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Application News

Gas Chromatography

Optimizing Single Injection, Dual Column, Dual FID Blood Alcohol (BAC) Samples with HS GC-FID

No. GC-008

■ Introduction

Blood alcohol (ethanol) analysis (BAC) is routinely performed in forensic laboratories for driver impairment and other casework. This analysis is typically done by gas chromatography with headspace sample (HS-GC) introduction. Historically the GC parameters for this application utilize constant high pressure (approximately 200 kPa) and a temperature of 50°C. In combination, these parameters have caused distortion in the chromatography for analysis with a dual column system and single injection.

Using the Shimadzu GC-2010 Plus or Nexis GC-2030 with HS-20 loop headspace sampler, column splitting, and dual flame ionization detectors (FID), several parameters were tested for system and method optimization to meet the needs of this analysis.

■ Standard and Sample Preparation

A standard mix with six analytes (methanol, acetaldehyde, ethanol, isopropanol, acetone, and N-propanol as the internal standard) was prepared for qualitative analysis of the baseline separation. For quantitative analysis, ethanol standards were prepared in concentrations of 0.01, 0.1, 0.2, 0.3, 0.4, and 0.5 g/dL with 0.013 g/dL of internal standard for the calibration curve.

■ Analytical Conditions

Headspace	HS-20 Loop Model
Operation Mode	Static headspace with loop
Sample	20-mL headspace vial
Sample Oven	80°C
Equilibration	13 min
Sample Loop	1.0 mL
Vial Pressurization	100 kPa
Sample Line Temperature	200°C
Transfer Line Temperature	200°C
Gas Chromatograph	**GC-2010 Plus or 2030 NX**
Injection	Split injection from HS-20, with 20:1 split ratio
Column 1 (Front)	Rtx- BAC Plus 1 – 30.0 m x 0.32 mm ID x 1.80 µm
Column 2 (Back)	Rtx- BAC Plus 2 – 30.0 m x 0.32 mm ID x 0.60 µm
Column Setting (LabSolutions)	30.0 m x 0.32 mm ID x 1.20 µm
Deactivated Column	Restek - 0.32 mm - 0.45 mm
Column Splitter	Restek - MXT union (0.32 mm)
Carrier Gas	Helium
Flow Control Mode	Constant Pressure - 125 kPa
Oven Program	Isothermal - 40°C; hold for 3 min
Detector	**GC-FID (Dual)**
Temperature	220°C
Stop Time	3.00 min
Makeup Gas	Helium
Makeup Flow	30.0 mL/min
H2 Flow	40.0 mL/min
Air Flow	400.0 mL/min

■ Results and Discussion

This BAC method was optimized to 3 minutes from 4 minutes by increasing the constant pressure supply and column oven temperature slightly, while still allowing the conservation of helium. Figure 1 shows the comparison between the optimized method parameters (black) to the initial parameters (red). Initial parameters use a constant pressure of 100 kPa and an oven temperature of 35°C, while the optimized method used a constant pressure of 125 kPa and a column oven temperature of 40°C.

