Analysis of Ethanol and Isopropanol in Alcohol-based hand sanitizers by direct injection GC/FID

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Introduction

The most commonly used alcohol-based hand sanitizers are prepared with isopropyl alcohol (IPA), or a form of denatured ethanol. The alcohol in both, ethanol and IPA-based hand sanitizers, is the major component of the hand sanitizer mixture, and its concentration in the aqueous solution determines its effectiveness as disinfecting agents. Other ingredients in hand sanitizers may include hydrogen peroxide and glycerin, but most regulatory methods only require determination of alcohol content by GC/FID to qualify the efficiency of alcohol-based hand sanitizers.

Methods for alcohol determination are widely known, and include USP <611> and ASTM D3695. These and other methods generally require a polar or mid-polar GC columns, such as WAX or 624 phases for the better selectivity and response for alcohol compounds. These traditional columns however, have had shortcomings, particularly with active components in the alcohol/water matrix. As a consequence, the analysis of alcohols suffer from a lack of column inertness, which results in tailing peaks and variable, inaccurate results, in particular, in aqueous samples.

Here, we will evaluate the use of Agilent J&W Ultra Inert GC columns, DB-624 UI and DB-WAX UI for the analysis of alcohol-based hand sanitizers. These phases offers excellent stability for samples containing high level of water-alcohol mixtures. The unique inertness of this phase delivers sharper, and more symmetric peak shapes for alcohols, ensuring better retention time reproducibility for more consistent and accurate quantifiable results.

Results

The GC columns used in this study are among the most popular columns used for analysis of alcohols. Both, the Agilent J&W DB-624 and DB-WAX Ultra Inert, meet column requirements for USP <611>[1,2] and ASTM D3695 methods. In the first set of chromatograms, we see the composition of an IPA-based hand sanitizer prepared following new guidelines set up by the USFDA. For the first chromatogram, we used a DB-624 UI connected to a 1-m retention gap. This setup can help with neat injections of aqueous solution by focusing the water peak prior to reaching the analytical column. Both columns shows good separation and adequate symmetry for IPA at high concentrations. The complimentary selectivity between both phases is also noticeable, and the column choice will depend on user preference or the method of choice.

