Application Note

Cosmetics, Biocides

Agilent

Trusted Answers

Ensuring Biocidal Effectiveness and Safety in Handwash Formulations by FTIR

Effortless and reliable phenoxyethanol quantification using the Agilent Cary 630 FTIR spectrometer

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Abstract

Manufacturers of personal-care products must ensure that they meet the required specifications to guarantee user safety and maintain compliance with quality control standards. This study outlines the development of a method for the rapid quantification of phenoxyethanol content in a biocidal handwash formulation, using the Agilent Cary 630 FTIR spectrometer equipped with a single reflection diamond Attenuated Total Reflectance (ATR) module. The quantitative method was evaluated for accuracy, precision, and selectivity/specificity, confirming its suitability for the routine analysis of phenoxyethanol in biocidal handwash.

Introduction

Phenoxyethanol is a commonly used preservative in cosmetic products and can be used as an adjunctive biocidal agent in biocidal formulations. Quantifying the amount of this material in a finished formulation is important to ensure a product's effectiveness and safety for consumer use. The analysis is usually done using an HPLC method, but FTIR offers an alternative approach with the following benefits:

- **Quicker to perform:** Sample measurement only takes seconds compared to 30 minutes for the HPLC method, perfect for a production laboratory QC setting where time is always an important factor.
- **Cost-effective:** Eliminates the need for sample preparation, reducing the potential for errors, time to produce results, and the cost per analysis.
- **Simpler:** The instrumentation is easier to maintain, requires no consumables, and operational qualification (OQ) checks can be performed in-house.
- **More-affordable:** Lower initial instrument capital costs and significantly lower running costs.

This application note describes an FTIR based analytical method developed using the Agilent Cary 630 FTIR spectrometer (see Figure 1) to quantify phenoxyethanol in a biocidal handwash containing 5% phenoxyethanol. Details of the method validation procedures are also included.

Figure 1. The Agilent Cary 630 FTIR spectrometer with diamond ATR module is ideal for the fast and easy quantification of phenoxyethanol in handwash products.

Experimental

Instrumentation

An Agilent Cary 630 FTIR spectrometer equipped with a Single Reflection Diamond ATR module was used for this application. Measurement parameters are shown in Table 1.

Table 1. Experimental parameters for the Agilent Cary 630 FTIR-ATR.

Measurement Mode	Absorbance
Spectral Range (cm^{-1})	4000–650
Number of Background/Sample Scans	32
Spectral Resolution (cm^{-1})	8
Approx. Measurement Time per Sample (s)	10

The Cary 630 FTIR combines robustness, flexibility, and high performance in an ultracompact design, making it suitable for routine analysis. The Cary 630 FTIR can be quickly reconfigured with a sampling module, requiring no user alignment. For ease-of-use, the Agilent MicroLab software provides step-by-step guidance with instructive pictures to intuitively navigate users through the entire analytical workflow. The software uses a method-based approach that allows users to create methods for identification, as well as for quantification. Results are displayed in an easy-to-understand format, using color-coding of the results based on customizable limits to ensure quick and intuitive data review (Figure 2).

Sample preparation

Standard samples of biocidal handwash were formulated in the laboratory to contain exactly 2, 3, 4, 5, and 6% (w/w) phenoxyethanol.

The specification for the finished product is 4.75 to 5.25% w/w phenoxyethanol at point of manufacture. Phenoxyethanol is no longer soluble when the concentration exceeds 6%, so 6% w/w was selected as the highest concentration for the standard set.

Figure 2. Using Agilent MicroLab software and Agilent FTIR spectrometers, the analysis can be performed in three simple steps, streamlining the method and minimizing training requirements.

Results and discussion

Data quality

Data quality was assessed through a visual inspection of the instrument response to the analyte in the sample matrix. As shown by the spectra in the MicroLab Quant application (see Figure 3), the peak corresponding to the analyte is well defined and free of interference from adjoining peaks, allowing quantitative measurement of the analyte based on peak area.

Calibration linearity

An analytical range is described as linear when there is a direct proportional relationship between the instrument response and concentration of the analyte in the sample matrix over the desired range. Linearity refers to how well test results correlate with concentrations over a given range.

Using the MicroLab Quant application, a simple Beer–Lambert law based on peak area (as shown in Figure 3) was used to instantly produce a calibration curve—a plot of instrument response versus actual concentration. The calibration curve displayed excellent linearity with a correlation coefficient (R^2) greater than 0.997 (Figure 4).