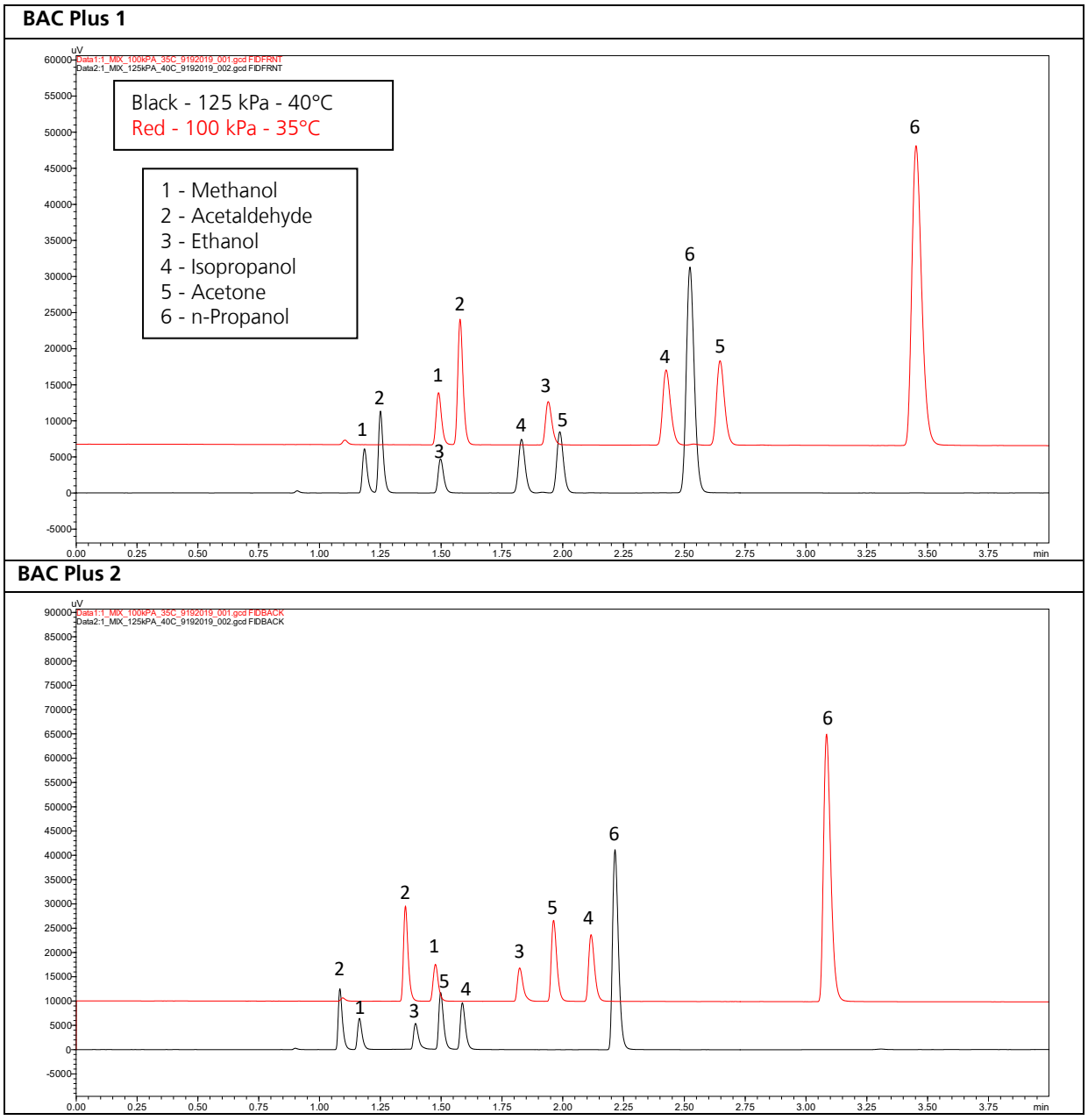

Figure 1: Comparison of 2 methods using BAC Plus 1 and BAC Plus 2 columns

Using the optimal parameters of 125 kPa and 40°C, the internal standard (N-propanol) elutes at 2.5 minutes for BAC Plus 1 and 2.2 minutes for BAC Plus 2. Using the Shimadzu HS-20 headspace sampler, the total injection to injection time is also decreased from over 8 minutes to 5.5 minutes.

Traditional methods use an operating pressure of 200 kPa, which results in a column flow close to 7 mL/min. To conserve helium and reduce column flow, the previous method was developed for a lower column flow (3.5 mL/min), extending the overall run time significantly. This caused long delays between injections and the need for further optimization was required.

Several injections were performed varying the constant pressure and column oven temperature. Once the optimal parameters were determined, the column flow was set to approximately 4.75 mL/min. A column flow over 6.0 mL/min caused poor peak shape (peak tailing) and lack of resolution (less than 2 by USP calculations) primarily for the BAC Plus 2 column. Lowering the flow rate improved the overall chromatography in both columns due to increased interaction with the column phase.

Calibration

Figure 2 shows an overlay comparison of 7 injections including a blank with the internal standard (N-propanol) and increasing concentrations of ethanol.

Figure 3 shows a 6-point calibration of ethanol concentrations 0.01, 0.1, 0.2, 0.3, 0.4, and 0.5 with an R^2 = 0.9997 for the BAC Plus 1 column. The BAC Plus 2 column also yields an R^2 value of 0.9997.

