

Complete Resolution of Benzene from Ethanol in Spark Ignition Fuels

Using a Modified ASTM D3606-06e1 Method and the New D3606 Column Set

By Barry L. Burger, Petroleum Innovations Chemist

- Easy, accurate quantification of aromatics.
- Fully conditioned column set—ready to use out of the box.
- Each column set is tested for method applicability and includes chromatogram.

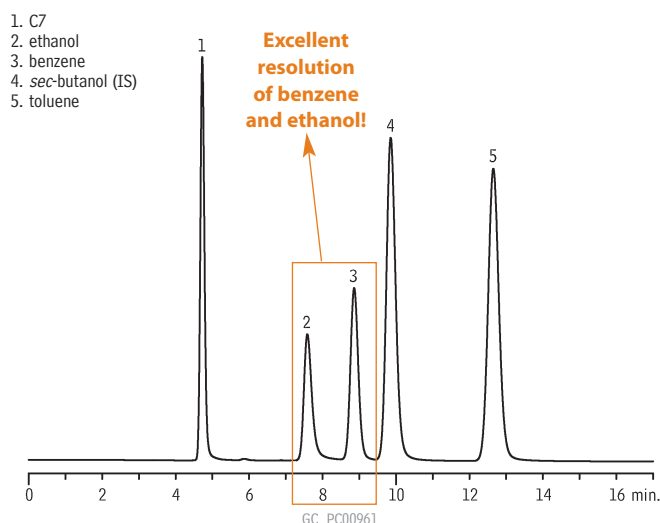
Laboratories analyzing reformulated spark ignition fuels that contain ethanol for the determination of benzene and toluene must use a modified ASTM D3606-06e1 method to prevent the coelution of ethanol and benzene. This method modification is also a requirement of the US EPA. The benzene range of determination is 0.1 to 5% by volume, and the toluene range is 2 to 20% by volume. The primary challenge in this analysis is twofold: the tailing of the ethanol peak, and the retention time shift of the aromatics towards ethanol, specifically benzene merging quickly into the ethanol peak and preventing accurate quantification.

Restek has resolved these issues by developing a new D3606 column set for this modified ASTM D3606-06e1 application. Column 1 is a 6' x 1/8" OD (1.8m x 2mm ID) nonpolar Rtx®-1 phase which separates components by boiling point. After the elution of *n*-octane (C8), Column 1 is backflushed to prevent heavier compounds from entering Column 2, the main analytical column. The light compounds pass into Column 2, a 16' x 1/8" OD (4.9m x 2mm ID) column packed with a new proprietary polymer that fully resolves the aromatic compounds.

To demonstrate the performance of this new column set, we installed it in an Agilent 6890 GC equipped with a flame ionization detector (FID). Helium was used as the carrier gas at 20mL/min. in the constant flow mode. The data in Figure 1 show that the aromatic compounds are fully resolved, and can easily be quantified using the internal standard, *sec*-butanol.

This column set is fully conditioned and ready to use right out of the box. Only a brief (10 min.) carrier gas purge at ambient temperature, followed by a 30 min. hold at 165°C, is required. If your laboratory has been struggling with ASTM method D3606-06e1 for reformulated fuels containing ethanol, Restek's new column set is the solution.

Figure 1 Complete resolution of benzene from ethanol using a D3606 column set and modified ASTM D3606-06e1 method.



Column: D3606 Column Set
column 1: 6' x 1/8" OD (1.8m x 2mm ID), nonpolar Rtx®-1 polymer
column 2: 16' x 1/8" OD (4.9m x 2mm ID), proprietary packing

Sample: 0.05µg/µL; C7 (26%), ethanol (10%), benzene (10%), *sec*-butanol (26%), toluene (26%)

Inj.: 0.05µL, direct injection
Inj. temp.: 200°C
Carrier gas: helium, constant flow
Flow rate: 20mL/min.
Oven temp.: 135°C, isothermal
Det.: FID @ 250°C

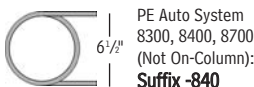
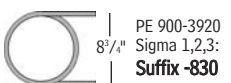
D3606 Application Column (2 column set) **new!**

Description	cat.#**
D3606 Application Column (2 column set)**	
Column 1: 6' (1.8m), 1/8" OD, 2.0mm ID, nonpolar Rtx®-1	
Column 2: 16' (4.9m), 1/8" OD, 2.0mm ID, proprietary packing material	83606-

*Please add column instrument configuration suffix number to cat.# when ordering.

**This column set is for a valving system; therefore, packing material is filled to ends of columns.

Column Instrument Configurations



Note: Initial 2" of column will be empty, to accommodate a needle. For a completely filled column (not on-column), add suffix -901.