Calibration curve

Each of the five standards of biocidal handwash was measured in triplicate. A plot of the peak area vs concentration level yielded a straight line with an R^2 value of 0.99764 (Figure 4). This high correlation coefficient demonstrates sufficient linearity to quantitatively assess unknown samples within the lower and upper concentration ranges. The software automatically generates the calibration curves and calculates the correlation coefficients.

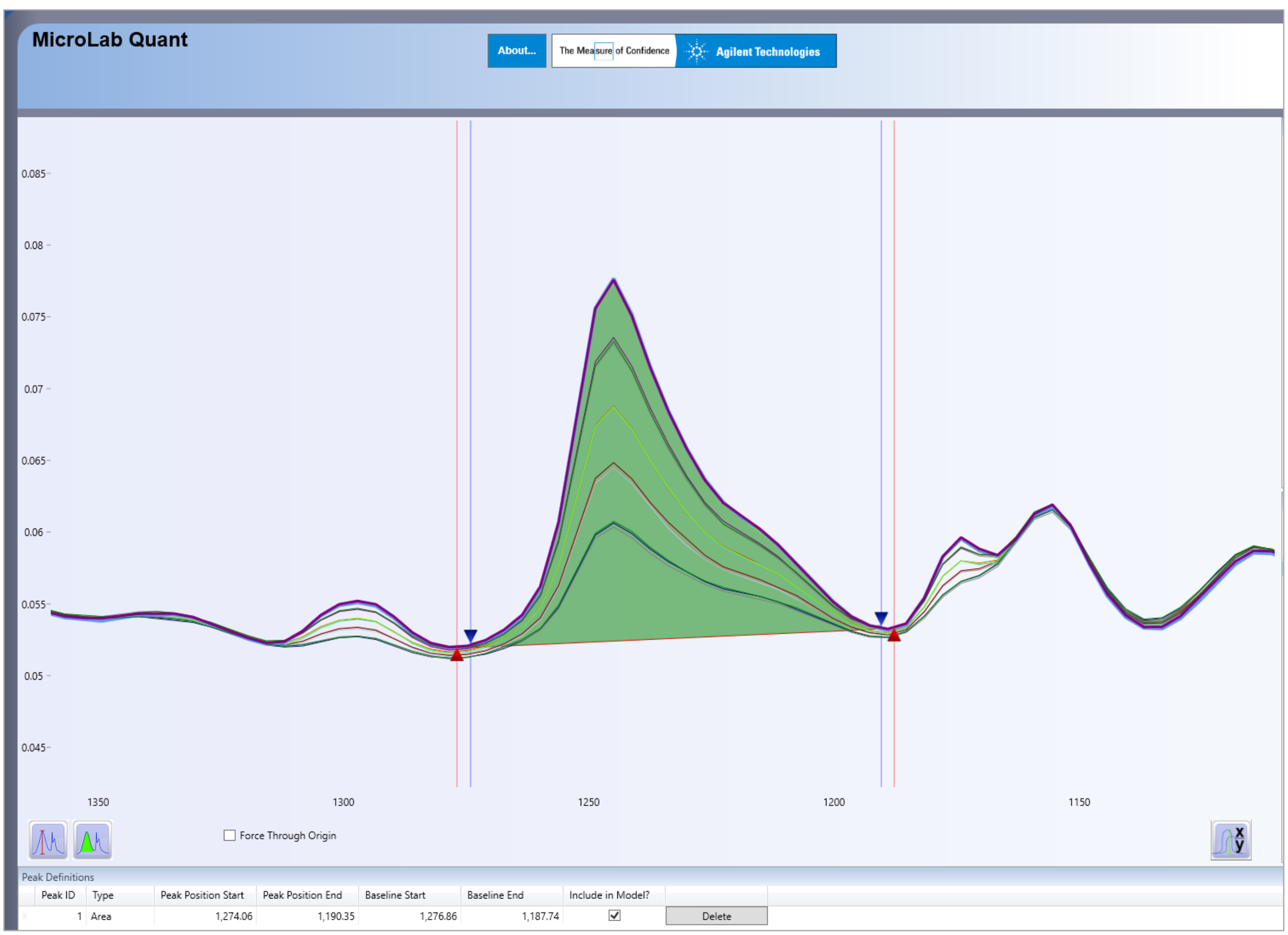

Figure 3. The Agilent MicroLab Quant application allows identifing the most suitable band for analyte quantification, enabling the optimization of analysis parameters such as peak position, and peak start and end points. The band at 1250 cm^{-1} for phenoxyethanol is distinct, making it suitable for calibration curve generation.