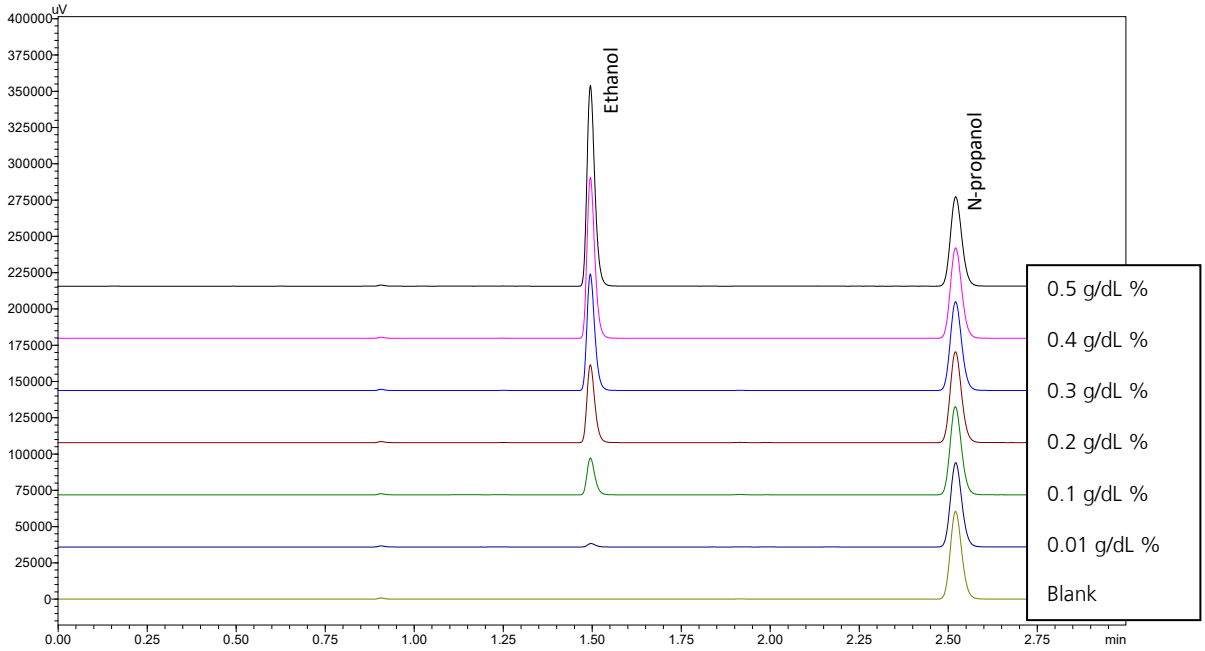

Figure 2: Overlay of Ethanol calibration standards on BAC Plus 1 column

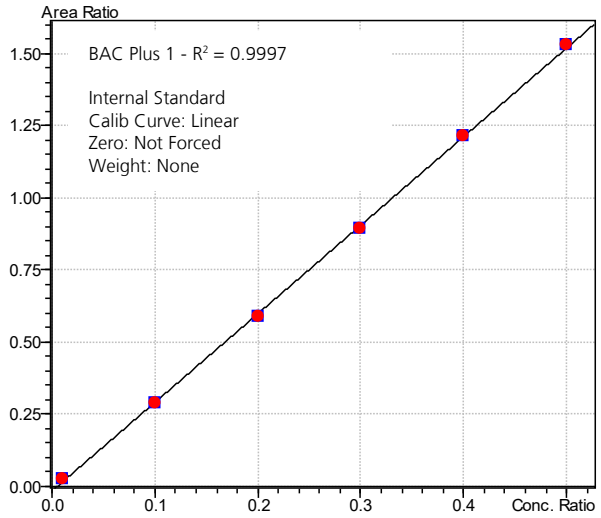

Figure 3: 6-point calibration curve of Ethanol on BAC Plus 1 column

■ Conclusion

To increase BAC sample throughput, a faster method has been developed to allow for an injection to injection time of 5.5 minutes using the Shimadzu GC with HS-20 headspace autosampler. This method allows for the conservation of helium, full resolution of ethanol from other analytes, and preservation of chromatography when split between 2 columns with different film thicknesses.

■ Consumables

- Restek (#18004) - Rtx- BAC Plus 1 – 30.0 m x 0.32 mm ID x 1.80 µm
- Restek (#18006) - Rtx- BAC Plus 2 – 30.0 m x 0.32 mm ID x 0.60 µm
- Restek (#10001) - 0.32 mm - 0.45 mm
- Restek (#21388) - MXT union (0.32 mm)

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