APPLICATION NOTE

Gas Chromatography

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Analysis of Hand Sanitizers to Support Label Claims

Introduction

Worldwide, nations continue to face the challenges of the COVID-19 pandemic, and critical personal protective equipment (PPE) supplies, such as alcohol-based hand sanitizers, are in high demand. At the height of the first wave of the pandemic, hand sanitizers were very difficult to obtain, and many stores had little to no supplies for consumers. To fill the rising demand of alcohol-based hand sanitizer, lower grades of ethanol and isopropanol (IPA) started to appear on the market. Several cases of methanol poisonings were reported, thus the need for improved quality control and testing emerged.

As new hand sanitizer products began to enter the market, the US Food and Drug Administration (FDA) released a statement warning consumers and healthcare providers to avoid methanol and 1-propanol containing products, owing to toxicity and the ability of these compounds to be absorbed through the skin[1]. The Unites States Pharmacopeia (USP) responded quickly with *Excerpted USP-NF and FCC Standards: A Hand Sanitizer Resource,* a collection of standards provided as a resource to assist with the challenges posed by COVID-19[2].

This application note outlines a fast, robust and accurate solution for the quantification of ethanol and IPA in hand sanitizers for label claim purposes.

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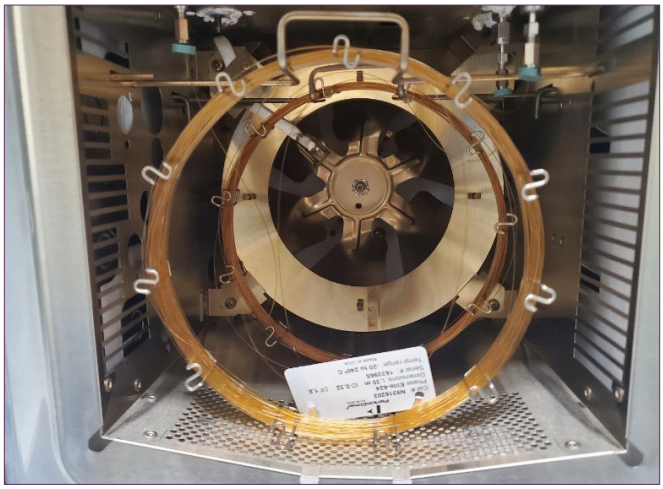

Figure 1. Dual columns installed in Clarus 690 GC Oven.

Instrumentation

A PerkinElmer Clarus® 690 gas chromatograph (GC) with integrated liquid autosampler was configured with a capillary (split/splitless) injector and two flame ionization detectors (FID). PerkinElmer Elite 624 and BAC-1 columns were installed in the injector via a two-hole ferrule (a "Y" splitter may also be used), as shown in Figure 1.

A single column approach is also acceptable for the work contained herein supporting label accuracy claims. However, the power of the dual column approach is needed if the laboratory is required to analyze incoming raw materials, such as ethanol and isopropanol, for impurities using the prescribed USP procedure, or to test for impurities in the final product. The dual column approach is required to confirm the presence of benzene in ethanol, and in ethanol-containing hand sanitizers. The GC configuration described herein is configured to run analyses for both label claim accuracy and ethanol/isopropanol impurities (USP). The GC conditions used for the analyses are listed in Table 1.

Experimental

Chromatography

The GC parameters were optimized for quick elution of the alcohol to enhance sample throughput and laboratory efficiency. After the alcohols eluted, the GC oven was rapidly elevated in temperature to clean the column from any other materials from the hand sanitizer that may have been injected.

Sample Preparation

As the concentration of alcohol is high, the samples should be diluted. The recommended dilution, which was used in these experiments, is 1 to 400. The workflow document suggests procedures for dilution. Another benefit of this dilution is that the system remains cleaner, allowing for less maintenance and enhanced instrument uptime which allows the laboratory to be at the highest capacity producing results and shipping product.

Table 1. GC method parameters.

