

Volatile Profile Comparison of Flavored and Non-Flavored Vodkas by Purge & Trap Thermal Desorption GC/MS

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Introduction

The ability to profile volatile and semi-volatile components present in food and beverage products is critical to help improve batch to batch consistency and avoid any potentially dangerous contaminants, whether they originate from the raw materials, packaging, or were created during the manufacturing process. A series of flavored and non-flavored commercially available vodkas were analyzed using a purge and trap thermal desorption GC/MS instrumentation. The use of purge and trap thermal followed by thermal desorption has several advantages over classical extractions including increasing sample throughput, reducing loss of volatile components in the extraction process and eliminating the need for solvents.

Materials & Methods

Samples included an internal analytical standard of deuterated glycerol (1,1,2,3,3-d5) from Sigma-Aldrich (454524-1G), a series of commercially available vodkas, Vendor A (plain, chocolate, raspberry and lemon variants), Vendors B, C, D, & E were plain variants. A 10 ml aliquot of a vodka sample was placed in a glass purge & trap tube. The commercially available purge & trap system (Adaptas Solutions) was purged with UHP grade Helium at a rate of 15 ml/min for 15 minutes. A pre-conditioned thermal desorption tube packed with 200 mg of Tenax GR (60/80 mesh) was attached. **(Figure 1)** The tubes were 4 mm ID x 0.25” OD x 4.00” L and were Silco coated to prevent catalysis with the SS body. The tubes were preconditioned at 320C for 4 hours in a commercial conditioning oven (Adaptas Solutions) while a steam of UHP nitrogen (30 ml/min) was passed through the tubes. After the purge & trap collection, the desorption tube was placed on an Adaptas Solutions TD5 thermal desorption system. **(Figure 2)** A 35 mm preconditioned SS desorption needle was attached. The TD5 was coupled to an Agilent 6890 GC utilizing a 5973 MSD as a mass detector. The 6890 GC had a SIS cryotrap installed on the injector that cooled the samples to -40C with liquid CO2 to cryofocus the sample during the desorption process. The TD5 was programmed to allow for a 30 second dry purge to remove any residual oxygen from the tube. The samples were desorbed at 200C for a duration of 5 minutes. After

the desorption period the cryotrap is ballistically heated to 250C for the 3 minutes and the GC/MS acquisition is initiated. The MSD is scanning a mass range of 45-500 u at 0.5 S per scan. The GC column used in this analysis was a Trajan BPX-5 (0.25 mm ID x 60M) with a 0.25 µm film and was operated from 40-280C with a ramp rate of 5C/min. The GC was operating in split mode at a 5:1 slit ratio. Mass Spectral data was compared with the NIST17 AMDIS software for component identification.

