

Application News

Gas Chromatography

No.G262

Determination of Ethanol Content of Denatured Fuel Ethanol by ASTM D5501-04

With rising concern over the need to reduce carbon dioxide emissions as one measure for controlling global warming, bioethanol is receiving greater attention as an alternative fuel for gasoline.

In the United States, a denaturant is added to fuel grade ethanol, and the quality standard for this denatured fuel ethanol is specified in ASTM Method D4806. As part of this specification, D5501-04 specifies the measurement method and allowable concentrations of methanol as an impurity and ethanol as the principle ingredient.

Furthermore, it also stipulates that gas chromatography

be used for this determination of the methanol and ethanol content in denatured fuel ethanol. Separately, the moisture concentration in fuel is specified in D1364 and E1064, in which the moisture concentration is subtracted from each of the constituent concentrations obtained by gas chromatography to calculate the final methanol and ethanol concentrations.

This Application News introduces an example of analysis of a pseudo denatured fuel ethanol sample by gas chromatography according to ASTM D5501-04.

■ Analytical Method

ASTM D5501-04 specifies that a 100 m or 150 m capillary column be used for the analysis to separate the methanol and denaturant hydrocarbons. Temperature-programmed analysis is conducted from 15 °C with a CRG cryogenic attachment when using a 100 m column, and from 60 °C when using a 150 m column. The analysis time with a 150 m column is somewhat lengthy, but this is offset by the lower cost and convenience of the analysis, since cryogenic cooling is not required when using a 150 m column.

The analytical conditions when using the 100 m and 150 m columns are shown in Tables 1 and 2, respectively. Quantitation of the methanol and ethanol is conducted by the corrected area percentage method using a standard solution consisting of ethanol, to which methanol and n-heptane have been added. However, for peaks other than methanol and ethanol, the calculation is conducted using 1 as the relative sensitivity correction coefficient.

Table 1 Analytical Conditions (column 100 m × 0.25 mm I.D. df = 0.5 μm)

Model	: GC-2010 AF/AOC + CRG-2010 + GCsolution	Air	: 300 mL/min
Column	: Rtx-1 PONA (100 m × 0.25 mm I.D. df = 0.5 μm)	Make Up Gas	: N ₂ , 30 mL/min
Column Temp.	: 15 °C (12 min) - 30 °C/min - 250 °C (19 min)	Det Temp.	: 300 °C
Carrier Gas	: He, 24 cm/sec (Constant Linear Velocity Mode)	Injection Method	: Split
Injection Temp.	: 300 °C	Split Ratio	: 1:200
Detector	: FID	Injection Volume	: 0.5 μL
H ₂	: 30 mL/min		

* In this method, N₂ is specified as the make-up gas to be used with the FID.

Table 2 Analytical Conditions (column 150 m × 0.25 mm I.D. df = 1.0 μm)

Model	: GC-2010 AF/AOC + GCsolution	Air	: 300 mL/min
Column	: Rtx-1 (150 m × 0.25 mm I.D. df = 1.0 μm)	Make Up Gas	: N ₂ , 30 mL/min
Column Temp.	: 60 °C (15 min) - 30 °C/min - 250 °C (23 min)	Det Temp.	: 300 °C
Carrier Gas	: He, 24 cm/sec (Constant Linear Velocity Mode)	Injection Method	: Split
Injection Temp.	: 300 °C	Split Ratio	: 1:200
Detector	: FID	Injection Volume	: 0.5 μL
H ₂	: 30 mL/min		

* In this method, N₂ is specified as the make-up gas to be used with the FID.

■ Sample Analysis

Ethanol spiked with 0.5 % (v/v) methanol and 5.0 % (v/v) commercially-available regular gasoline was prepared for use as a pseudo denatured fuel ethanol sample, and analysis was conducted according to

ASTM D5501-04.

The resultant chromatograms using the 100 m and 150 m columns are shown in Figs. 1 and 2, respectively.