Parameter	Setting
Columns	Elite 624 Sil ms (G43): 30 m x 320 µm x 1.8 µm
	Elite BAC-1: 30 m x 320 µm x 1.8 µm
Carrier Gas	Helium (other carriers may be used)
Linear Column Velocity	50 cm/sec
Autosampler	
Injection Volume	0.2 µL using a 0.5 µL syringe
Injection Speed	Slow
Number of Pumps	8
Pre-sample Washes	4
Post-solvent Washes	0
Viscosity Delay	10
Oven Parameters	40 °C hold for 0.5 min, ramp to 100 °C @ 20 °C/min, ramp to 230 °C @ 70 °C/min no hold
Capillary Injector Temperature	175 °C
Detector (FID) Temperature	200 °C

Standard Preparation

Analytical grade ethanol and isopropanol were both added to DI water equivalent to a sample concentration range of 55 - 90% at the 1:400 dilution level. Since the lab needs to check for possible adulterants, it is also possible to add other targets, such as methanol and 1-propanol, for screening purposes.

Repeatability

To determine method and instrument precision, 10 injections were made on the mid-point standard of 70 % concentration.

Table 2. Standard preparation reflecting a 1:400 sample dilution.

Sample Concentration (Volume %)	Standard Amount Reflecting Sample Dilution (µL/L)
55%	1375 ppm
70%	1750 ppm
90%	2250 ppm

Samples

Five (5) different hand sanitizers were purchased from stores in various locations for testing.

Data Acquisition

Data acquisition and processing were completed utilizing PerkinElmer TotalChrom chromatography data system software.

Results and Discussion

Chromatography

The chromatograms from both columns obtained using the conditions in Table 1 are displayed in Figure 2. If label claim is only required and not impurities, only one column and one FID is needed. The BAC 1 column is preferred.

The GC method was optimized for separation and speed. The result was a fast 5.36 minute runtime, including column cleanup. This fast analysis time will allow quick sample turnaround and optimized sample throughput, enabling quick decision making on the production floor and quicker release of product.

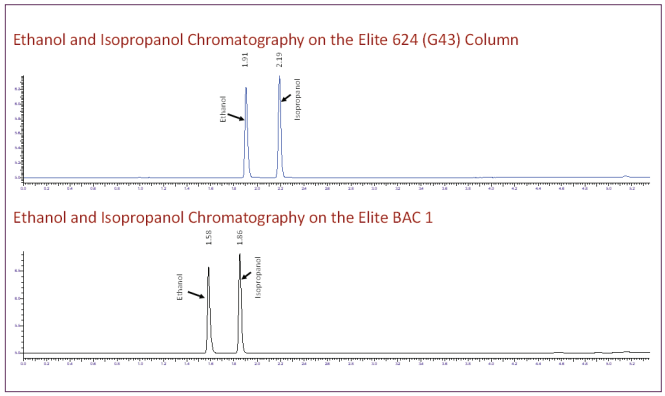

Figure 2. Chromatograms displaying two alcohols on the Elite 624 (top) and BAC 1 columns (bottom).

Calibration

A calibration curve was created in the range from 55 to 90% using both ethanol and isopropanol. Samples and standards were injected using the integrated liquid autosampler at a 0.2 µL injection volume. The standard calibration curves of both ethanol and isopropanol on both columns are displayed in Figures 3a and 3b (ethanol) and Figures 4a and 4b (isopropanol), demonstrating excellent linearity.

Accurate and repeatable results are paramount in the analysis of both ethanol and isopropanol label claims. Table 3 demonstrates excellent repeatability of 10 consecutive replicates of 70% ethanol/ isopropanol at 1:400 dilution level. The percent relative standard deviation (% RSD) was calculated from the raw area counts.

Table 3. Precision results on raw area counts from 10 consecutive injections.

Component	Precision (% RSD) n=10	
	624 (G43) Column	BAC-1 Column
Ethanol	0.90%	0.65%
Isopropanol	0.54%	0.48%

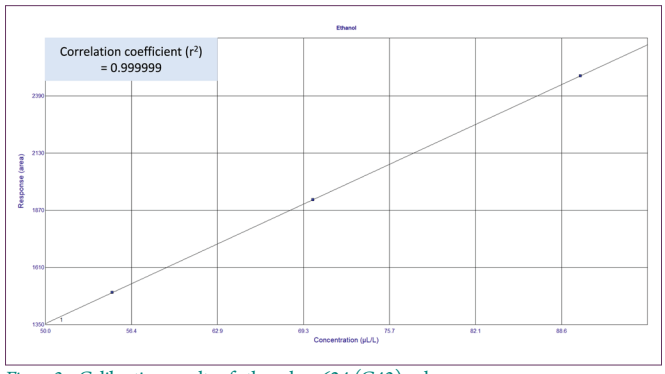

Figure 3a. Calibration results of ethanol on 624 (G43) column.

