

Application Note 163

The GC/MS Analysis of Impurities in Denatured Ethanol on the SPB™-1 Capillary Column

A 60m x 0.25mm ID, 1.0µm SPB-1 capillary column is an excellent choice for the analysis of impurities in denatured ethanol, a solvent commonly used in industry. The 60-meter length provides the efficiency necessary for the separation and the column elutes the ethanol and methanol in front of the peaks of interest.

Key Words:

• impurities • GC/MS • SDA • alcohol

Denatured ethyl alcohol is a solvent commonly used in industry. SDA is the most widely used form. There are over 50 different compositions of SDA and each has a different end use.

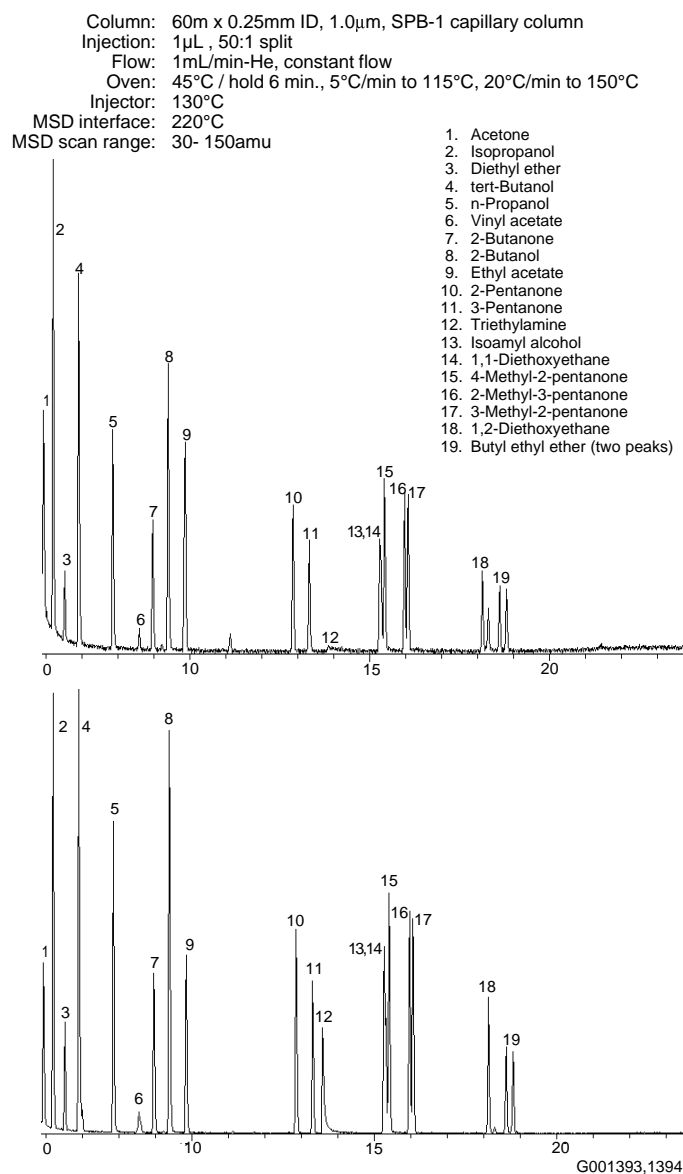
Organic impurities present in the denatured ethanol, if at a high enough level, can effect the product in which it is used. In this application, we analyzed for impurities commonly found in SDA-3A. This form of SDA is ethanol denatured with 5% methanol. GC/MS was used for positive compound identification, which presented a special analytical challenge. To protect the mass spectrometer source and filaments from solvent front overload and damage, the MSD was turned off while the ethanol/methanol peaks eluted. The MSD was turned back on in time to detect the peaks of interest.

To perform the analysis, we chose the 60m x 0.25mm ID, 1.0µm SPB-1 capillary column. This nonpolar column eluted the ethanol and methanol in front of the peaks of interest, and the 60-meter length provided the efficiency necessary for the separation. These two factors allowed the MSD to remain "off" in the solvent delay mode at the start of the run and be turned back on in time to detect the first peak.

