# Analysis of Spices for Steam Volatile Oils using EQTA Near Infrared Methodology

A EUROFINS WHITE PAPER

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**This paper describes a fast, accurate, and simple technique for analysis of spices using Fourier Transform Near Infrared (FT-NIR) technology.**

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## Steam Volatile Oil (SVO) in Spices

Steam volatile oil is an important property of spices. The volatile components present in spices contribute to the aroma and flavor of the spice. Spice oils are commonly extracted by water solvent methods (ie, steam distillation). Spice oils are used in many industries such as food and beverage, pharmaceutical, chemical and aromatherapy. The demand of spice oils is increasing significantly in developing countries, primarily due to the fast food and snack industries.

## How are spices traditionally analyzed for SVO?

The standard laboratory approach to obtaining the SVO value from a spice sample is to distill the oil using a reflux apparatus. The spice sample is added to approximately 500 mL of water which is heated to distill the oil. Other solvents may be used if the sample does not readily distill using water alone. The volume or weight of the isolated oil is divided by the total sample weight to obtain the SVO percent. This "wet chemistry" approach suffers from the following disadvantages:

- Long analysis time: Analysis may take several hours to complete.
- Technical training: The method requires technical training to perform the test with high accuracy.
- Resources and consumables: The wet chemistry approach may use solvents (such as xylene other reagents) that can be costly and have high toxicity to operators and the environment.
- Method Error: The method error can be high. The error of one commonly used method, American Spice Trade Association (ASTA) Method 5.0 (Modified Clevenger Method) has a relative standard deviation of ±14% according to a round robin study using a black pepper sample. (http://www.astaspice.org)

## EQTA NIR Approach to SVO analysis:

Eurofins Quality Trait Analysis (EQTA) is a total package solution that employs Near-Infrared (NIR) technology for fast, convenient, and accurate analysis of a wide range of products and materials. Some key aspects of the EQTA approach are described below:

- Fourier Transform instrumentation: Analysis is performed with a Fourier Transform Near Infrared (FT-NIR) spectrometer; this instrument has the highest wavelength accuracy and is considered the gold standard for NIR analytical chemistry applications. The instrument provided by EQTA gives the customer the best possible data quality that can be generated using a NIR measurement technique. It is also flexible – the sampling presentation and measurement parameters can be tailored to the application. The FT instrument is not only high performance but also rugged and suitable for an industrial plant environment.
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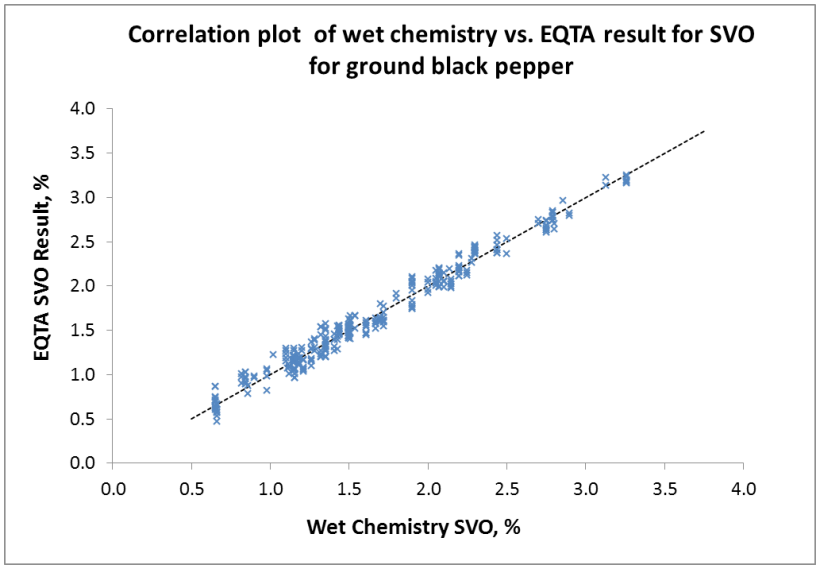
- Ease of operation: a non-skilled operator can perform the analysis. The sample of spice is simply placed in a vial or a cup then placed in a sample holder for analysis.
- Speed: Results are returned within 2-3 minutes of starting the analysis.
- Reliability: The NIR analysis utilizes chemometric algorithms. These algorithms are based on a large database of samples, comprised of many different varieties of spice. More varieties are added on a continual basis, improving the robustness reliability of the algorithm over time.
- Consistency: ETQA algorithms use special methods that have been developed by expert chemometricians. The EQTA patented "Chingometrics" employed in the algorithms allow for the same algorithm to be used by multiple instruments at multiple locations without the need for slope/bias adjustment. In this way, product consistency can be checked across labs at multiple sites.
- EQTA internet-based analysis: In order to maintain the highest quality algorithms, they are kept on a central server and only able to be modified by expert chemometricians. This also ensures consistency across multiple sites that use the same algorithm. When the user analyzes a sample, the measured spectrum is sent to a central server via the internet. At the central server, the chemometric algorithm computes the received spectrum and produces the final result of SVO. That result is sent back to the user and displayed on their interface screen. The whole process takes only 2-3 minutes. All results are compiled and stored in a database, accessible to the user at any time.
- Method Error: EQTA is considered a secondary method because it utilizes wet chemistry data as reference values for the SVO calibration. However, the error of the EQTA analysis often matches or is very close to the most accurate wet chemistry analysis. In general, there is not a significant loss in accuracy when switching from wet chemistry analysis to EQTA. For certain applications, the consistency of NIR analysis can be superior to wet chemistry. The correlation plot below shows the accuracy of EQTA analysis compared to the wet chemistry value.

A useful tool for visualizing a calibration is a correlation plot. In the correlation plot below, wet chemistry values for SVO are plotted on the x-axis. The EQTA predicted values are plotted on the y-axis. There is very strong correlation, indicating that the EQTA result is a good match to the wet chemistry value.




- Instrument maintenance: NIR spectrometers require minimal maintenance. Occasionally the desiccant needs to be exchanged and every few years, the light source or laser will be swapped. The EQTA support team can talk the customer through any necessary maintenance steps, or visit the customer if necessary.
- Support: All aspects of instrument maintenance, computer interface, results interpretation, etc. are provided to the customer. The service is designed to be a complete package so the customer can just "plug and play" the instrument right out of the box. The customer does not need any science or technical background to use the EQTA instrument.
- Other Testing Capabilities and ASTA check sample program: NIR can analyze many other traits such as moisture, ash, color, water activity, piperine, Scoville heat. EQTA has participated in the ASTA check sample program since 2016 for black pepper (SVO, moisture, piperine, water activity), oregano (SVO, moisture, ash, acid Insoluble ash), and red pepper (Scoville heat, water activity, and color). The plot below shows the ETQA results for black pepper SVO in terms of the Z score. The Z scores for QTA are very low, indicating excellent performance.

Below is a plot of EQTA Z scores for SVO in ground black pepper for all quarters which QTA submitted results. The closer the Z score is to zero, the closer the result is to the mean of all participating labs. Z scores are close to zero for EQTA, indicating excellent performance