

RESOLVE BENZENE, OTHER AROMATICS, AND OXYGENATES IN REFORMULATED GASOLINE

Using a One-Column Approach with an SLB®-ILD3606 Column

The amount of benzene in gasoline is a concern because it is a known human carcinogen, and exposure to it is linked to detrimental health effects. The challenge with the analysis lies in the complex composition of gasoline, which consists of hundreds of different compounds. Reformulated gasoline also contains additives to produce more complete combustion and subsequent lower emissions of harmful compounds. These additives accomplish this by boosting the oxygen content, and are commonly referred to as "oxygenates." Ethanol is a commonly used oxygenate. Therefore, to measure benzene in reformulated gasoline, it must be resolved from the aliphatic hydrocarbons, other aromatics, ethanol, plus any other oxygenates. This typically requires the use of a two-column switching procedure.¹

We developed SLB-ILD3606 specifically for the determination of benzene, other aromatics, and oxygenates in gasoline. Table 1 lists its specifications. It employs an ionic liquid stationary phase and provides great peak shapes for oxygenates, resulting in improved resolution for all analytes.

Table 1. SLB-ILD3606 Column Specifications

Application: Modified (deactivated) version of SLB-IL111 provides better inertness. Each column is tested to ensure resolution and sharp peak shapes of aromatics and alcohols. Excellent at separations involving benzene (and other aromatics) and oxygenates in petroleum products, such as gasoline. Also a good GCxGC column choice.

USP Code: None

Phase: Non-bonded; 1,5-di(2,3-dimethylimidazolium)pentane bis(trifluoromethanesulfonyl)imide

Temp. Limits: 50 °C to 260 °C (isothermal or programmed)

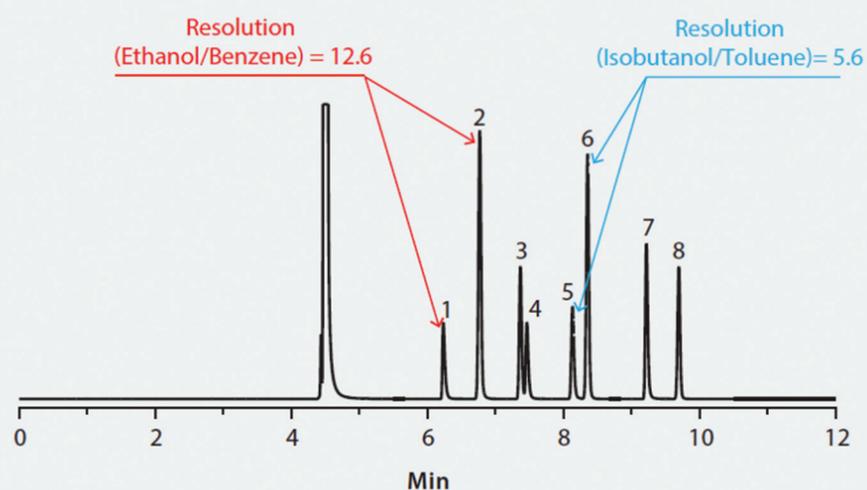
Resolution Test

To show selectivity and inertness capabilities, a mixture containing two aromatics, five alcohols, and one ketone was prepared in isooctane following the guidelines for a control standard to be used with ASTM® D3606. This mix was analyzed using temperature programming and the resulting chromatogram is shown in Figure 1. The inertness of the column resulted in sharp peak shapes for all alcohols, which in turn resulted in great resolution between ethanol and benzene ($RS=12.6$), and also between isobutanol and toluene ($RS = 5.6$).

