Application note

Streamlined FTIR analysis of dimethicone with OMNIC Paradigm Workflow

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Thermo Scientific Nicolet Apex FTIR Spectrometer.

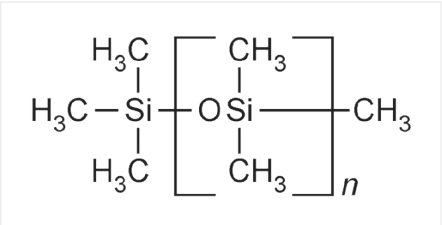

Figure 1. Chemical structure of dimethicone.

Fourier transform infrared spectroscopy (FTIR) has become an indispensable analytical tool in modern chemical analysis. FTIR measures the absorption of infrared radiation by a sample, thereby providing valuable information about the sample's molecular structure and chemical composition. FTIR analysis has long been an integral part in quality assurance (QA) and quality control (QC) in many industries, from pharmaceuticals, food & beverages, and cosmetics to polymers and other chemicals. which helps ensure product quality, safety, and regulatory compliance. Typical QA/QC applications of FTIR include raw material identification, product authentication, confirmation of batch-to-batch consistency, contaminant detection, formulation analysis, and stability testing.

QA/QC analysts and other scientists in many of today's analytical laboratories are often tasked with a wide range of assays that employ multiple techniques from chromatography to spectroscopy. It is therefore highly desirable that analytical instruments offer fit-for-purpose workflows, powered by easy-to-operate hardware and intuitive and flexible software, to streamline and automate assays.

In this application note, we demonstrate a quantitative analysis by FTIR streamlined by an OMNIC Paradigm Workflow, using dimethicone as an example. Dimethicone, also known as polydimethylsiloxane (PDMS), is a silicone-based polymer that is extensively used in personal care products, pharmaceutical formulations, medical devices, foods and beverages, and many other industrial applications. The structure of dimethicone (Figure 1) consists of repeating dimethyl siloxane units $[-(CH_3)_2SiO-]_n$ stabilized with trimethylsiloxy end-blocking units $[(CH_3)_3SiO-]$.

According to the United State Pharmacopeia (USP), the attenuated total reflectance (ATR) FTIR method is employed for the quantitative analysis of dimethyl siloxane in dimethicone[1]. Figure 2 is a representative ATR FTIR spectrum of dimethicone. The experiments were performed using a Thermo Scientific™ Nicolet™ Apex FTIR Spectrometer configured with a diamond ATR accessory. All spectra were collected with a spectral resolution of 4 cm^{-1} and co-addition of 40 scans. The average data acquisition time is less than 40 s per spectrum.

Dimethicone exhibits IR peaks at 789–796 cm^{-1} (Si–CH_3 asymmetric rock), a double peak between 1,007–1,074 cm^{-1} (asymmetric Si-O-Si stretch and bend), a narrow sharp peak at ~ 1,258 cm^{-1} (CH_3 symmetric bend), and 2,950–2,960 cm^{-1} (asymmetric CH_3 stretch).

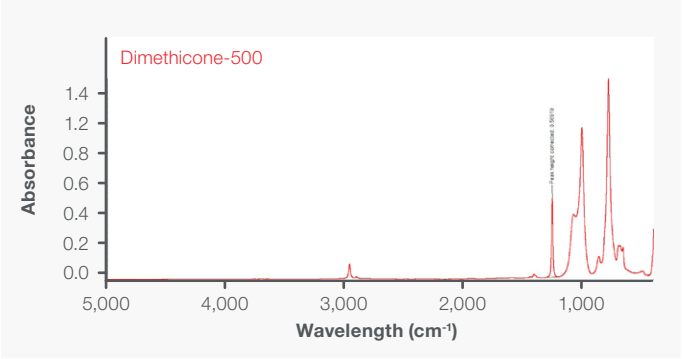

Figure 2. Representative ATR FTIR spectrum of a dimethicone reference standard.

$$100 \frac{A_u}{A_s} \frac{D_s}{D_u}$$

Formula 1.

The peak at 1,258 cm^{-1} is used for the quantitative determination of the percent of dimethyl siloxane in dimethicone using formula 1, in which A_U is the absorbance of the dimethicone; A_S is the absorbance of polydimethylsiloxane reference standard; and D_S and D_U are the specific gravities of polydimethylsiloxane reference standard and dimethicone, respectively.

An OMNIC Paradigm Workflow (Figure 3) was created to streamline this quantitative analysis. The workflow includes the reference standard and sample information input (ID and specific gravity), background and sample measurements using predefined experimental parameters, peak height measurement of the resulting spectra, wt.% calculation based on the USP schema, and the final report. Five replicate measurements were performed on a reference standard and a commercial sample, and the results are summarized in Table 1. Good repeatability was obtained for both samples, with a coefficient of variation (CV) 0.07% for the reference standard and 0.27% for the commercial sample.

