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Application News

Fourier Transform Infrared Spectrophotometer IRSpirit™-X

Silicone Quantitation from Siliconized Rubber Stoppers by Using FTIR

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User Benefits

- The present work provides an easy extraction method for fast and accurate quantitation of silicone.
- The methodology can be adapted for extractable and leachable study of materials like pre-filled syringes, medical tubings and various drug delivery systems.

■ Introduction

Silicones are extensively employed in various industries due to their unique properties. In the pharmaceutical industry, they find numerous applications such as active ingredients, antifoaming agents, and excipients. They are also utilized in siliconization, pharmaceutical manufacturing operations, and packaging materials. Siliconization plays a vital role in the production of pre-fillable syringes, cartridges and rubber stoppers. Its purpose is to lubricate between the components which facilitates smooth gliding. It also enables a secure connection between the components like glass bottle and rubber stopper. By carefully optimizing the siliconization process parameters, it is possible to achieve a uniform coating while minimizing the presence of free silicone. To obtain quantitative information on the siliconization layers, it is essential to extract the silicone from siliconized components using organic solvents and then perform FTIR spectroscopy. This technique can provide crucial insights into the amount of silicone used on to the rubber stoppers. The present work demonstrates the application of FTIR for quantification of silicone in rubber stoppers.

■ Experimental

For the current study, the siliconized and non-siliconized rubber stoppers were collected from one of the manufacturers. Calibration curve was prepared by using USP reference standard Polydimethylsiloxane (PDMS). The measurements were performed on IRSpirit-ZX (Figure 1). The instrumental parameters are given in Table 1. The parameters used for measurement are referred from United States Pharmacopea[1)] .

Sample preparation

50 number of siliconized and non-siliconized rubber stoppers were kept in 250 mL beaker and 100 mL of Hexane was added to each beaker. The mixture was rapidly agitated for about one minute. It was then poured into a 100 mL beaker. At 90 °C, the content was evaporated on a water bath. After evaporation, the beaker was cooled to room temperature and 10 mL of Hexane was added to dissolve the residue. The solution was then centrifuged for 10 min at 7800 rpm and supernatant was employed for measurement. The sample preparation was performed in duplicate.

Standard preparation

5000 ppm of PDMS : Weighed accurately 250 mg of USP reference standard Polydimethylsiloxane into a 50 mL volumetric flask, added approximately 20 ml of Hexane, stirred vigorously for approximately 5 minutes and diluted up to the mark with the Hexane. Table 2 represents the concentration of calibrations standards used in present study.

Figure 2 represents graph of standard calibration curve. Figure 3 and 4 represents overlay spectra of calibration standards and samples, respectively. Peak height at 1259 cm^{-1} was used for the quantitation.

Spiked sample preparation

50 number of siliconized and non-siliconized rubber stoppers were kept in 250 mL beaker , 2 mL of 5000 ppm PDMS and 100 mL of Hexane was added to each beaker. The mixture was rapidly agitated for about one minute. It was then poured into a 100 mL beaker. At 90 °C, the content was evaporated on a water bath. After evaporation, the beaker was cooled to room temperature and 10 mL of Hexane was added to dissolve the residue. The solution was then centrifuged for 10 min at 7800 rpm and supernatant was employed for measurement. The sample preparation was performed in duplicate.

Figure 1. IRSpirit™-ZX

Table 1: Analytical conditions

Instrument	IRSpirit-ZX spectrophotometer
Measurement Cell	0.1 mm NaCl Fixed Thickness Cell
Resolution	4 cm^{-1}
Number of scans	45
Apodization function	Happ-Genzel
Detector	DLATGS
Measurement wavelength range	1250-1270 cm^{-1}