Figure 1. Aromatic and Alcohol Standard

column: SLB-ILD3606, 60 m × 0.25 mm I.D., 0.20 μm (29691-U)
oven: 50 °C (6 min), 15 °C/min to 265 °C (10 min)
inj. temp.: 250 °C
detector: FID, 250 °C
carrier gas: helium, 21 cm/sec
injection: 1 μL, 100:1 split
liner: 4 mm I.D., split type, cup design
sample: 8 analytes at various concentrations in isooctane

- | | |
|----------------|----------------------------------|
| 1. Ethanol | 5. Isobutanol |
| 2. Benzene | 6. Toluene |
| 3. sec-Butanol | 7. n-Butanol |
| 4. n-Propanol | 8. Methyl isobutyl ketone (MIBK) |



Reformulated Gasoline Sample

Figure 2 shows the chromatogram resulting from the analysis of a reformulated gasoline sample on SLB-ILD3606. As shown:

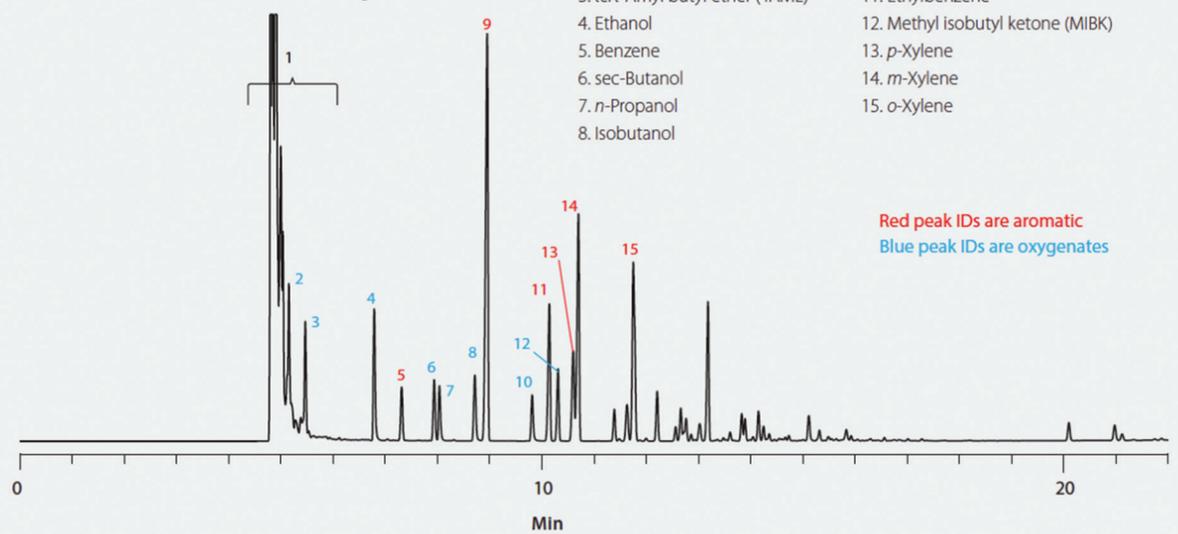
- The extremely polar selectivity of this column resulted in the elution of benzene after the aliphatic portion and also great resolution between benzene and ethanol
 - Several other aromatic and oxygenate compounds are also fully resolved
 - A few aromatics (e.g. *p*-/*m*-xylene) and oxygenates (e.g. MTBE, TAME) are partially resolved
 - This column can be used up to 260 °C, so allows the timely elution of the heavy polycyclic aromatic hydrocarbon (PAH) constituents in gasoline
 - The phase stability of SLB-ILD3606 produces minimal bleed, even when subjected to a temperature ramp
- These observations indicate that the SLB-ILD3606 is an effective alternative to the two-column switching procedure (according to ASTM D3606) currently required for the determination of benzene and other aromatics in reformulated gasoline.

Ordering Information

Description	Cat. No.
SLB-ILD3606, 30 m × 0.25 mm I.D., 0.20 μm	29687-U
SLB-ILD3606, 60 m × 0.25 mm I.D., 0.20 μm	29691-U

Figure 2. Reformulated Gasoline

sample: reformulated gasoline (contains 10% ethanol) with 7 other oxygenates added (at 2.5-5%)
All other conditions the same as Figure 1



1. C5-C11 aliphatic hydrocarbons
2. Methyl tert-butyl ether (MTBE)
3. *tert*-Amyl butyl ether (TAME)
4. Ethanol
5. Benzene
6. *sec*-Butanol
7. *n*-Propanol
8. Isobutanol
9. Toluene
10. *n*-Butanol
11. Ethylbenzene
12. Methyl isobutyl ketone (MIBK)
13. *p*-Xylene
14. *m*-Xylene
15. *o*-Xylene

