Specac

Application Note

Quantifying Simeticone according to European Pharmacopoeia Standard 10.0 (01/2017:1470) using the Specac Pearl™

The Specac Pearl™

INSIDE: Learn how the Specac Pearl™ can be used to quantify the percentage content of Simeticone in your tablets using a toluene-acid extraction.

Introduction

Determination of the API (active pharmaceutical incipient) in a finished pharmaceutical product is critical to ensure the patient receives a safe dosage. FTIR is a rapid, low cost technique that can give quantitative measurements in seconds to the QC analyst.

Simeticone is used for the treatment of flatulence, trapped wind and colic in babies. The European Pharmacopoeia Standard 10.0 (01/2017:1470) [1] sets out a method for the use of an FTIR transmission measurement to determine the % content of a sample, using a toluene extraction method. Here we demonstrate that the Pearl™ is fully compliant with this standard and more broadly a great choice for the quantitative analysis of pharmaceutical products.

The Pearl™ is a revolutionary liquid transmission FTIR accessory. The magic of the Pearl™ stems from the horizontal Oyster™ cell at its core. Unlike a tradition vertical cell which can suffer from leaks and poor pathlength reproducibility when the cell is dismantled the Pearl™ is designed to be leak free and have a reproducibility greater than 1 µm when the cell is disassembled and reassembled. This allows for significantly faster sample throughput. A boon to any lab with high volumes of samples to test!

- **Highly reproducible pathlength**
  Change pathlengths in seconds
- **Spacer-free, defined pathlength liquid transmission cell**
  ZnSe or CaF_2 windows
  Wedged or Parallel
  Pathlengths from 25 to 1000 µm
- **Handles viscous materials with ease**
  Great choice for large volumes of samples

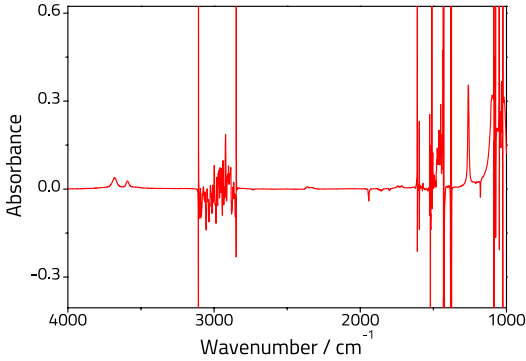

Figure 1: *FTIR Spectrum of Simeticone in Toluene recorded against a Toluene background.*

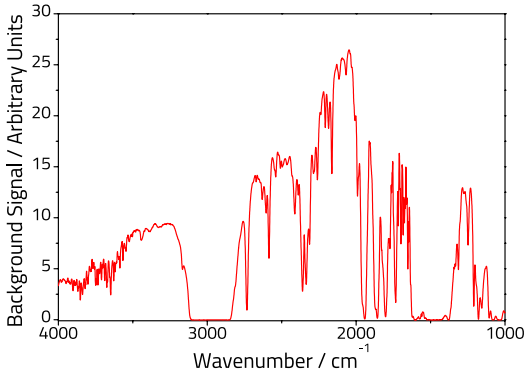

Figure 2: *Background FTIR spectrum of Toluene.*

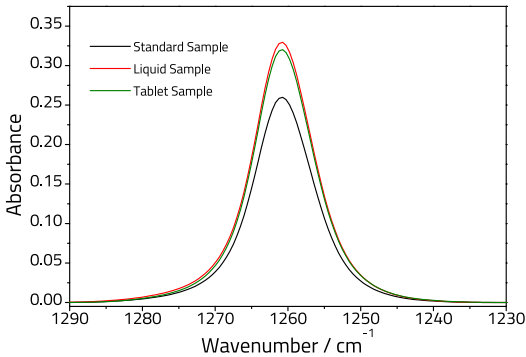

Figure 3: *FTIR Spectra showing the CH_3 deformation vibration of simeticone, for 3 different formulations.*

Experimental

Test and reference samples containing *ca.* 100 mg of simeticone were dissolved in 50 ml toluene, and then stirred for 5 min with 100 ml dilute HCl. The organic layer was separated and 5 ml extracted and dried over 1 g anhydrous sodium sulfate. The resultant liquid was then centrifuged using a home built centrifuge as described in [2]. Three solutions were prepared, 1 from a dimethicone standard sample (the reference solution), and two from a liquid simeticone solution and the other from a tablet form (the test solutions), both obtained from a local pharmacy.

FTIR spectra were recorded on a commercially available spectrometer using a Specac Pearl™, fitted with a 500 micron wedged CaF_2 Oyster Cell.

Results and Discussion

Figure 1 shows the spectrum of simeticone in toluene against a toluene background, and is a good illustration of the impact of using a solvent spectrum as the background. Nonsense data is observed in the regions *ca.* 3140-2800, 1650-1350 & 1200-1000 cm^{-1}. An inspection of the background spectrum of toluene (Figure 2) reveals that in these regions the solvent is totally absorbing and no light reaches the detector. It is standard practice either to zoom into the solvent-free window (as shown in Figure 3), or to blank out the regions of nonsense and explain in the figure caption that regions obscured by the solvent have been hidden for clarity.

The European Pharmacopoeia Standard specifies the use of a peak at 1260 cm^{-1} for the quantification of simeticone. This peak is assigned to the symmetric deformation vibration of the methyl group [3] and is shown in Figure 3. From the absorbance values at 1260 cm^{-1} the experimentally determined % composition of each of the test samples was determined using the formula:

$$\%Content_{Test} = \frac{V_{Test} \times C_{Ref} \times Abs_{Test} \times 100}{Abs_{Ref} \times M_{Test}}$$

where:

Abs_{Test} = Absorbance of the test sample at 1260 cm^{-1}

Abs_{Ref} = Absorbance of the reference sample at 1260 cm^{-1}

C_{Ref} = Concentration of the reference solution (mg/ml)

V_{Test} = Total Volume of Toluene in test solution (ml)

M_{Test} = Mass of test sample (mg)

Table 1 shows the Nominal percentage simeticone in each sample, alongside the Absorbance recorded for the solutions prepared from each and the simeticone content as determined by FTIR. In both cases nominal and calculated are within the measurement error from the mass of the sample alone, confirming that both pharmaceutical products match the label.

Sample I.D.	Nominal Simeticone Mass	Nominal Simeticone %	Abs_T	Calculated Simeticone
Liquid Sample	120 mg	4 ± 0.3 %	0.326	4.3
Tablet Sample	125 mg	72 ± 0.2 %	0.317	72.1

Table 1: Nominal and spectroscopically determined simeticone content.

Conclusion

In conclusion, the Pearl™ and Oyster™ system is fully compliant with the European Pharmocopoeia standard for the determination of simeticone content. The Pearl is an excellent choice for the pharmacuetical industry QC lab owing to its high sample throughput capability. Existing methods can be rapidly deployed without modfication or training.

References

[1] Simeticone - European Pharmocopoeia 10.0, 01/2017:1470

[2] Bhalma et al. Nature Biomedical Engineering, 1, 0009, DOI: 10.1038/s41551-016-0009

[3] Infrared and Raman Characteristic Group Frequencies, Socrates, G., John Wiley & Sons, London, Third Edition, 2001, Chapter 18, ISBN: 0470093072

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