The separation of the 19 impurities in SDA-3A is illustrated in Figure A. The GC/MS remained in solvent delay mode until the ethanol peak had eluted and was turned back on in time to detect the acetone peak. Isoamyl alcohol and 1,1-diethoxyethane were the only peaks not at least 70% resolved in the analysis (Figure B), however, these compounds are mass resolved by the MSD. The inertness of the SPB-1 allowed the impurities to be detected at on-column amounts of 0.30 – 1ng.

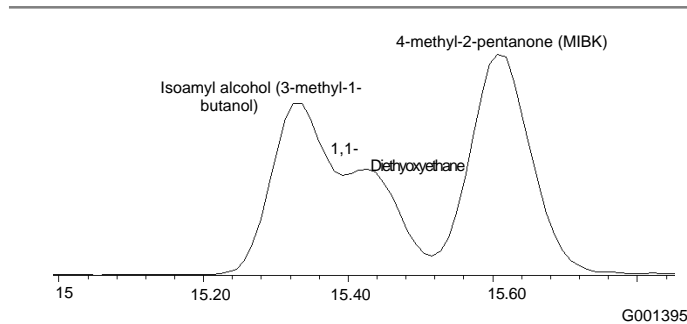
The stability of the SPB-1 column was tested by injecting the SDA-3A mix repeatedly over the course of nine days. Overlays of the runs made on day one, four and nine are presented in Figure C. No retention time shift was evident over the nine-day test period. Retention time shifts are especially troublesome for GC/MS methods, because analytes are often identified and quantified from specific ions that are seen by the data system within predetermined retention time windows.

Figure A. Separation of Organic Impurities in Denatured Ethyl Alcohol, 0.30-1.0ng and 1.8-6.0ng On-Column



The resolution, inertness, and stability of the 60m x 0.25mm ID, 1.0µm SPB-1 makes it an excellent choice for the analysis of impurities in denatured ethanol. If mass spectrometric detection is used, the column provides adequate resolution between the solvent front and peaks of interest to protect the detector through use of solvent delay.

Figure B. Partial Separation of Isoamyl Alcohol and 1,1-Diethoxyethane

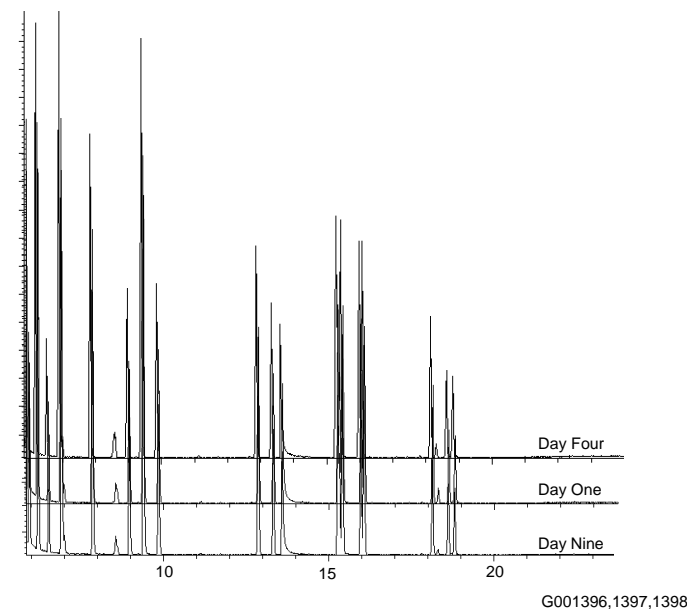


Ordering Information:

Description	Cat. No.
SPB-1 Fused Silica Capillary Column	
60m x 0.25mm ID x 1.0µm ID	24031

Contact our Technical Service Department (phone 800-359-3041 or 814-359-3041, FAX 814-359-5468) for expert answers to your questions.

Figure C. Injection of SDA-3A Sample Over Nine Day Period on a 60m x 0.25mm ID, 1.0µm SPB-1 Capillary Column



*Runs have been deliberately offset for comparison

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Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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