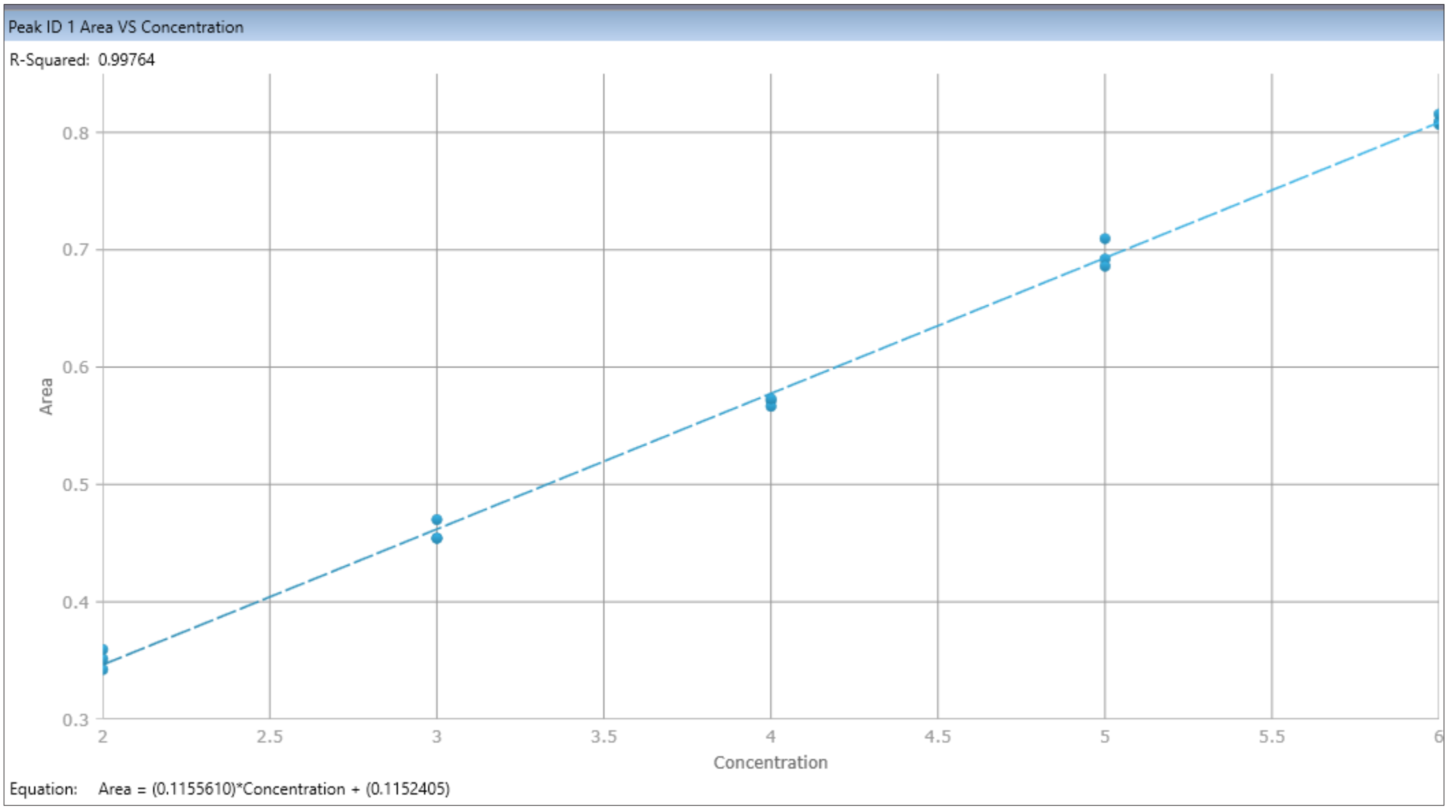

Figure 4. The Agilent MicroLab Quant application was used to generate a calibration curve and to evaluate the linearity of the FTIR spectral response. The peak definition from Figure 3 for phenoxyethanol was used to generate the calibration curve, based on the analysis of the five standards of biocidal handwash.

Accuracy/recovery

The accuracy of an analytical procedure is defined as the closeness of agreement between the true value, or an accepted reference value, and the measured value. Accuracy should be determined over the entire range of quantification. The percentage recoveries of known analyte concentrations in a spiked sample matrix are a typical measure of accuracy.

