 ml 16.6% v/v DMSO/H_2O mixture to the vial, seal with septum and cap.
3. Shake for one hour on a mechanical shaker.
4. Place vial in headspace sampler and analyze following the same operational parameters as for the standard.
5. At the conclusion of standards and samples, calculate mg/kg (ppm) benzene in the samples. (See Calculations).

Calculations:

A data processing system can be programmed to calculate results of samples.

If a data system with data processing capability is not available, the following calculations can be performed to yield results as mg/kg (ppm) benzene.

1. Calculation of mg/kg benzene in standards:

   Primary Standard:

   Conc. of Primary Std. (g/g) = A/B

   A = Weight of benzene (g)
   B = Total weight of Primary Std. (g)
   (combined wt. of benzene + DMSO)

   Secondary Standard:

   Concentration of Secondary Std. (g/g) =
   (C/D)*Concentration of Primary Std.

   C = Weight Primary Std. (g)
   D = Total weight Secondary Std. (g)

   Working Standard:

   Concentration of Working Std. (mg/kg) =
   E*F*(1,000,000/0.05)

   E = Weight Secondary Std. (g)
   F = Concentration of Sec. Std. (g/g)
   0.05 = Sample weight (g)

2. Calculation of response factor for benzene:

   RF = Working Std. (mg/kg) / (G-H)

   G = Working Std. benzene peak area
   H = Blank peak area (see Note 1)

3. Repeat calculations for the remaining Working Standards. Calculate the average of the three response factors.

4. Calculation for mg/kg benzene in sample:

Benzene (mg/kg) = Average RF * Sample peak area

Special Instructions:

1. 16.6% DMSO: To a 1 liter volumetric flask, add 166 mL DMSO. Dilute to the mark with HPLC water and mix.

Notes:

1. Subtracting the peak area of the blank corrects for the response of the benzene in the sample, yielding the response for the standard.

References:

- *Current edition of the United States Pharmacopeia <467>*