



PETROCHEMICAL

## Complete Resolution of Benzene from Ethanol in Spark Ignition Fuels

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

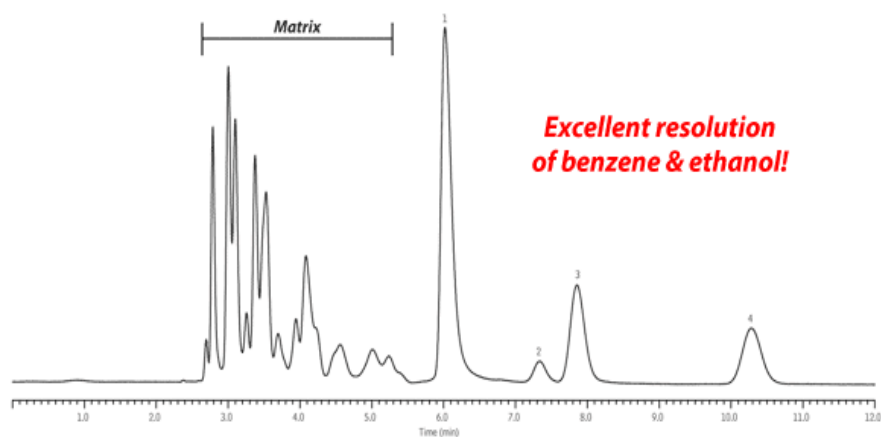
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- Easy, accurate quantification of aromatics.
- Fully conditioned column set—ready to use out of the box.
- Each column set is tested for method applicability and includes chromatogram.

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 into benzene, 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 TCD detector. Helium was used as the carrier gas at 20mL/min. in the constant flow mode. ChemStation® was used as the data acquisition software. 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 (10min.) carrier gas purge at ambient temperature, followed by a 30min. 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.

1. ethanol
2. benzene
3. 2-butanol (IS)
4. toluene



Column: D3606 Application Column (2 column set, cat.# 83606-800)  
Column 1: nonpolar Rtx®-1, 6' (1.8m), 1/8" OD, 2.0mm ID  
Column 2: proprietary packing material, 16' (4.9m), 1/8" OD, 2.0mm ID

Sample: commercial gasoline

Inj.: 1µL, direct  
Inj. temp.: 200°C  
Valve box temp.: 200°C

Backflush time: 3 min.

Carrier gas: helium, constant flow  
Flow rate: 20mL/min.

Oven temp.: 135°C, isothermal  
Det.: TCD @ 230°C  
Instrument: Agilent 6890

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