The amount of analyte present in each spiked sample based on the instrument response should be compared to the amount of analyte added to each sample. Accuracy/recovery is calculated as the percentage of analyte grams recovered from the analyte grams spiked. For the target 5% analyte concentration, the mean accuracy/recovery level should be within 97 to 103% of the theoretical spiked analyte content.

Table 2 shows the accuracy/recovery of the FTIR method for samples of biocidal handwash created with phenoxyethanol concentrations from 2 to 6% w/w (increasing in intervals of 1% w/w). Each sample was measured in triplicate, and the accuracy/recovery was calculated using the mean value. All recoveries were within 100 ± 3%, confirming the accuracy of the method.

Table 2. Theoretical and mean measured content of phenoxyethanol in biocidal handwash samples, demonstrating the accuracy and recovery of the analytical method.

Theoretical Phenoxyethanol Content (% w/w)	Mean Measured Phenoxyethanol Content (% w/w)	Mean Accuracy/ Recovery (%)
2.00	2.06	102.77
3.00	2.98	99.26
4.00	3.93	98.33
5.00	5.03	100.54
6.00	6.01	100.24

Precision (inter-operator)

Precision is the closeness or agreement between a series of measurements of the same sample or set of samples. Inter-operator precision may be assessed by having additional analysts prepare and analyze separate sets of six surrogate solutions at 100% of the target concentration. Test results obtained by different analysts using different instruments on different days should still meet a precision of ≤ 2% RSD.

For verification of precision, a batch of biocidal handwash containing 5% w/w phenoxyethanol was analyzed using the newly created FTIR method by another laboratory staff member. This process was repeated six times by a different analyst using various instruments on separate days. The results demonstrated excellent precision with an RSD of ≤ 1% (Table 3).

Table 3. Phenoxyethanol levels in six biocidal handwash samples analyzed by a different analyst using various instruments on separate days, ensuring method reliability and accuracy.

Sample Number	Phenoxyethanol Content (% w/w)
1	4.96
2	4.96
3	4.84
4	5.00
5	4.92
6	4.94
Mean	**4.94**
Standard Deviation	**0.049**
% RSD (Precision)	**0.99**

Selectivity and specificity

The selectivity/specificity of a method is determined by comparing the instrument response of a blank sample matrix (containing no analyte) to the instrument response of the blank sample matrix spiked with a known amount of the analyte. Ideally, the blank sample matrix should not create an instrument response that interferes with the instrument response to the analyte.

A sample of biocidal handwash was prepared without phenoxyethanol. When this sample was analyzed using the phenoxyethanol quantification method, it returned a result of 0%. The result, which is shown in Figure 5, indicates that the method is specific.

The quantitative Cary 630 FTIR-ATR method exhibits excellent linearity, accuracies/recoveries, precision, and selectivity/specificity, highlighting its suitability for the analysis of phenoxyethanol.

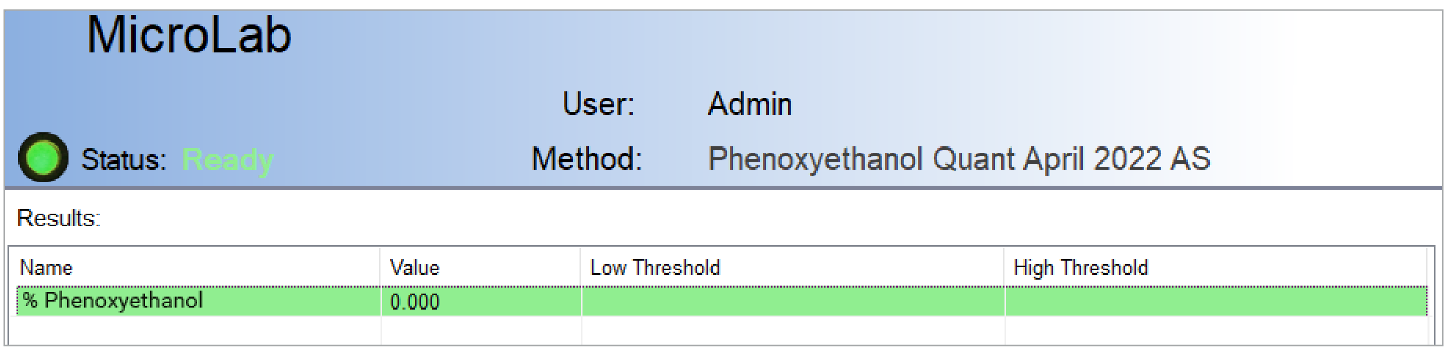

Figure 5. The result for a blank sample matrix containing 0% phenoxyethanol shows no matrix interference, demonstrating the method's specificity.

