METHOD 310C - DETERMINATION OF RESIDUAL N-HEXANE IN EPDM RUBBER THROUGH GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for the determination of residual hexane in EPDM wet crumb rubber in the 0.01 - 2% range by solvent extraction of the hexane followed by gas chromatographic analysis where the hexane is detected by flame ionization and quantified via an internal standard.

1.2 This method may involve hazardous materials operations and equipment. This method does not purport to address all the safety problems associated with it use, if any. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2.0 SUMMARY

2.1   Residual hexane contained in wet pieces of EPDM polymer is extracted with MIBK. A known amount of an internal standard (IS) is added to the extract which is subsequently analyzed via gas chromatography where the hexane and IS are separated and detected utilizing a megabore column and flame ionization detection (FID). From the response to the hexane and the IS, the amount of hexane in the EPDM polymer is calculated.

3.0 DEFINITIONS

3.1 Hexane - refers to n-hexane

3.2 Heptane - refers to n-heptane

3.3 MIBK - methyl isobutyl ketone (4 methyl 2 - Pentanone)

4.0 INTERFERENCES

4.1 Material eluting at or near the hexane and/or the IS will cause erroneous results. Prior to extraction, solvent blanks must be analyzed to confirm the absence of interfering peaks.

5.0 SAFETY

5.1   Review Material Safety Data Sheets of the chemicals used in this method.

6.0   EQUIPMENT AND SUPPLIES

6.1   4 oz round glass jar with a wide mouth screw cap lid.

6.2   Vacuum oven.

6.3   50 ml pipettes.

6.4   A gas chromatograph with an auto sampler and a 50 meter, 0.53 ID, methyl silicone column with 5 micron phase thickness.

6.5   Shaker, large enough to hold 10, 4 oz. jars.

6.6   1000 and 4000 ml volumetric flasks.

6.7   Electronic integrator or equivalent data system.

6.8   GC autosampler vials.

6.9   50 uL syringe.

7.0   REAGENTS AND STANDARDS

7.1   Reagent grade Methyl-Iso-Butyl-Ketone (MIBK)

7.2   n-heptane, 99% + purity

7.3   n-hexane, 99% + purity

8.0   SAMPLE COLLECTION

8.1   Trap a sample of the EPDM crumb slurry in the sampling apparatus. Allow the crumb slurry to circulate through the sampling apparatus for 5 minutes; then close off the values at the bottom and top of the sampling apparatus, trapping the crumb slurry. Run cooling water through the water jacket for a minimum of 30 minutes. Expel the cooled crumb slurry into a sample catching basket. If the crumb does not fall by gravity, force it out with demineralized water or nitrogen. Send the crumb slurry to the lab for analysis.

9.0   QUALITY CONTROL

9.1   The Royalene crumb sample is extracted three times with MIBK containing an internal standard. The hexane from each extraction is added together to obtain a total hexane content. The percent hexane in the first extraction is then calculated and used as the recovery factor for the analysis.

9.2   Follow this test method through section 11.4 of the method. After removing the sample of the first extraction to be

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run on the gas chromatograph, drain off the remainder of the extraction solvent, retaining the crumb sample in the sample jar. Rinse the crumb with demineralized water to remove any MIBK left on the surface of the crumb. Repeat the extraction procedure with fresh MIBK with internal standard two more times.

9.3 After the third extraction, proceed to section 11.5 of this method and obtain the percent hexane in each extraction. Use the sample weight obtained in section 12.1 of this method to calculate the percent hexane in each of the extracts.

9.4 Add the percent hexane obtained from the three extractions for a total percent hexane in the sample.

9.5 Use the following equations to determine the recovery factor (R):

% Recovery of the first extraction = (% hexane in the first extract/total % hexane)X100

Recovery Factor (R) = (% Hexane Recovered in the first extract)/100

10.0 CALIBRATION

10.1 Preparation of Internal Standard (IS) solution:

Accuracy weigh 30 grams of n-heptane into a 1000 ml volumetric flask. Dilute to the mark with reagent grade MIBK. Label this Solution "A". Pipette 100 mls. of Solution A into a 4 liter volumetric flask. Fill the flask to the mark with reagent MIBK. Label this Solution "B". Solution "B" will have a concentration of 0.75 mg/ml of heptane.

