

Analyzing Liquid Fractions of Biogas Processes by HPLC

Application Note

Biofuels

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Introduction

For process control of the ever-growing number of biogas plants, knowledge of acetic and propionic acid concentration is crucial, since high levels of propionic acid can indicate biological problems. Analysis of free fatty acids can be done by GC or by HPLC. This application note shows the analysis of a specimen of a biogas plant liquor using HPLC with an Agilent Hi-Plex H column.



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Materials and Methods

The sample was steam distilled according to *German standard methods for the examination of water, waste water and sludge — Sludge and sediments (group S) — Part 19: Determination of the steam-volatile organic acids (S 19)* and pretreated by filtration through a 0.45 µm membrane before analysis. Since caproic acid is very seldom found in biogas plant liquors, the method can be halted after the elution of isovaleric acid.

Conditions

Column	Agilent Hi-Plex H, 7.7 × 300 mm, 8 µm (p/n PL1170-6830)
Mobile phase	0.005 M H ₂ SO ₄
Gradient	Isocratic
Flow rate	0.7 mL/min
Injection volume	20 µL
Sample concentration	200 mg/mL for each acid
Temperature	60 °C
Pressure	4.6 MPa (46 bar, 670 psi)
Detector	RI (55 °C)

Results and Discussion

Figure 1 shows a separation of a standard mixture of free fatty acids.

Figure 2 shows the main constituents of the biogas liquor, which includes some of the fatty acids in the standard mix.

Conclusion

A sample of biogas plant liquor was successfully separated using HPLC with an Agilent Hi-Plex H column. Hi-Plex H is the column of choice for the analysis of organic acids, using dilute mineral acid as eluent. The use of a ligand-exchange chromatography column such as Hi-Plex H significantly reduces the need for complicated sample preparation (typically involving elution through an ion-exchange resin bed). This is because retention is brought about not only by ion exchange, but also by ion exclusion and partitioning on this type of column.

For More Information

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Peak identification (for both figures)

- | | |
|--------------------|--|
| 1. Formic acid | 7. 3,3-Dimethyl butyric acid (Internal Standard) |
| 2. Acetic acid | 8. Valeric acid |
| 3. Propionic acid | 9. Isocaproic acid |
| 4. Isobutyric acid | 10. Caproic acid |
| 5. Butyric acid | x. Ethanol |
| 6. Isovaleric acid | |

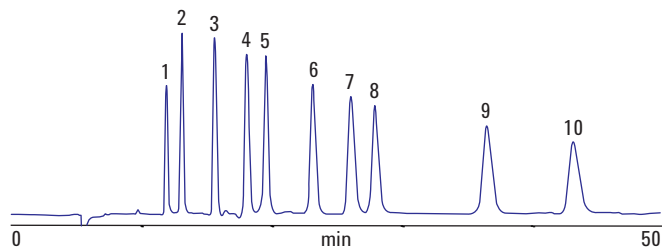


Figure 1. Analysis of a standard mixture of free fatty acids on an Agilent Hi-Plex H column.

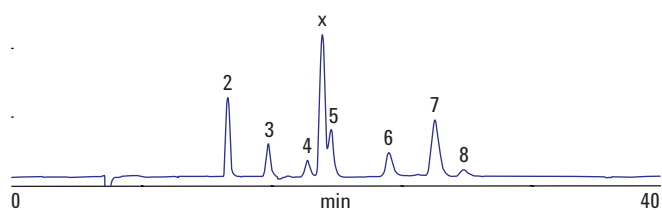


Figure 2. Separation of a biogas plant liquor by an Agilent Hi-Plex H column.

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