Routine analysis of biocidal handwash batches

Following a successful evaluation, the quantification model was integrated into a MicroLab method and pass/fail criteria were established based on acceptance criteria. The results are then color-coded accordingly to facilitate easy data analysis and interpretation, as shown in Figure 6. The Cary 630 FTIR method has been implemented in the SC Johnson Professional QC laboratories for the QC of 5% phenoxyethanol in biocidal handwash batches.

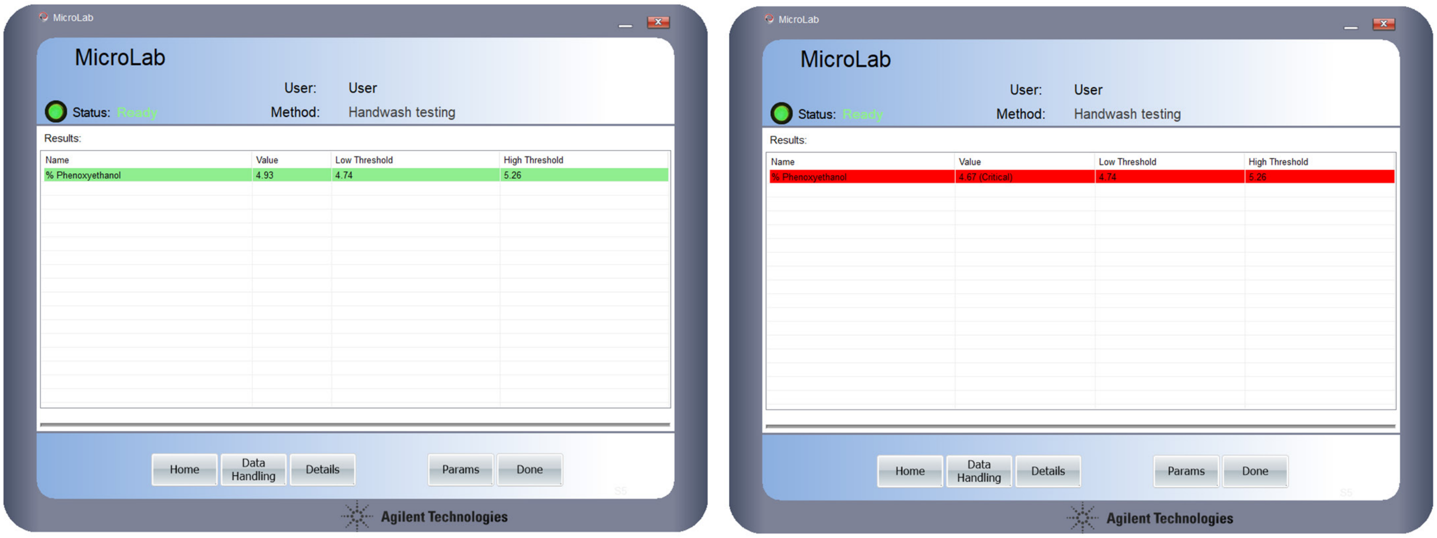

Figure 6. The integration of the Agilent Cary 630 FTIR-ATR quantification model into the Agilent MicroLab software method simplifies routine analysis of biocidal handwash batches. The software determines percentage concentration and provides color-coded, actionable results after data acquisition, highlighting in-spec samples in green and out-of-spec samples in red.

Conclusion

An analytical method for the rapid quantification of phenoxyethanol in biocidal handwash formulation was created and validated using the Agilent Cary 630 FTIR spectrometer equipped with a diamond ATR module.

The method was found to be specific, linear, accurate, and precise. The Cary 630 FTIR is now being used in the SC Johnson Professional QC laboratories as part of the manufacturing process for a biocidal handwash formulation containing 5% phenoxyethanol.

The FTIR method is significantly faster than the previous HPLC method that it has replaced. It is also much simpler for laboratory technicians to perform, requires no solvent, and produces no chemical waste. The easy-to-understand color-coding of the results removes the risk of data misinterpretation, making the Cary 630 FTIR the ideal tool for pass/fail testing in the QC lab.

Further information

- Agilent Cary 630 FTIR Spectrometer
- Agilent MicroLab Software
- Agilent MicroLab Expert Software
- FTIR Analysis & Applications Guide
- FTIR Spectroscopy Basics FAQs

www.agilent.com/chem/cary630

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