10.2 Preparation of Hexane Standard Solution (HS):

Using a 50 uL syringe, weigh by difference, 20 mg of n-hexane into a 50 ml volumetric flask containing approximately 40 ml of Solution B. Fill the flask to the mark with Solution B and mix well.

10.3 Conditions for GC analysis of standards and samples:

Temperature: Initial   = 40°C
              Final   = 150°C
              Injector   = 160°C
              Detector   = 280°C
              Program Rate   = 5.0°C/min

Initial Time = 5 minutes   Final Time = 6 minutes

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Flow Rate = 5.0 ml/min
Sensitivity = detector response must be adjusted to
keep the hexane and IS on scale.
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10.4 Fill an autosampler vial with the HS, analyze it three times and calculate a Hexane Relative Response Factor (RF) as follows:

$$RF = (A_{IS} \times C_{HS} \times P_{HS})/(A_{HS} \times C_{IS} \times P_{IS}) \qquad (1)$$

Where:
A_{IS}= Area of IS peak (Heptane)
A_{HS}= Area of peak (Hexane Standard)
C_{HS}= Mg of Hexane/50 ml HS
C_{IS}= Mg of Heptane/50 ml IS Solution B
P_{IS}= Purity of the IS n-heptane
P_{HS}= Purity of the HS n-hexane

11.0 PROCEDURE

11.1 Weight 10 grams of wet crumb into a tared (W1), wide mouth 4 oz. jar.

11.2 Pipette 50 ml of Solution B into the jar with the wet crumb rubber.

11.3 Screw the cap on tightly and place it on a shaker for 4 hours.

11.4 Remove the sample from the shaker and fill an autosampler vial with the MIBK extract.

11.5 Analyze the sample two times.

11.6 Analyze the HS twice, followed by the samples. Inject the HS twice at the end of each 10 samples or at the end of the run.

12.0 CALCULATIONS

12.1 Drain off the remainder of the MIBK extract from the polymer in the 4 oz. jar. Retain all the polymer in the jar. Place the uncovered jar and polymer in a heated vacuum oven until the polymer is dry. Reweigh the jar and polymer (W2) and calculate the dried sample weight of the polymer as follows:

$$\text{Dried SW} = W2 - W1 \qquad (2)$$

12.2 Should the polymer be oil extended, pipette 10 ml of the MIBK extract into a tared evaporating dish (W1) and evaporate to dryness on a steam plate.

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Reweigh the evaporating dish containing the extracted oil (W2). Calculate the oil content of the polymer as follows:

Gram of oil extracted = 5 (W2 – W1)   (3)

% Hexane in polymer = $(A_s \times RF \times C_{IS} \times P_{IS})/(A_{IS} \times SW)$ (4)

Where:

A_s = Area of sample hexane sample peak.
A_{IS} = Area of IS peak in sample.
C_{IS} = Concentration of IS in 50 ml.
P_{IS} = Purity of IS.
SW = Weight of dried rubber after extraction. (For oil extended polymer, the amount of oil extracted is added to the dry rubber weight).

% Corrected Hexane = (% Hexane in Polymer)/R   (5)

R = Recovery factor determined in section 9 of this method.

13.0 METHOD PERFORMANCE

13.1 Performance must be determined for each sample type by following the procedures in section 9 of this method.

14.0 WASTE GENERATION

14.1 Waste generation should be minimized where possible.

15.0 WASTE MANAGEMENT

15.1 All waste shall be handled in accordance with Federal and State environmental regulations.

16.0 REFERENCES

(Reserved)