

Application Note: Surfactant Monitoring on Blender Output

Introduction

Semiconductor manufacturing processes utilize aqueous surfactant solutions to clean the silicon wafer surface. In some cases, the surfactant is applied as a plain aqueous solution. In other cases, the cleaning solution additionally contains tetramethyl ammonium hydroxide (TMAH), a highly alkaline cleaning / developing agent.

In order to save on transportation costs, the cleaning solution is typically prepared in-line. A solution of water (or TMAH in water) is fed into an industrial blender, together with a stream of surfactant concentrate. The blender mixes appropriate amounts of both feed streams into a final cleaning solution.

To ensure consistent cleaning results, an on-line analyzer was developed to measure surfactant concentration in the blender output solution. The analyzer uses two alternative sample processing modes to address samples with or without TMAH.

Principle

The FIALab fluidic analyzer draws an aliquot of the cleaning solution from the blender output. If the solution contains TMAH, the sample is sent through a module that removes TMAH and neutralizes the sample pH. (The alkaline nature of TMAH would severely interfere with the subsequent colorimetric detection step.)

The neutralized sample is combined with a buffer solution that sets optimal pH conditions for the colorimetric reaction. Then, a colorimetric indicator is added that reacts with the surfactant in an inverse manner (the presence of surfactant reduces the color intensity of the indicator).

Abs. @ 550 nm



The reaction mixture is passed through a flow cell where absorbance is measured to quantify the color intensity of the solution.

Application benefits

- Automation → On-line monitoring possible
- Real-time operation → Immediate result availability

Experimental

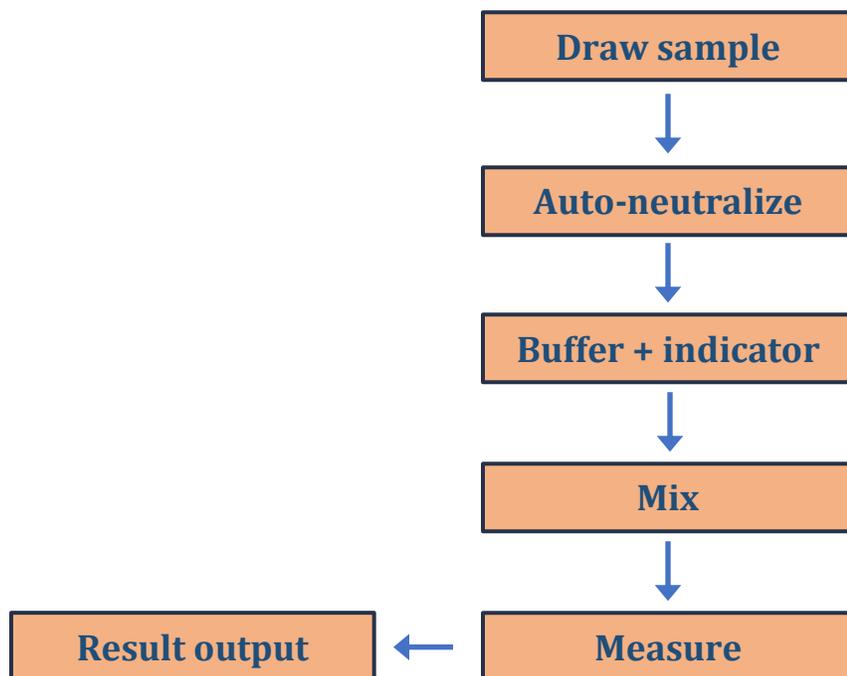
Reagent Composition

- Diluent: Water
- Buffer: Citrate pH 3.9
- Indicator: Proprietary colorimetric indicator

Instrument Configuration

- Analyzer: FIALab Process Analyzer
- Detection: 550 nm, flow-through conditions

Measurement Sequence



Results

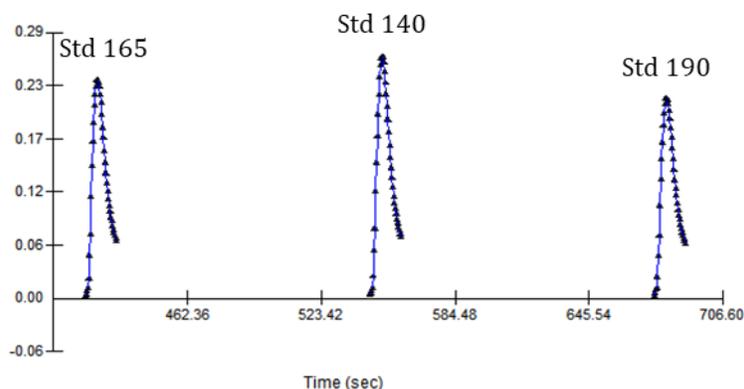


Figure 1 Surfactant response, determined as peak height. Results are shown for measurement of three different surfactant standard solutions (140, 165 and 190 ppm).

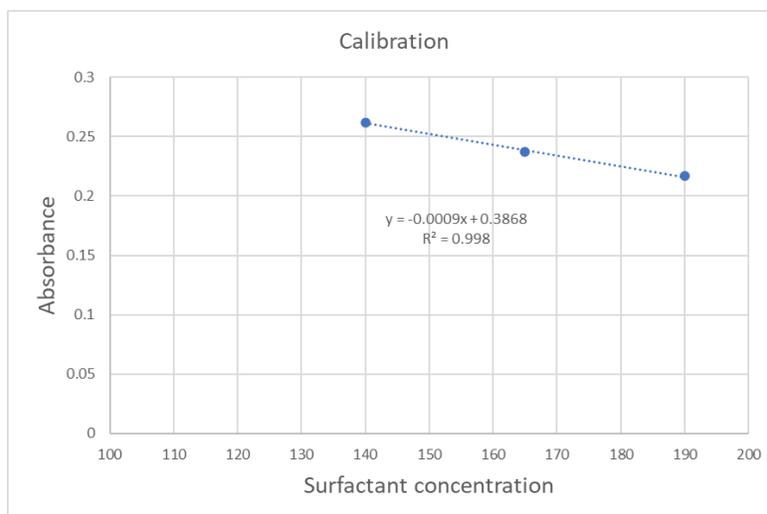


Figure 2 Calibration curve for surfactant concentration. Note the inverse relation between surfactant concentration and indicator absorbance.

Conclusions

The FIALab fluidic analyzer is capable of successful and reliable quantification of surfactant concentrations in solutions with and without TMAH. The setup solves the following obstacles for successful on-line measurements of such solutions:

- Complete, reliable and automated neutralization of TMAH in the sample matrix.
- Reproducible mixing of sample, buffer and indicator for each measurement cycle. (Indicator deteriorates within minutes when in contact with the buffer. As a result, all of the solutions have to be mixed in-line immediately before measurement.)