Micro Analytical Systems Department
Technology–Micropyrolyzer

Fact Sheet

Introduction

A hand-held gas-phase chemical analysis system has been developed that uses three microfabricated stages. The first stage is a preconcentrator that collects and concentrates analytes. The second stage is a gas chromatographic (GC) column used to achieve analyte separation. The third stage is an array of surface acoustic wave (SAW) sensors used to detect the separated analytes. The chemical analysis system called µChemLab™ [1] combines these stages along with a miniature pump (Figure 1). This combination approach provides rapid and discriminating analyses, and a pattern of detector responses providing an additional stage of discrimination. The following describes advances toward a new application for the preconcentrator and additional applications for µChemLab™.

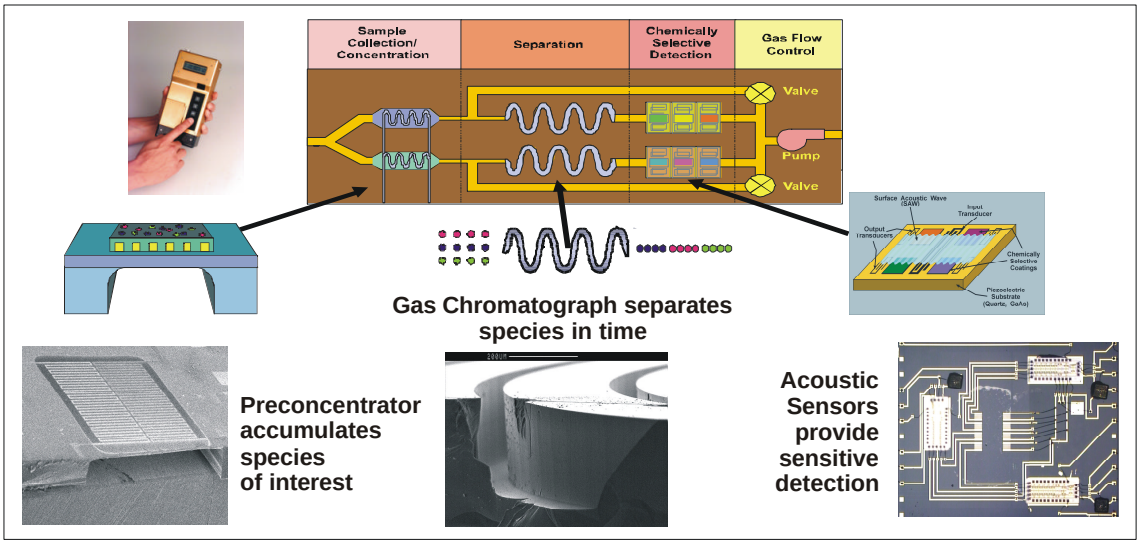

Figure 1: Schematic for the µChemLab™ gas-phase chemical analysis system along with scanned electron micrographs of the microfabricated stages.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

Micropyrolyzer

Based upon the microfabricated stages already demonstrated at Sandia National Laboratories in the µChemLab™ effort, there is ongoing development of miniaturized pyrolysis/GC instrumentation that capitalizes on that expertise.

The microfabricated preconcentrator device can also be used for pyrolysis; this is when a chemical change is brought about by heating. The preconcentrator consists of a "microhotplate" fabricated on a thin dielectric membrane that is capable of pyrolysis due to the low heat capacity and thermal conductivity of the membrane. When the preconcentrator is used in this manner it is called a micropyrolyzer. Two essential characteristics of the micropyrolyzer are time-temperature response and steady-state power consumption, reaching 300°C in less than 30 milliseconds. The micropyrolyzer enables the unique µChemLab™ application described below.

FAME Detection using Micropyrolyzer

Rapid detection and identification of bacteria and other pathogens is important for many civilian and military applications. The profiles of biological markers such as fatty acids can be used to characterize biological samples or to distinguish bacteria at the gram-type, genera, and even species level. The taxonomic significance, or the ability to differentiate one microorganism from another, using fatty acid content and distribution is well known [2]. Bench top methods of extracting, derivatizing, and analyzing fatty acid content are commercially available [3]. These methods chemically derivatize fatty acids to produce more volatile fatty acid methyl esters (FAMEs) as shown in Figure 2.

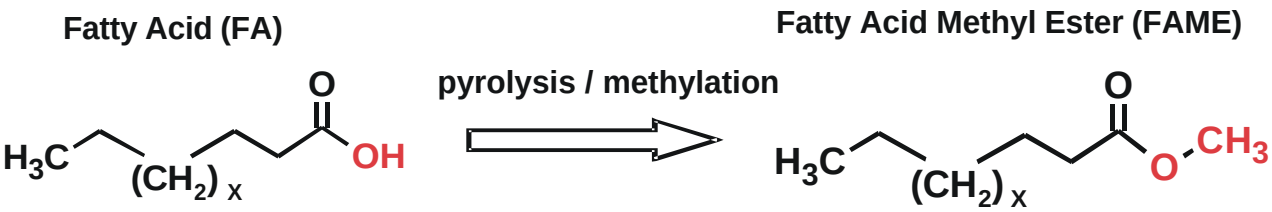

Figure 2: Schematic of general pyrolysis/methylation reaction for free fatty acids.