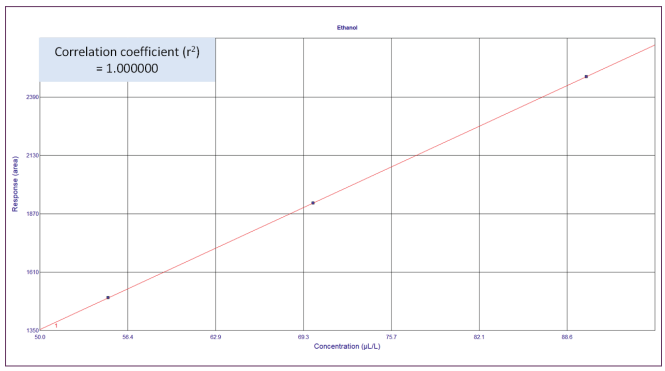

Figure 3b. Calibration results of ethanol on BAC-1 column.

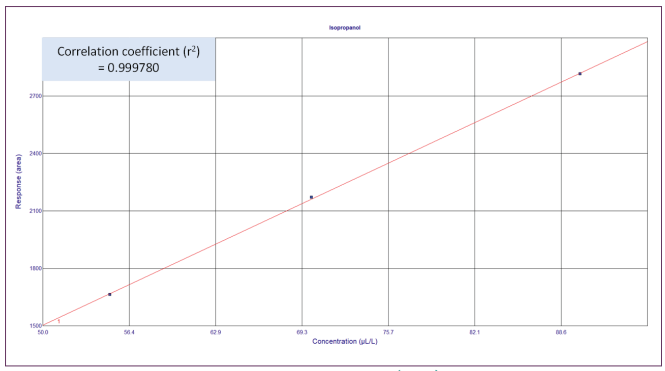

Figure 4a. Calibration results for isopropanol on 624 (G43) column.

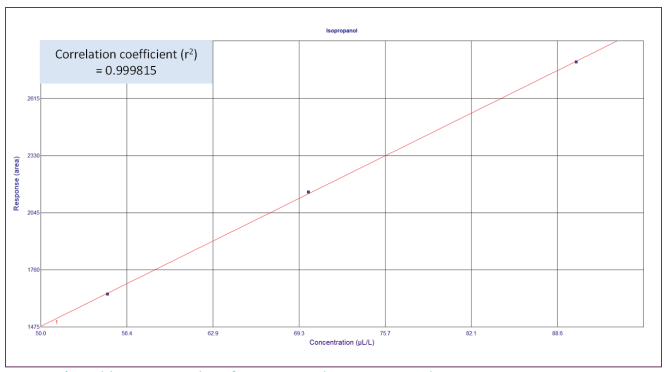

Figure 4b. Calibration results of isopropanol on BAC-1 column.

Samples

Each of the five hand sanitizers were analyzed using the above conditions, with the results shown in Table 4. All samples contained ethanol as the sanitizing agent.

Table 4. Calculated results of five (5) hand sanitizers commercially purchased.

Sanitizer	Label Claim	Amount Calculated
Sample 1	62%	71.2%
Sample 2	70%	75.4%
Sample 3	70%	78.3%
Sample 4	70%	70.8%

Summary

Demonstrated herein, the PerkinElmer Clarus 690 GC easily detects and quantifies ethanol and isopropanol in hand sanitizer for label claim determination with excellent accuracy and precision. Sample preparation is easy, offering the ideal "dilute and shoot" technique. The fast run time of 5.36 minutes per sample enables high productivity, enhanced sample throughput and rapid turnaround of results, maximizing profits for commercial and industrial laboratories.

A workflow document for this analysis is provided with each analyzer, and all method parameters will be implemented on installation. As such, the laboratory can start running samples immediately after installation/implementation.

Acknowledgements

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References:

1. https://www.fda.gov/drugs/drug-safety-and-availability/fda-updates-hand-sanitizers-consumers-should-not-use
2. Excerpted USP-NF and FCC Standards: A Hand Sanitizer Resource A collection of standards provided as a resource to assist with the challenges posed by COVID-19

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