METHOD 310A - DETERMINATION OF RESIDUAL HEXANE THROUGH GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 This method is used to analyze any crumb rubber or water samples for residual hexane content.

1.2 The sample is heated in a sealed bottle with an internal standard and the vapor is analyzed by gas chromatography.

2.0 SUMMARY OF METHOD

2.1 This method, utilizing a capillary column gas chromatograph with a flame ionization detector, determines the concentration of residual hexane in rubber crumb samples.

3.0 DEFINITIONS

3.1 The definitions are included in the text as needed.

4.0 INTERFERENCES

4.1 There are no known interferences.

5.0 SAFETY

5.1 It is the responsibility of the user of this procedure to establish safety and health practices applicable to their specific operation.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph with a flame ionization detector and data handling station equipped with a capillary column 30 meters long.

6.2 Chromatograph conditions for Sigma 1:

6.2.1 Helium pressure: 50# inlet A, 14# aux

6.2.2 Carrier flow: 25 cc/min

6.2.3 Range switch: 100x

6.2.4 DB: 1 capillary column

6.3 Chromatograph conditions for Hewlett-Packard GC:

6.3.1 Initial temperature: 40°C

6.3.2 Initial time: 8 min

6.3.3 Rate: 0

6.3.4 Range: 2

6.3.5 DB: 1705 capillary column

6.4 Septum bottles and stoppers

6.5 Gas Syringe - 0.5 cc

7.0 REAGENTS AND STANDARDS

7.1 Chloroform, 99.9+%, A.S.C. HPLC grade

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 A representative sample should be caught in a clean 8 oz. container with a secure lid.

8.2 The container should be labeled with sample identification, date and time.

9.0 QUALITY CONTROL

9.1 The instrument is calibrated by injecting calibration solution (Section 10.2 of this method) five times.

9.2 The retention time for components of interest and relative response of monomer to the internal standard is determined.

9.3 Recovery efficiency must be determined once for each sample type and whenever modifications are made to the method.

9.3.1 Determine the percent hexane in three separate dried rubber crumb samples.

9.3.2 Weigh a portion of each crumb sample into separate sample bottles and add a known amount of hexane (10 microliters) by microliter syringe and 20

microliters of internal standard. Analyze each by the described procedure and calculate the percent recovery of the known added hexane.

9.3.3 Repeat the previous step using twice the hexane level (20 microliters), analyze and calculate the percent recovery of the known added hexane.

9.3.4 Set up two additional sets of samples using 10 microliters and 20 microliters of hexane as before, but add an amount of water equal to the dry crumb used. Analyze and calculate percent recovery to show the effect of free water on the results obtained.

9.3.5 A value of R between 0.70 and 1.30 is acceptable.

9.3.6 R shall be used to correct all reported results for each compound by dividing the measured results of each compound by the R for that compound for the same sample type.

10.0 CALIBRATION AND INSTRUMENT SETTINGS

10.1 Calibrate the chromatograph using a standard made by injecting 10 µl of fresh hexane and 20 µl of chloroform into a sealed septum bottle. This standard will be 0.6 wt.% ***total*** hexane based on 1 gram of dry rubber.

10.2 Analyze the hexane used and calculate the percentage of each hexane isomer (2-methylpentane, 3-methylpentane, n-hexane, and methylcyclo-pentane). Enter these percentages into the method calibration table.

10.3 Heat the standard bottle for 30 minutes in a 105°C oven.

10.4 Inject about 0.25 cc of vapor into the gas chromatograph and after the analysis is finished, calibrate according to the procedures described by the instrument manufacturer.

11.0 PROCEDURE

11.1 Using a cold mill set at a wide roller gap (125-150 mm), mill about 250 grams of crumb two times to homogenize the sample.

11.2 Weigh about 2 grams of wet crumb into a septum bottle

and cap with a septum ring. Add 20 µl of chloroform with a syringe and place in a 105°C oven for 45 minutes.

11.3 Run the moisture content on a separate portion of the sample and calculate the grams of dry rubber put into the septum bottle.

11.4 Set up the data station on the required method and enter the dry rubber weight in the sample weight field.

11.5 Inject a 0.25 cc vapor sample into the chromatograph and push the start button.

11.6 At the end of the analysis, the data station will print a report listing the concentration of each identified component.

11.7 To analyze water samples, pipet 5 ml of sample into the septum bottle, cap and add 20 µl of chloroform. Place in a 105°C oven for 30 minutes.

11.8 Enter 5 grams into the sample weight field.

11.9 Inject a 0.25 cc vapor sample into the chromatograph and push the start button.

11.10 At the end of the analysis, the data station will print a report listing the concentration of each identified component.

12.0 DATA ANALYSIS AND CALCULATION

12.1 For samples that are prepared as in section 11 of this method, ppm n-hexane is read directly from the computer.

12.2 The formulas for calculation of the results are as follows:

$$ppm_{hexane} = (A_{hexane} x R_{hexane}) / (A_{is} x R_{is})$$

where: A_{hexane} = area of hexane
R_{hexane} = response of hexane
A_{is} = area of the internal standard
R_{is} = response of the internal standard

$$\% \text{ hexane in crumb} = (\text{ppm}_{\text{hexane}}/\text{sample amount})100$$

12.3 Correct the results by the value of R (as determined in sections 9.3.4, 9.3.5, and 9.3.6 of this method).

13.0 METHOD PERFORMANCE

13.1 The test has a standard deviation of 0.14 wt% at 0.66 wt% hexane. Spike recovery of 12 samples at two levels of hexane averaged 102.3%. Note: Recovery must be determined for each type of sample. The values given here are meant to be examples of method performance.

14.0 POLLUTION PREVENTION

14.1 Waste generation should be minimized where possible. Sample size should be an amount necessary to adequately run the analysis.

15.0 WASTE MANAGEMENT

15.1 All waste shall be handled in accordance with federal and state environmental regulations.

16.0 REFERENCES AND PUBLICATIONS

16.1 DSM Copolymer Test Method T-3380.