Sample	Reference Sample	Unknown
1	0.56429	0.47024
2	0.56544	0.46908
3	0.56484	0.46865
4	0.56491	0.46965
5	0.56470	0.47191
Average	0.56484	0.46991
Standard Deviation	0.000414403	0.00127013
CV%	0.07	0.27

Table 1. Peak height at 1,258 cm^{-1}.

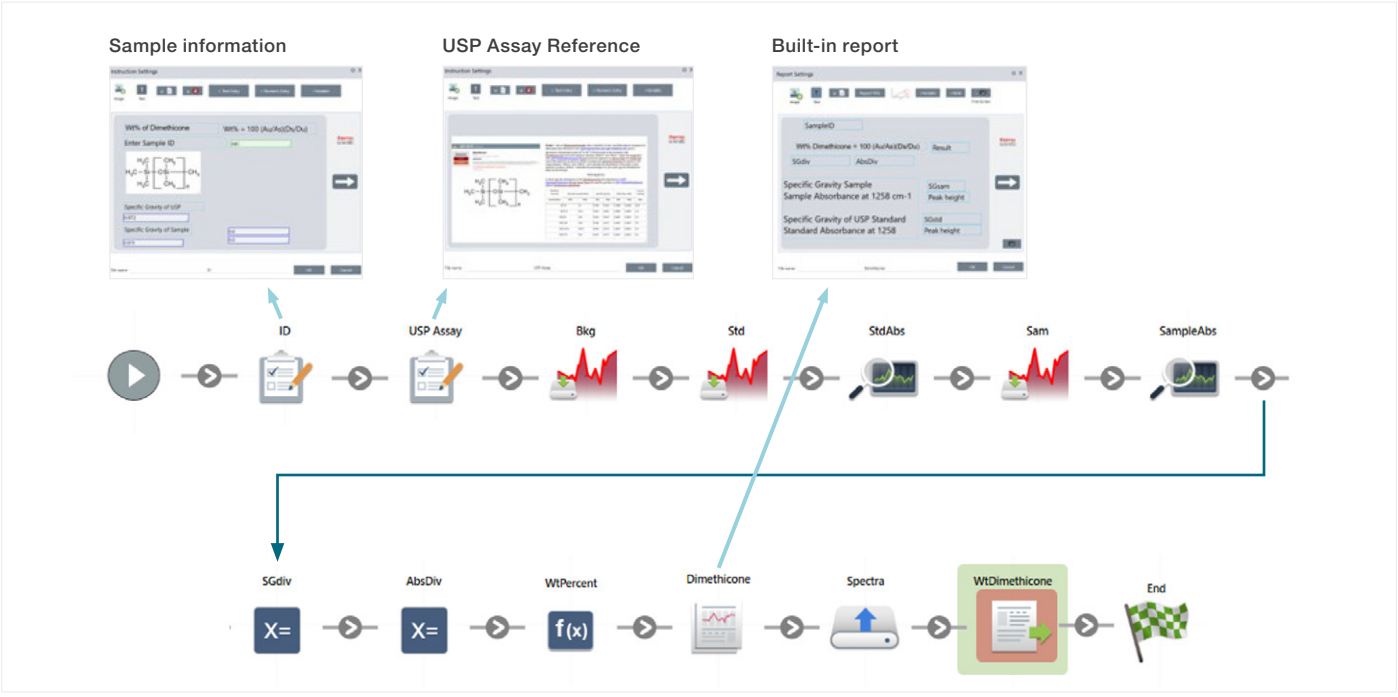

Figure 3. OMNIC Paradigm Workflow for the quantitative analysis of dimethicone.

powered by

Thermo Scientific Nicolet Apex FTIR Spectrometer (left) and Thermo Scientific Nicolet Summit X FTIR Spectrometer (right) powered by Thermo Scientific OMNIC Paradigm Software.

Conclusion

In this application note we have described the quantitative analysis of dimethicone by ATR FTIR, defined by the US Pharmacopeia, using an OMNIC Paradigm Workflow. The ATR FTIR method is simple and straightforward with no sample preparation. The replicate experiments show good repeatability for both the reference standard and the commercial sample. The quantitative analysis is streamlined by using an OMNIC Paradigm Workflow, which greatly simplifies an otherwise tedious and repetitive analytical task while minimizing operator intervention to ensure consistent and unbiased analyses. The principles demonstrated thereof are applicable for many QA/QC analyses by FTIR.

Reference

1. USPC Official Monographs: Dimethicone

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