The Determination of Ethanol in Fermentation Off-Gas using Real-Time GC with a Modified GC

Lee N. Polite, Jackson O'Donnell, Nikolas L. Polite, Theodore N. Covello, Erick D. Walts, Dennis L. Polite, Mary Beth Smith

Axion Analytical Labs, Inc – Chicago, IL

Introduction

The traditional approach to process monitoring involves sending a production sample to the lab for testing and then waiting for the results. Unfortunately, the results often come back after it is too late to address the problems. Ideally, analytical results should be available while there is time to correct the underlying causes. In this study, we demonstrate how some minor modifications to a benchtop GC can result in an automated, online instrument. Fermenter off-gas was monitored in near real-time by adding a gas sampling valve to a benchtop GC and connecting the sampling valve to the off-gas port of the fermenter. This configuration allowed the operator to monitor the progress of the fermentation as it was happening by simply pressing the GC start button. A custom chromatographic method was developed and optimized to provide results in less than 3 minutes. The system was further automated by employing a sequence table to control the sampling valve. This additional automation provides data every 4 minutes, without having to manually request it. The method also provided early warning of microbial contamination by measuring indicators like acetic acid.

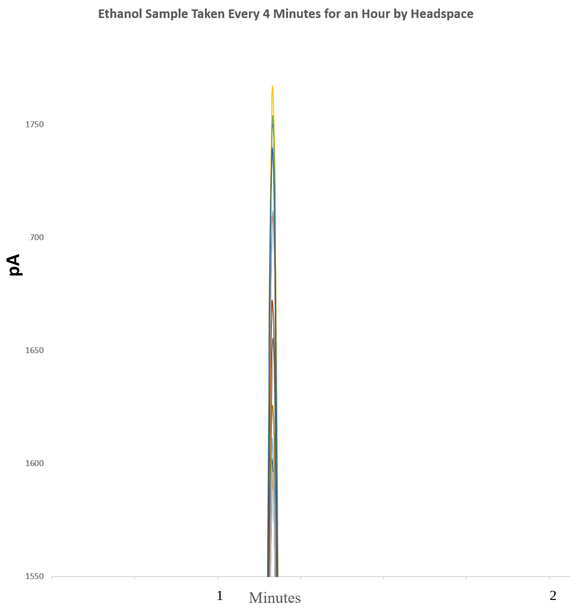
Experimental Conditions

GC: Agilent 6850 GC with a gas sampling valve
Column: J&W DB-ALC1 (modified length) 6 m x 0.32 mm x 1.80 µm
Temperature: Isothermal at 40°C
Flow Mode: Constant Flow
Linear Velocity: 32 cm/sec
Carrier Gas: Hydrogen
Injection Mode: Sampling Valve
Split Ratio: 10:1
Injection Volume: 0.50 mL
Valve Temperature: 150°C
Detector Temp: 250°C

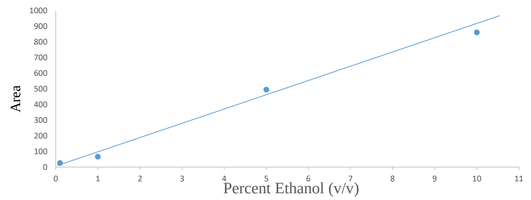
Materials

Fermentation: Dry ale yeast (Safale US-05), dextrose and DI Water
Standards (v/v) absolute ethanol in DI water 0.1%, 1.0%, 5.0%, and 10.0%
A generic heating pad was used to prevent sample condensation in the transfer line

Automated Ethanol Determination



Calibration of Ethanol Standards



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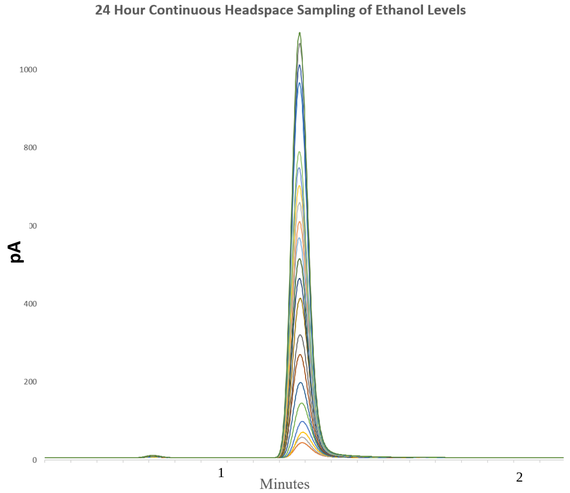
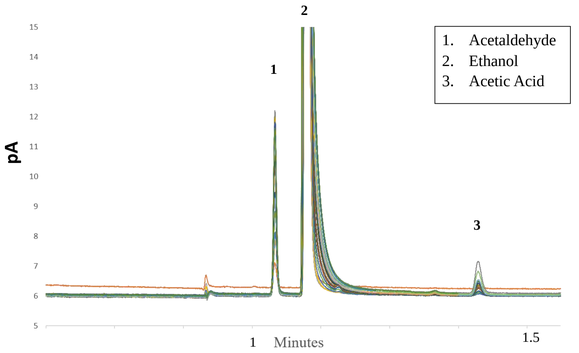
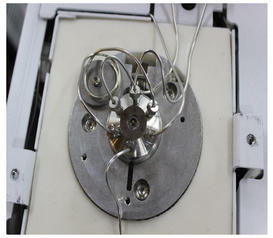
Ethanol Level Monitoring By Gas Sampling Valve



Monitoring Levels of Contamination

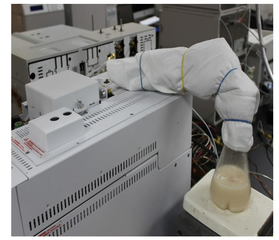


Equipment



Gas Sampling Valve

Fermentation Apparatus



Conclusions

Real time analysis of ethanol and contaminants can be accomplished by adding a sampling valve to a standard bench-top GC. Combine this automated sampling system with a modified fast GC method, and the operator can monitor (and respond to) contamination in real time.

This same approach can be applied to the monitoring of any size or type of reactor, as long as the desired stream is in the vapor state. This approach allows the analyst to convert nearly any GC into an online monitor, simply by adding a sampling valve and adjusting the GC conditions appropriately.

Acknowledgements

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