APPLICATION NOTE

ANALYTICAL CONTROLS by PAC

Determination of Trace Thiophene in Refined Benzene by Gas Chromatography and Sulfur Selective Detection (ASTM D7011)

- **Fast analysis**
- **<20 ppb LDL, Repeatability 3% at 0.5 ppm**
- **High level of automation allows unattended, around-the-clock analyses**
- **4 decades of Linear Dynamic Working Range for SCD**

Keywords:
Thiophene, Chromatography, Benzene, SCD, ASTM D7011

INTRODUCTION

Benzene is a natural constituent of crude oil, and is one of the most basic petrochemicals. Benzene is a colorless and highly flammable liquid with a sweet smell. Because it is a known carcinogen, its use as an additive in gasoline is now limited, but it is an important industrial solvent and precursor to basic industrial chemicals including drugs, plastics, synthetic rubber, and dyes. At room temperature, Thiophene is a colorless liquid with a mildly pleasant odor reminiscent of benzene, with which Thiophene shares some similarities. The separation of Thiophene from benzene is difficult by distillation due to their similar boiling points (4 °C difference at ambient pressure).

The majority of Thiophene is removed from the benzene fraction by washing with sulfuric acid, however traced will remain. The analysis of low concentrations Thiophene in benzene is necessary as it is a building block for many other aromatic chemicals.

SYSTEM DESCRIPTION

The AC THIOPHENE IN BENZENE (ASTM D7011) application uses an Agilent Technologies 7890 gas chromatograph configured with:

- a Split / Splitless Inlet (S/SL) for sample introduction
- Electronic Pneumatics Control (EPC) for setting flow and pressure parameters
- a Sulfur Selective Detector (SCD) for signal generation
- an Automatic Liquid Sampler
- a 30 meter capillary wax column

The sample is introduced by syringe into the split/splitless inlet, where it is mixed with clean carrier gas. One part of the blend will be directed to the split vent, while the other part enters the analytical column. The capillary column (wax type) separates the trace Thiophene and the matrix in a temperature-programmed run. Detection is done by the SCD (Antek). The signal-processing of the SCD is combined into the Antek 7090 controller box positioned on the right side of the Agilent 7890 GC.

The Agilent ChemStation software takes care of instrument control, data handling and storage.

CHROMATOGRAPHIC CONDITIONS

Inlet	S/SL
Injection Volume	1 µl
Split Ratio	1:4
Injection Temp	160 °C
Oven initial Temp.	40 °C
Oven initial Time	2 min
Ramp	10 °C / min
Final Temperature	150 °C
Final Time	2 min
Flow mode	Constant flow
Flow	2 ml/min

Column dimensions	30 m x 0.32 mm x 1 µm
Column type	Polyethylene Glycol (Wax)
Detector	Antek 7090 SCD
Detector Temp	950 °C
O_2 flow (oxidation)	10 ml/min
H_2 flow (reduction)	90 ml/min
Make-up flow	150 ml/min
O3 flow	25 ml/min
High Voltage PMT	750 V
Vacuum Furnace	Approx. 300 Torr
Vacuum Detector	Approx. 30 Torr

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PERFORMANCE SPECIFICATIONS

LDL	Typically <20 ppb
Range	20 ppb – 2 ppm
Linearity (R^2)	> 0,999
Repeatability	Better than 3 % RSD at 500 ppb – 2 ppm
(n = 10)	Better than 10 % RSD at 20 ppb – 500 ppb

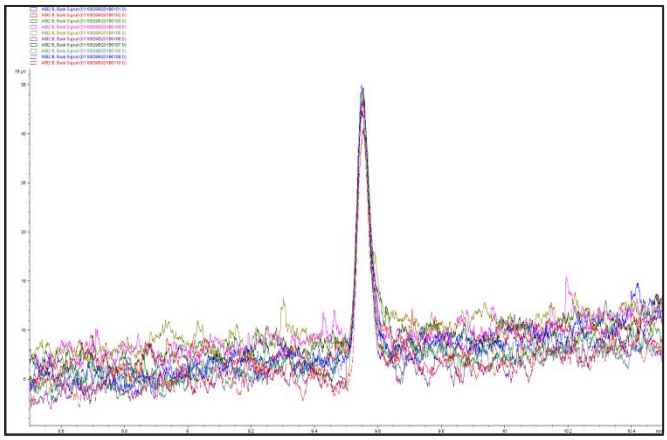

Figure 1. Repeatability 30 ppb Thiophene in Benzene (n=10)