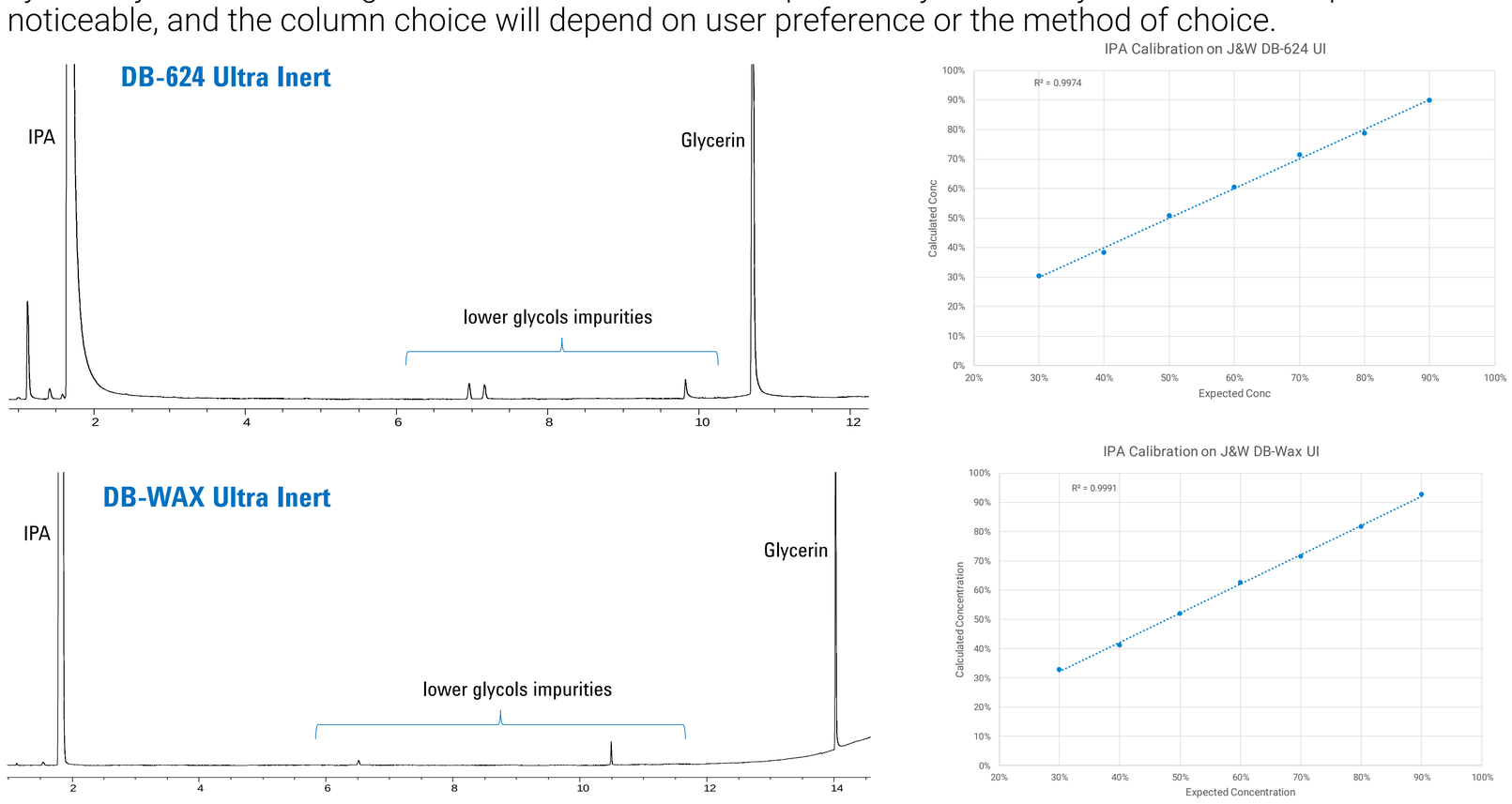

Figure 1. GC/FID chromatograms of an IPA-based hand sanitizer using a DB-624 UI, 30 m x 0.32 mm id x 1.8 µm df (top) and DB-WAX UI, 30 m x 0.25 mm id x 0.25 µm df (bottom). Calibration curves were prepared for IPA in a 30 to 80% v/v range. R2 were 0.997 and 0.999 for DB-624 UI and DB-WAX UI, respectively.

The chromatograms in Figure 2 are Ethanol-based hand sanitizers obtained using the same set of columns. Distilleries have begun to make Ethanol based hand sanitizers[3]. These products, in addition to Ethanol, contain fusel oils that are by products occurring naturally during the fermentation process. Fusel oils will often be present and may present themselves as unpleasant odors in the hand sanitizer. The presence of these alcohols are apparent with both, the DB-624 UI and DB-WAX UI chromatograms. DB-624 UI has the selectivity to fully resolve fusel oils if their analysis is required.