Figure 1 - Purge & Trap Device

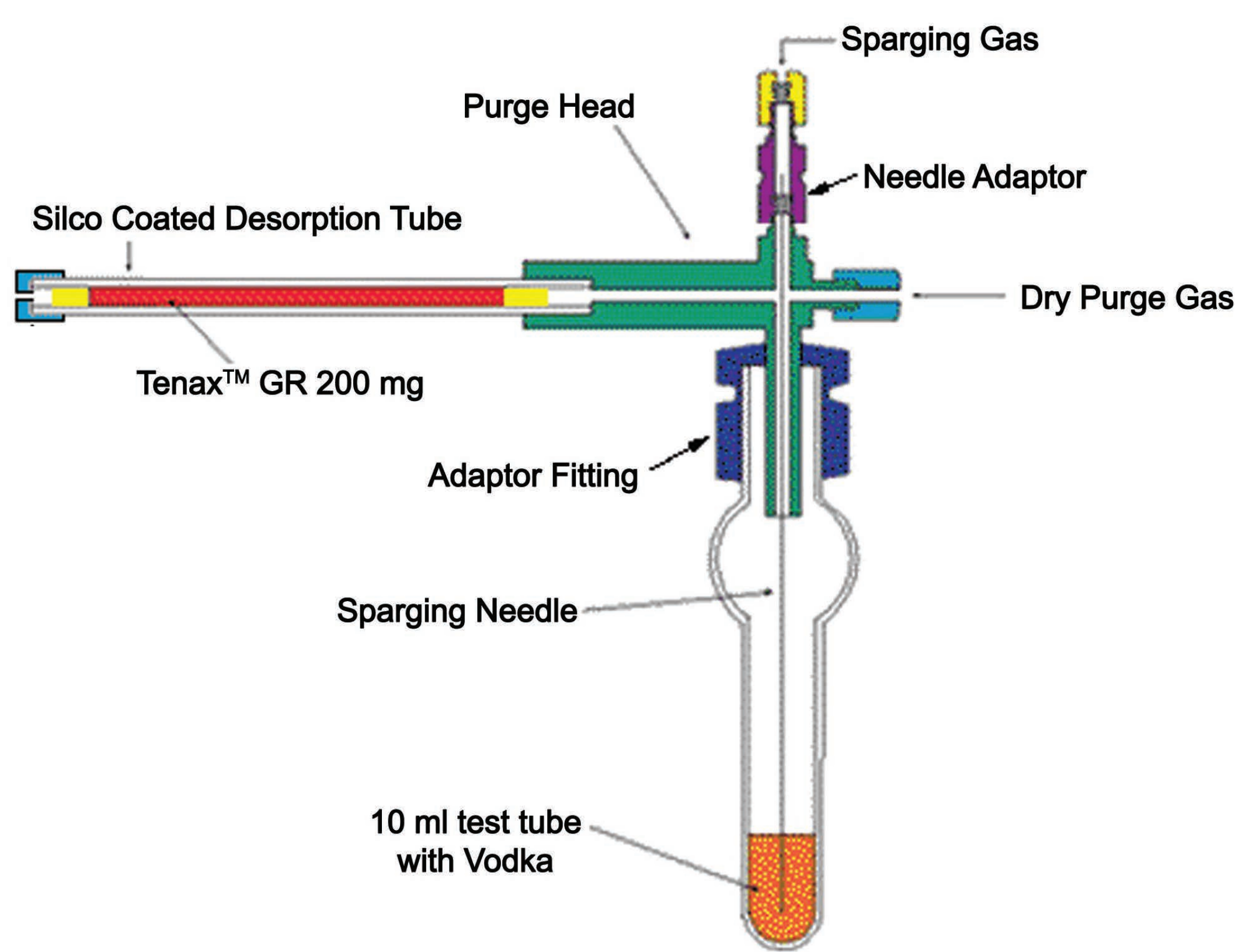


Figure 2 - TD5 Unit



Figure 3

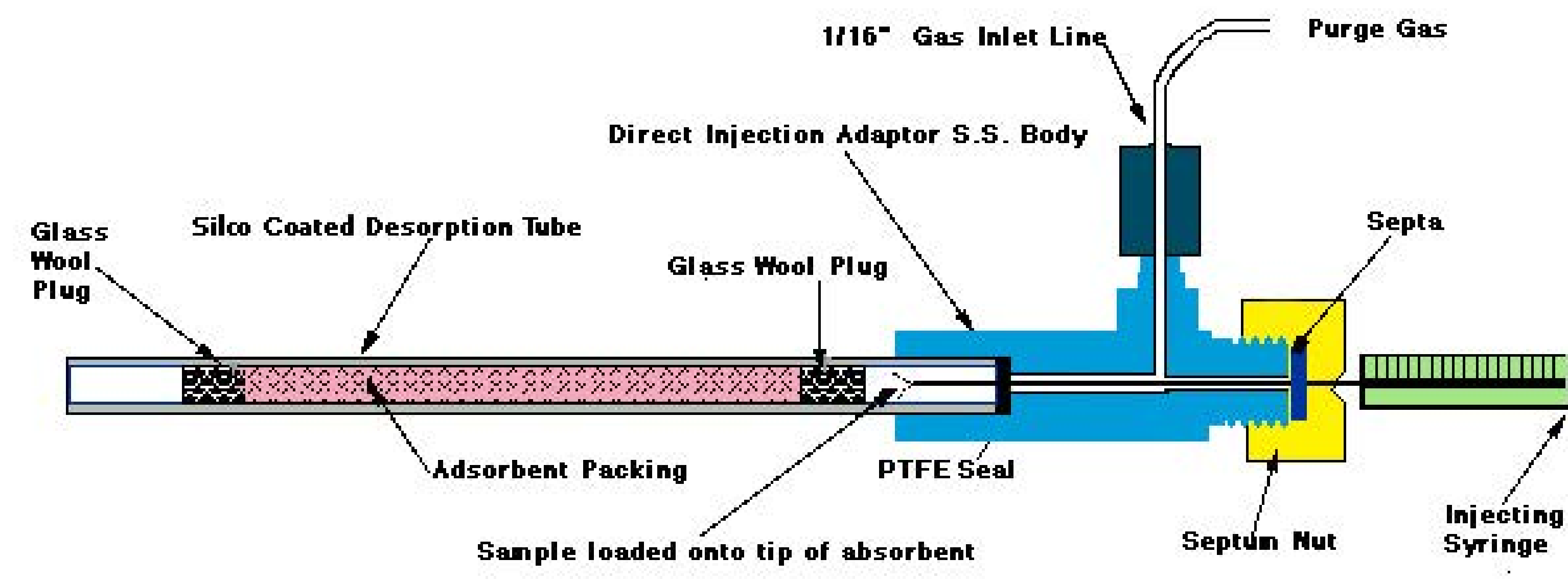
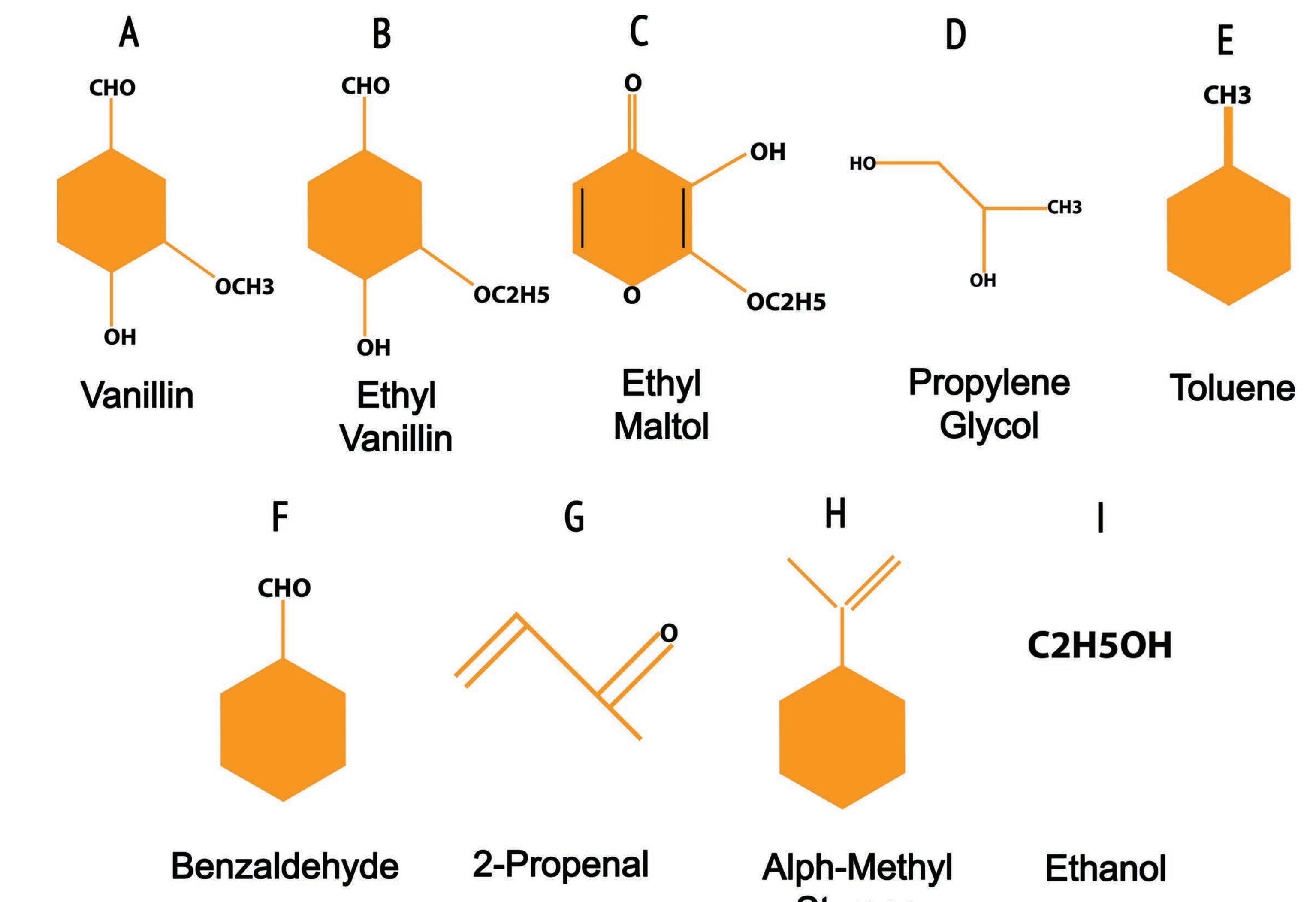


Figure 4



Results & Discussions

A deuterated standard of glycerol (1,1,2,3,3-d5) (1 µg/µl) was diluted in methanol (10:1) and pipetted into the desorption tube as an internal standard after the purge & trap collection was completed, using a special designed desorption tube injection head **(Figure 3)**. This device reduces the chance of the Internal standard from being lost due to evaporation, details of this operation are shown in Figure 4.

Vendor	Flavor	Components observed
A	Chocolate	A, B, C, D, I
A	Lemon	D, I
A	Raspberry	A, D, I
A	Non-Flavored	D, I
B	Non-Flavored	E, F, G, H, I
C	Non-Flavored	E, G, I
D	Non-Flavored	F, G, I
E	Non-Flavored	F, I

Conclusion

TD/GC/MS is an efficient process to determine the composition and concentrations of the volatile and semi-volatile components in flavored and non-flavored vodka products. The advantage of the short-path desorber is the ability to analyze directly either liquid, solid or gas samples with each sample having its own unique transfer line to eliminate cross-contamination or carryover between samples.