

## Determination of Aromatics in Mineral Spirits Using a TCEP Capillary GC Column

The TCEP capillary column meets or exceeds the performance requirements of ASTM D3257, and provides higher resolution, compared to the packed GC column specified in the method. The TCEP capillary column also provides much more rapid separations, eluting cyclohexanone (the specified internal standard) in 17 minutes, compared to 50 minutes for the packed column. Quantitative results for mineral spirits blends containing aromatics in concentrations ranging from 0 - 20% showed very good agreement with the known amounts. (ChromFax No.: 394023)

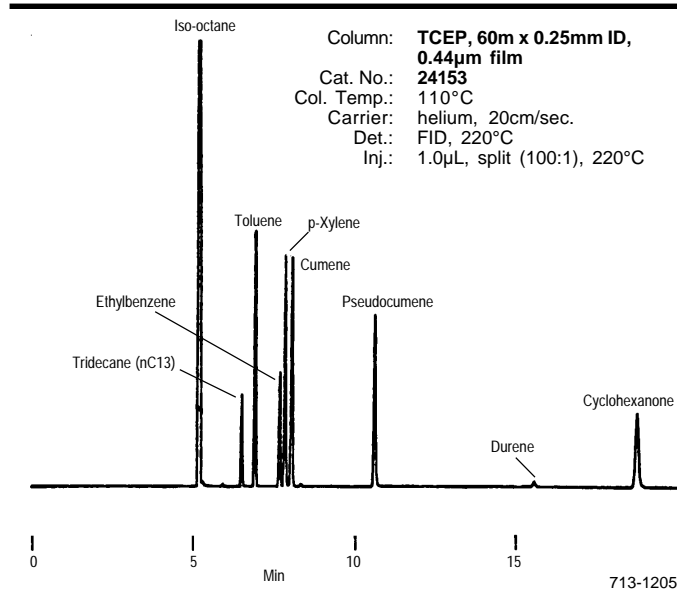
### Key Words:

- aromatics • ASTM D3257
- industrial hygiene • mineral spirits

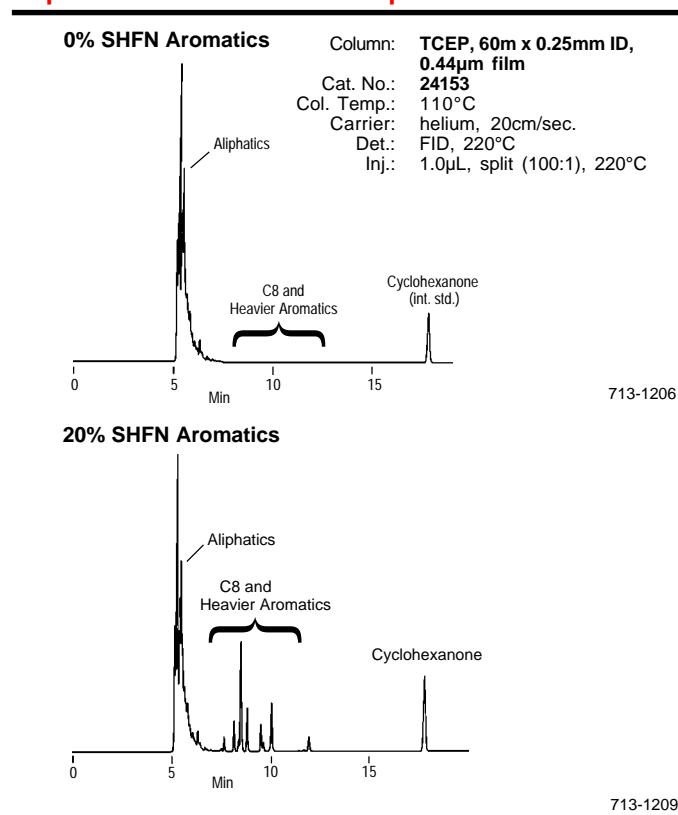
Mineral spirits, a petroleum-derived solvent widely used as a cleaning material and for the manufacture of paints (1), contains varying amounts of aromatics ranging from 0 - 30% by volume. ASTM Test Method D3257 describes a packed column GC procedure for determining ethylbenzene, other C8 aromatics, and heavier aromatics in mineral spirits within the concentration range of 0.1 - 30% (2). The method requires a highly polar stationary phase to retain the aromatic components longer than the nonpolar hydrocarbons which make up the bulk of the mineral spirits.