Methylation is the most common derivatization reaction used because the fatty acid methyl esters, or FAMEs, produced are more easily analyzed and detected than the nascent fatty acids. In the commercial FAME method, analysis is performed by gas chromatography with computer database comparison for identification. The analysis requires a significant amount of instrument time and labor for preparation, extraction, and derivatization. While these methods yield good results, the process is not amenable for field applications. An alternative to extraction/derivatization is the use of pyrolysis (rapid heating). Based upon the microfabricated stages already demonstrated at Sandia National Laboratories, the concept for such a pyrolysis-based, field-deployable device for FAME analysis is shown in Figure 3.

Field-deployable instrumentation for bacterial detection using FAME signals has concentrated to date on using either infrared or resistive heating pyrolysis followed by mass spectral analysis. These methods use large amounts of power in the pyrolysis step. A low-power pyrolyzer and/or chromatograph could reduce the size and power required of existing instrumentation. The initial goal of this work was to determine whether microfabricated components could facilitate a pyrolysis/methylation reaction and therefore demonstrate the potential for a hand-held FAME sensor. A “bare” micropyrolyzer is shown in Figure 4 resting on a dime. The heated area is the 2.5mm square in the center. Pyrolysis/methylation of fatty acids and an edible oil have been demonstrated using only milliwatts of power. FAMEs have also been produced by pyrolysis/methylation of whole cell bacteria using micropyrolyzers.

With respect to the system shown in Figure 3, we have also demonstrated rapid (<5 minutes) separation of relevant FAMEs using a microGC column (Figure 5). Each peak in the chromatogram was well resolved and Gaussian in shape. The small size of the micro GC column allowed rapid temperature ramping while using only small amounts of power. Other stationary phases for the micro GC column are available to tailor the separation to the analyte set.

The ability of a micropyrolyzer to effect the pyrolysis/methylation reaction on fatty acids and the ability of a microGC column to separate FAME species has been demonstrated. The micropyrolyzer advances the portability of FAME analysis with its rapid heating and small size and is a step in the advancement toward an easily portable and/or battery operated bacterial detection system. These results demonstrate the capability of the micropyrolyzer combined with other µChemLab™ components towards fulfilling the goal of a portable, rapid detection and early warning of the presence of pathogens in air or water.

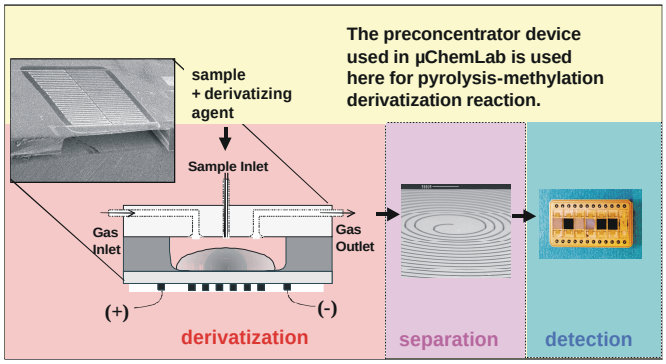

Figure 3: Schematic of the FAME modified µChemLab™

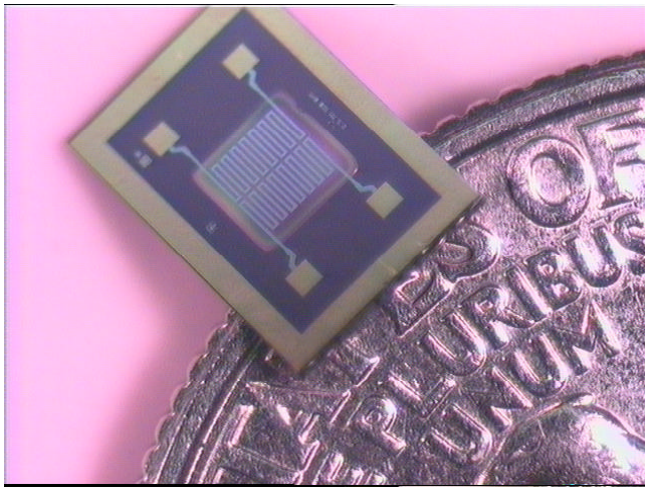

Figure 4: Micropyrolyzer

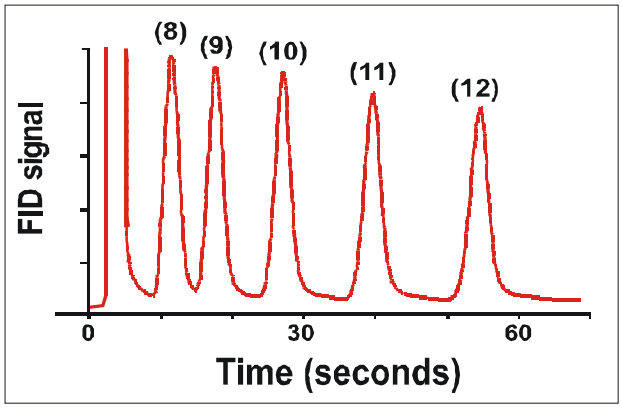

Figure 5: FAME separation (temperature ramped) from 100°C to 140°C using a microGC column using nitrogen carrier gas. FAMEs indicated by main chain length (C8:0 M.E. labeled "8").

References

1. μChemLab Technology Team, “Autonomous Micro-Chemical Analysis Laboratory (μChemLab Technologies),” Sandia Report, SAND2001-1997, Sandia National Laboratories, Albuquerque, NM, Printed July 2001.

2. N. R. Krieg Bergey's Manual of Systematic Bacteriology, Williams & Wilkins, Baltimore,1984

3. Microbial Identification System Operating Manual Ver. 3.0, Microbial ID, Inc., Newark, DE,1993

For additional information or questions, please email us at MGA@sandia.gov