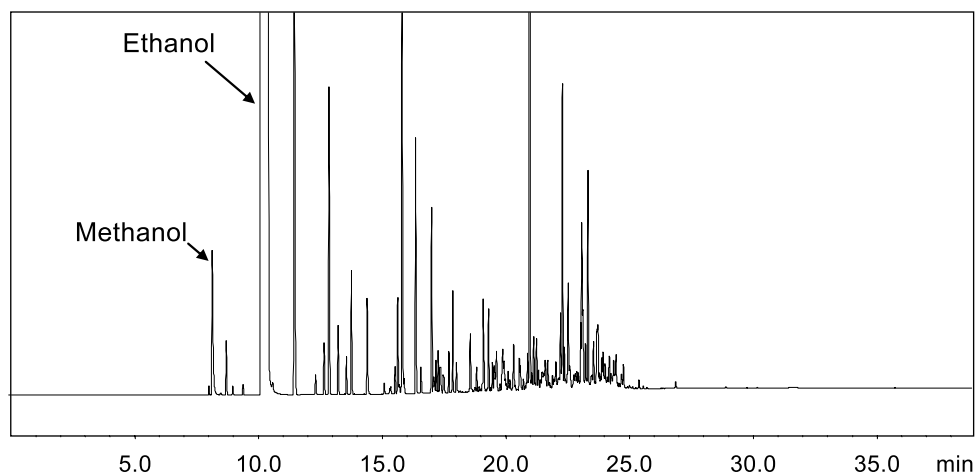


Fig.1 Chromatogram of Denatured Fuel Ethanol
Column: Rtx-1 PONA (100 m × 0.25 mm I.D. df = 0.5 μm)

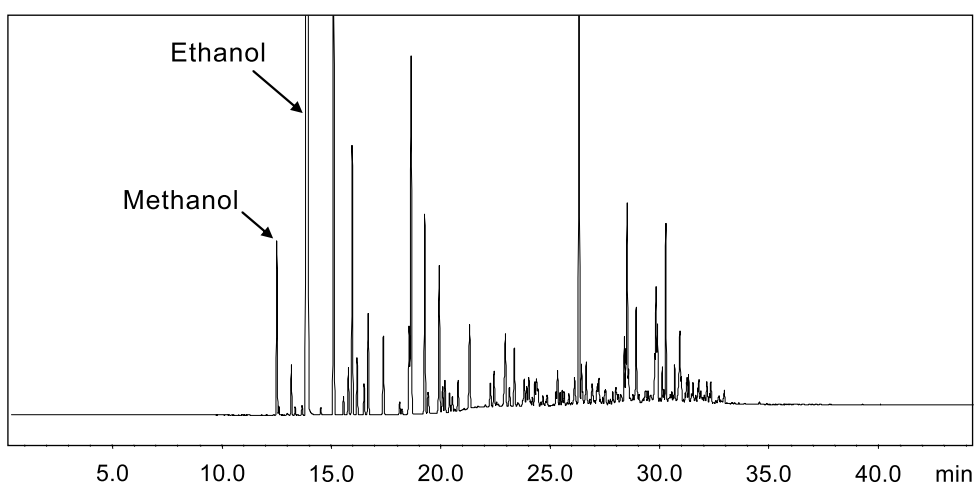


Fig.2 Chromatogram of Denatured Fuel Ethanol
Column: Rtx-1 PONA (150 m × 0.25 mm I.D. df = 1.0 μm)

Please refer to D5501-04 for the detailed operations and conditions.

[References]

- ASTM D5501-04 "Standard Test Method for Determination of Ethanol Content of Denatured Fuel Ethanol by Gas Chromatography"
- ASTM D4806-06 "Specification for Denatured Fuel Ethanol for Blending with Gasolines for use as Automotive Spark-Ignition Engine Fuel"
- ASTM D1364 "Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)"
- ASTM E1064 "Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration"

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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