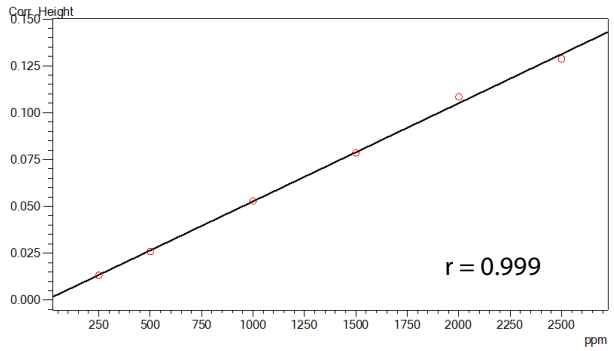

Figure 2. Graph of standard calibration curve

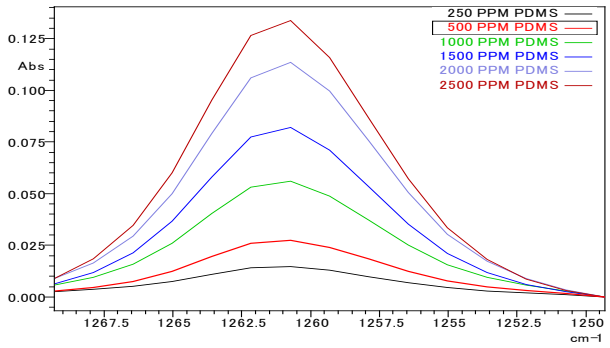

Figure 3. Overlaid spectra of standard calibration levels

Table 2: Concentrations of calibration standards

Volume of 5000 ppm PDMS (ml)	Diluted to (ml)	Conc of PDMS (ppm)
0.5	10	250
1.0	10	500
2.0	10	1000
3.0	10	1500
4.0	10	2000
5.0	10	2500

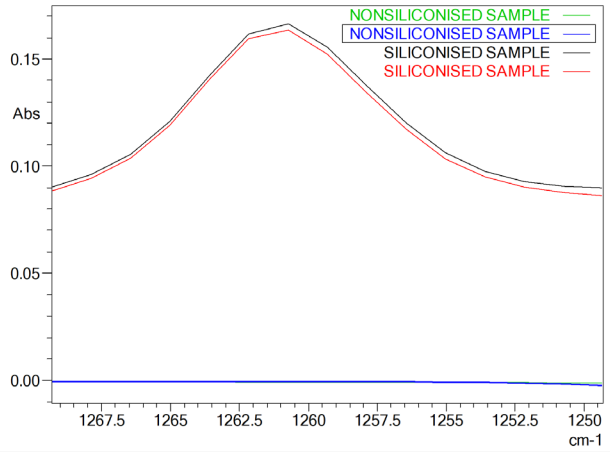

Figure 4. Overlaid spectra of siliconized and non-siliconized samples

■ Results & Discussion

Table 3 represents the amount of Silicone per unit of rubber stopper (Average of two measurement).

Table 3: Quantitation results obtained in present study

Sample	X	Y	N	µg per unit stopper
Siliconized	1421	10	50	284.2
Nonsiliconized	BCCL	10	50	BCCL*

* BCCL: Below calibration curve concentration i.e., 250 ppm

The average amount of silicone per unit of rubber stopper was determined by the following formula.

Amount of silicone per part of rubber = X *Y/N

X = Concentration obtained in ppm

Y = Volume of Hexane used to redissolve the residue in mL

N = Number of rubber stopper taken for analysis

The siliconized and non-siliconized samples were spiked with 1000 ppm PDMS standard to check the accuracy of the extraction method. The % recovery obtained is given in Table 4. The recoveries obtained were between 80 to 120 %.

Table 4: Recovery results obtained in present study

Sample Name	% recovery at 1000 ppm
Siliconized	82.1
Non-siliconized	90.7

■ Conclusion

Different grades of rubber samples were analyzed by FTIR using fixed thickness cell of NaCl, and excellent repeatability was obtained. The use of Hexane for extraction proved to be effective method for extraction of silicone from rubber stoppers. The % recovery obtained demonstrates the accuracy and reliability of the developed methodology. The method can be used for the routine analysis of rubber stopper samples to measure the content of silicone.

■ Reference

1) USP monograph of Dimethicone

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