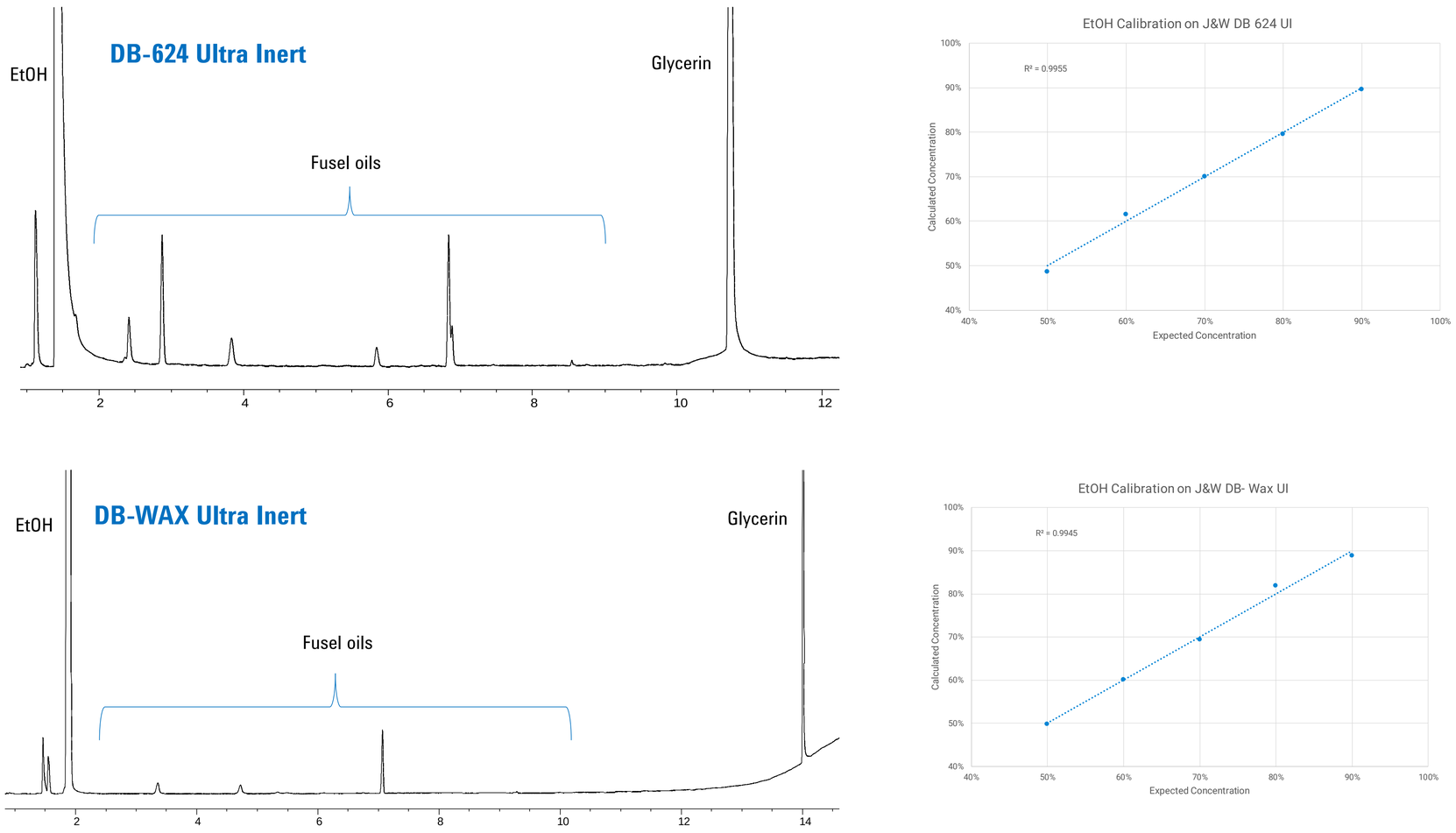

Figure 2. GC/FID chromatograms of an Ethanol hand sanitizer obtained from a distillery. The chromatograms were obtained using a DB-624 UI, 30 m x 0.32 mm id x 1.8 µm df (top) and DB-WAX UI, 30 m x 0.25 mm id x 0.25 µm df (bottom). Calibration curves were prepared for EtOH in a 50 to 90% v/v range. R2 were 0.995 and 0.994 for DB-624 UI and DB-WAX UI, respectively.

Experimental

Instrumentation

- The analyses were performed using an **Agilent 7890 GC** equipped with a flame ionization detector (FID).
- Sample introduction was done using an Agilent **7693A automatic liquid sampler** with 5 µL syringe.

GC Conditions

Method 1

Column	**Agilent J&W DB-624 Ultra Inert**, 30 m x 0.32 mm x 1.8 µm (p/n 123-1334UI) **Deactivated fused silica**, 1 m x 0.32 mm (p/n 160-2325-1)
Carrier	Helium, constant flow, 6 mL/min (73 cm/s)
Oven	40 °C (5 min), 20 °C/min to 225 °C (2.5 min). Runtime: 16.75 min
Inlet	Split mode, 140 °C, split ratio 40:1
Inlet liner	Ultra Inert, flow pressure drop with glass wool (p/n 5190-2295)
Injection	1 µL

Method 2

Column	**Agilent J&W DB-WAX Ultra Inert**, 30 m x 0.25 mm x 0.25 µm (p/n 122-7032UI)
Carrier	Helium, constant flow, 6 mL/min (80 cm/s)
Oven	40 °C (5 min), 20 °C/min to 225 °C (2.5 min). Runtime: 16.75 min
Inlet	Split mode, 140 °C, split ratio 40:1
Inlet liner	Ultra Inert, flow pressure drop with glass wool (p/n 5190-2295)
Injection	1 µL

Conclusions

This study shows the positive impact the Agilent J&W DB-WAX UI and DB-624 UI columns have on the quantification of alcohols in alcohol-based hand sanitizers. Retention times and peak shapes for the alcohols were consistent over the course of several direct sample injections, even in the presence of water being injected as a solvent. Calibration curves up to 90% of the alcohol compound with an R^2 > 0.99 were prepared. If analysis of fused oils is required, DB-624 UI gives a better resolution for these compounds, however, both columns, show superior inertness and stability for the analysis of alcohol-based hand sanitizers.

References

1. Isopropyl Alcohol, Monograph US Pharmacopeia, 2020
2. USP <611> Alcohol Determination, Monograph US Pharmacopeia, 2020
3. Temporary policy for preparation of certain alcohol-based hand sanitizers products during the public health emergency, FDA

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