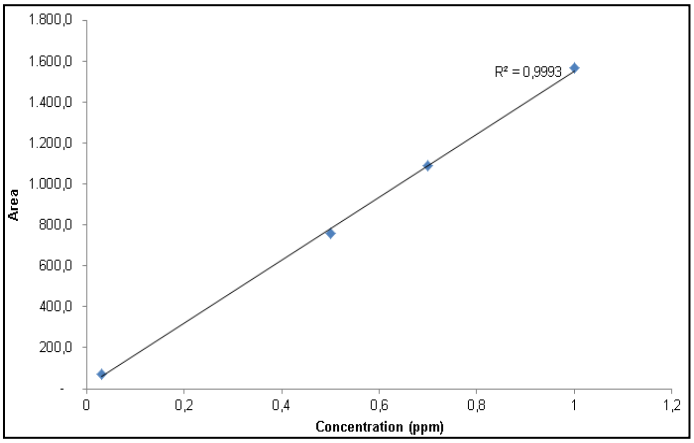

Figure 2. Linearity trace Thiophene in Benzene

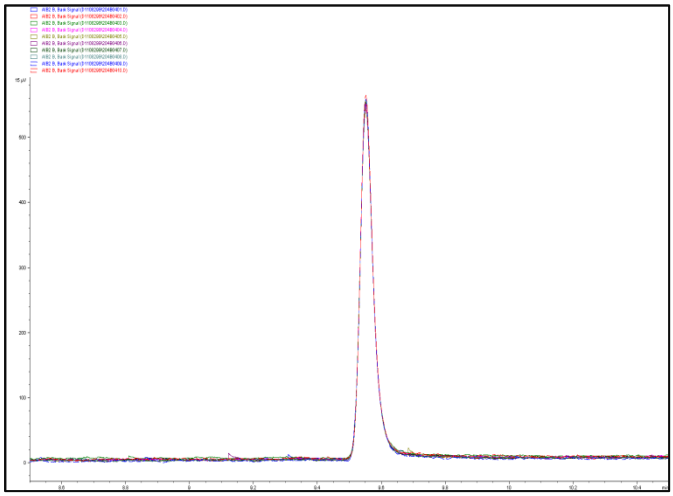

Figure 3. Repeatability 1 ppm Thiophene in Benzene(n=10)

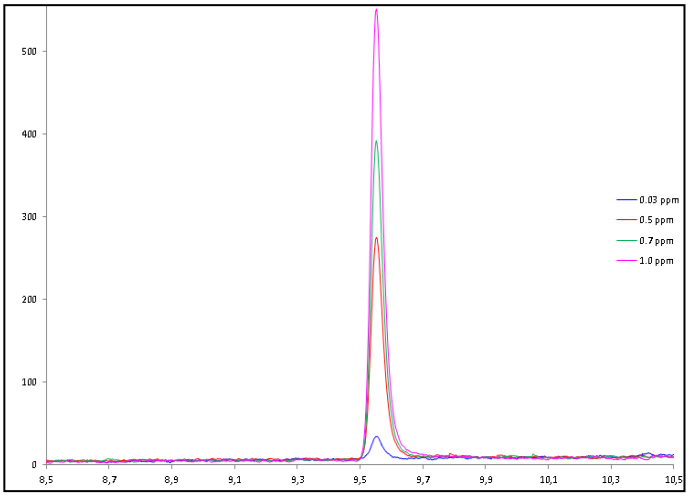

Figure 4. Overlay linearity Thiophene in Benzene

The AC THIOPHENE IN BENZENE ANALYZER is a complete, turnkey system that's completely assembled with all necessary connections and columns. It's ready to go, calibrated, tested to certified calibration blends, fine-tuned and delivered to the customer's facility in 'plug and play' condition. A qualified and dedicated Service Engineer will visit at the customer's convenience after delivery to perform installation and training, so that the customer can begin getting the most benefit out of their Thiophene in Benzene system right away. Plus, PAC's solid guarantee covers all parts and analytical performance during the first year.

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