

Distilled Spirit Analysis using the New 100 % Aqueous Stable Zebtron™ ZB-WAXplus

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*You can't inject water on your WAX phase GC column!
It's one of the oldest rules of gas chromatography...one that has
now been broken thanks to the Zebtron ZB-WAXplus!*

Introduction

WAX columns provide optimal selectivity for many aqueous soluble compounds such as those found in alcoholic beverages. Historically, polyethylene glycol (PEG) phases have been unstable with aqueous samples resulting in poor reproducibility and decreased lifetime. The new Zebtron ZB-WAXplus bonding procedure results in exceptional stability to repeated injections of aqueous matrices.

Alcoholic Beverage Testing

Distilled spirits are analyzed for compounds called congeners, which are formed during fermentation and add to the flavor but can be harmful if consumed in excess. Some spirits, such as vodka, undergo extra processing steps used to eliminate these compounds. Overabundance of a specific congener can also be used to identify problems with production.

Distilleries are also plagued by fraudulent claims made by customers trying to return an expensive bottle of whiskey that "tastes funny" for a full refund. Upon testing, the congener profile isn't consistent with the product, suggesting that the customer drank half the bottle and then diluted the rest with a clear alcohol, such as rum or vodka, to make the volume appear as if it had not changed.

The analysis of distilled spirits is challenging because the finished product is between 40-80 % water and congener levels are in the low parts per million (ppm) level. Headspace sampling can eliminate some of the matrix effects. However, many times a direct injection is still required to verify a specific sample. The aqueous stability offered by the Zebtron ZB-WAXplus makes it the perfect choice for this method.

Results & Discussion

Whiskey samples were analyzed without dilution using a split injection technique to improve sensitivity for low-level compounds. Baseline resolution was achieved for acetaldehyde, ethyl acetate, and methanol (important components that result from the distillation process). Storage conditions and alcohol content affect the resulting congener concentrations after barrel conditioning, so these compounds must be monitored to ensure product consistency.

In addition to providing aqueous stability, the Zebtron ZB-WAXplus also provides very low activity for acidic compounds. This allowed for the fatty acids eluting past 12 min to be analyzed within the same run. The lack of acetic acid in the sample suggests that the product was well stored prior to opening and that the cork seal from the bottle was not compromised.

Repeated injections were made to ensure that the analysis was stable and reproducible. The representative chromatograms below in Figure 1 demonstrate that the high water content of the whiskey sample does not adversely affect the reproducibility of the analysis. The percent Relative Standard Deviation for the analysis was less than 5 % for all measurements.

Experimental Conditions

Column: Zebtron ZB-WAXplus

Dimensions: 30 meter x 0.25 mm x 0.25 μ m

Injection: 0.2 μ L split 30:1 at 140 °C

Carrier Gas: Helium @ 1.4 mL/min (constant flow)

Oven Program: 35 °C for 5 min to 85 °C @ 10 °C/min to 200 °C @ 25 °C/min for 1 min

Detector: FID @ 200 °C

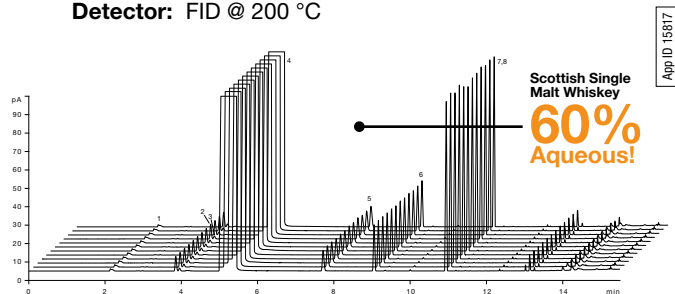


Figure 1. Peaks (1) Acetaldehyde (2) Ethyl Acetate (3) Methanol (4) Ethanol (5) Propanol (6) Isobutanol (7) 2-Methylbutanol (8) 3-Methylbutanol.

ORDERING INFORMATION

Part No.	Description
7HG-G013-11-TN	ZB-WAXplus, 30 m x 0.25 mm x 0.25 μ m



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