To improve the separation of the aromatics from the nonaromatics, we developed a 60m x 0.25mm ID capillary column coated with a 0.44µm film of 1,2,3-tris (cyanoethoxy) propane (TCEP), a highly polar stationary phase. Separation of a test mixture containing components described in Method D3257 demonstrates that the TCEP capillary column easily exceeds the resolution requirements of the method (Figure A). The method requires that the column have sufficiently high polarity to elute toluene after n-tridecane (n C13) with peak resolution (R) greater than 0.9. Also, ethylbenzene must be separated from p-xylene and isopropylbenzene (cumene) with the depth of the valley between them greater than 50%. Toluene is more than baseline resolved from tridecane, and ethylbenzene is essentially baseline resolved from p-xylene and cumene. As an added advantage of the capillary column over the packed column, more of the individual aromatics are resolved. For instance, p-xylene and cumene, which elute as a single peak using the N,N-bis (2-cyanoethyl) formamide packed column described in the ASTM method, are baseline resolved using the TCEP capillary column.

**Figure A. D3257 Calibration Standard Mixture**



**Figure B. Super High Flash Naptha Aromatics in Mineral Spirits**



Cyclohexanone, an internal standard required by the method, elutes as the last peak from the TCEP column in approximately 17 minutes. Using the packed column, a much longer time, on the order of 50 minutes, is required to elute the internal standard.

The potential for quantitative analysis, using the TCEP column, was demonstrated by evaluating samples of mineral spirits containing total aromatics concentrations ranging from 0 - 20% (Figure B). Super high flash naphtha (SHFN, aromatic solvent 100) was blended with aromatics-free mineral spirits to provide samples containing total aromatic concentrations of 1.5%, 5%, 10%, 15%, and 20% by volume. When separated using the TCEP column, the nonaromatics elute within 5-7 minutes. C8 and heavier aromatics, if present, elute in 7-15 minutes. The flat baseline in this region of Figure B1 indicates that essentially no C8 or heavier aromatics were present in the aromatics-free base sample.

**Table 1. Capillary GC Analysis of Blended Mineral Spirits With Known Aromatics Content**

Added <sup>▲</sup>	Volume % Aromatics	
	Added <sup>▲</sup>	Found
0		0.68
1.5		1.58
5.0		5.44
10.0		10.37
15.0		16.68
20.0		19.95

<sup>▲</sup> Added by volume as super high flash naphtha (SHFN, aromatics solvent 100) to an aromatics-free mineral spirits sample, provided by UNOCAL Petroleum Products and Chemicals Division, Unocal Corporation, Schaumburg, Ill.

Upon peak area integration and quantitation, the total aromatics content was calculated and compared to the known values for each sample (Table 1). All peaks eluting later than the retention time of toluene were quantified as aromatics. The level of compounds (0.68%) quantified as aromatics in the aromatics-free mineral spirits sample (Figure B1) most likely represents trace levels of nonaromatic hydrocarbons. These peaks eluted in the time between toluene and ethylbenzene, where no other aromatics are known to elute. In spite of this bias, the results show good agreement between the GC results and the known values and indicate the potential of this capillary GC technique for quantitative analysis of total aromatics in mineral spirits.

## Ordering Information:

### TCEP Fused Silica Capillary Column

60m x 0.25mm ID, 0.44µm film

24153

**Note:** The maximum operating temperature for TCEP is 145°C. Extended use of this phase (or any other nonbonded stationary phase having a low temperature limit) at or near the maximum operating temperature will produce a gradual loss of stationary phase. Over time, this typically shortens retention times. For this reason, we recommend that retention time calibration be performed on a regular basis. To maximize column lifetime, minimize use at the maximum operating temperature.

#### References

1. ASTM Standard Specifications D235-87, Standard Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent), Vol. 6.03, 1990 Annual Book of ASTM Standards.
2. American Society for Testing and Materials, Book of ASTM Standards, Test Method D3257-88, Volume 6.03.

Obtain references from ASTM, 1916 Race Street, Philadelphia, PA 19103 USA.

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