Red peak IDs are aromatic
Blue peak IDs are oxygenates

Reference

1. ASTM@ D3606, Benzene and Toluene in Unleaded Gasoline and Aviation Fuel.

Related Information

Additional chromatograms, product information, real-time availability, and ordering information is available 24 hours a day at sigma-aldrich.com/gc-petro

Author Contact Details

Lisa McCombie Merck KGaA, Darmstadt, Germany • 595 North Harrison Road Bellefonte, PA 16823 USA • Tel +1 814 359 3441
• Email: lisa.mccombie@sial.com • Web: SigmaAldrich.com/GC

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Versatile Automatic Viscometers

The U-Visc series, from **Omnitek** consists of 4 different models of fully automatic viscometers, ranging from 1 bath with 1 tube, up to 2 independent baths with 2 tubes each, offering a solution for each application and sample load. The modified Ubbelohde viscometers offer a 100-fold measuring range which eliminates the need to exchange tubes frequently. Fitted with an autosampler holding 16 positions per tube, and optional sample preheating as well as dual solvent cleaning, the system is extremely versatile and can be used for a multitude of applications. Its intuitive design combined with advanced control software provides ease of use yet allows for customisation based on sample characteristics.

For More Info, email: 29711pr@reply-direct.com



Stay Connected Under Pressure



Press-fit style connectors work well for many applications, but the connector seal can be broken by extreme temperature and pressure cycling. In these situations, SilTite μ-Unions, from **Restek**, are a better alternative as they create a permanent connection between fused silica analytical columns, guard columns, and retention gaps. SilTite FingerTite technology provides easy installation and a reliable, leak-tight connection. Data quality can be improved by using these connectors because their zero-dead-volume design and deactivated metal construction ensure optimal peak shapes. Robust SilTite μ-Unions are recommended for mass spectrometry work and any application with extreme temperature and pressure changes. Connector kits are available in six configurations that are designed to securely connect columns of either the same or different inner diameters. Each kit contains two SilTite μ-Union connectors, five double-taper ferrules, and installation tools.

For More Info, email: 38873pr@reply-direct.com

Expanded Range of Viscometers for Lubricating Oils

The SVMTM series of viscometers from **Anton Paar** has recently been expanded with the new SVM 4001 Stabinger Viscometer. It has a double-cell system for viscosity measurements at two different temperatures simultaneously, providing the fastest Viscosity Index determination from the lowest sample volume.



The innovative double-cell design of SVM 4001 allows for simultaneous measurements at 40 °C and 100 °C. As each of the measuring cells also contains an integrated density oscillator, density measurements according to ASTM D4052 at each temperature are simultaneously performed. The minimum sample volume needed for Viscosity Index determination and the density measurements is only 2.5 mL. The Viscosity Index results according to D2270 as well as API calculations are automatically shown on the touchscreen.

Moreover, SVM 4001 allows for simultaneous determination of viscosity and density at any two temperatures between 15 °C and 100 °C. This state-of-the-art SVM model also provides viscosity-temperature extrapolation according to ASTM D341 and freely selectable API grades which are shown on the 10x4" touchscreen.

SVMTM 4001 provides an unbeatable combination of features that simplify the measurement process. Both density cells are equipped with the FillingCheck technology compliant with D4052 requirements, which allows real-time monitoring of the filling quality of the density cells. This saves valuable time as bubbles are already detected during the measurement.

SVMTM 4001 is designed to provide Viscosity Index, kinematic viscosity and density of lubricating oils, base oils and additives, as well as in-service oils. In addition, the optional modular combination with Anton Paar refractometers allows for fast determination of the carbon-type composition of transformer oils with automatic calculations according to ASTM D2140 and D3238. In order to maximise productivity and meet the demands of high throughput, SVMTM 4001 can be used with a 71-position sample changer.

For More Info, email: 40640pr@reply-direct.com