Invisible-Light Labs

Application Note

NEMS-FTIR-TD WITH EMILIE™

Nanoelectromechanical Fourier Transform Infrared Spectroscopy with in-situ Thermal Desorption

October 17, 2024

1 INTRODUCTION

Nanoelectromechanical Fourier Transform Infrared Spectroscopy (NEMS-FTIR) with EMILIE™ is a unique and powerful analytical tool which allows for in-situ thermal desorption (TD) analysis [1]. Unlike standard themogravimetric method, the analyte is placed directly on the EMILIE™ sampling and sensing chip and monitored in-situ as the sensor is heated, allowing for thermal desorption of semi-volatile compounds and separation of mixtures of analytes [1].

2 METHODOLOGY

Theobromine (Sigma-Aldrich C0750-5G) was sampled from a liquid solution (10 µg/mL in distilled deionized water) using standard aerosol techniques as described in the EMILIE™ sampling guide. The aerosol was generated using a pneumatic jet nebulizer (Meinhard, CytoNeb) with a self-aspiration capillary (20 µL min^{-1}) and pressurized dry air at 3 bar. A spray chamber (Meinhard, CytoSpray) was used for removal of larger droplets. The analyte flow was directed through a diffusion dryer (TOPAS DDU 570/L) prior to sampling by impaction on the EMILIE™ nanomechanical sampling and sensing chip using the EMILIE™ sampling accessory.

Measurements with EMILIE™ are performed at a pressure of $p < 1 \times 10^{-3}$ mbar. The EMILIE™ nanomechanical sampling and sensing chip was subjected to twenty successive ten-minute heating cycles at 80°C. After 10 minutes heating, a spectrum was recorded at 10°C and a new heating cycle was started. The procedure was reported until the theobromine sample was completely desorbed from the surface of the chip.

3 RESULTS

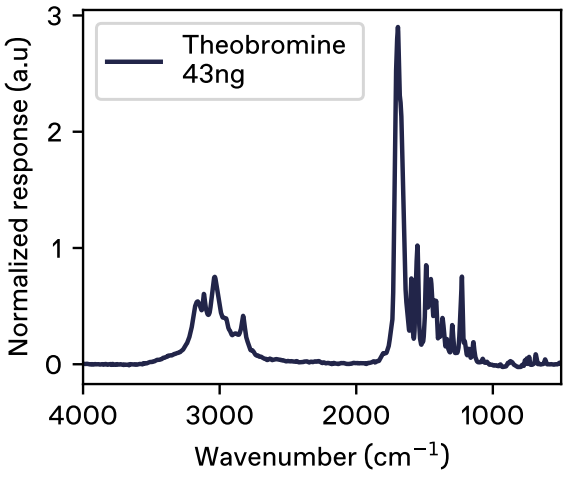


Figure 1: NEMS-FTIR spectrum of 43ng theobromine recorded with EMILIE™ .

Due to thebromine's semivolatility in vacuum, it undergoes sublimation and desorbs from the EMILIE™ nanomechanical sampling and sensing chip during the heating cycle. EMILIE™ allows for the simultaneous monitoring of the process by a combination of photothermal IR spectroscopy (Figure 1), and thermogravimetric analysis (Figure 2).

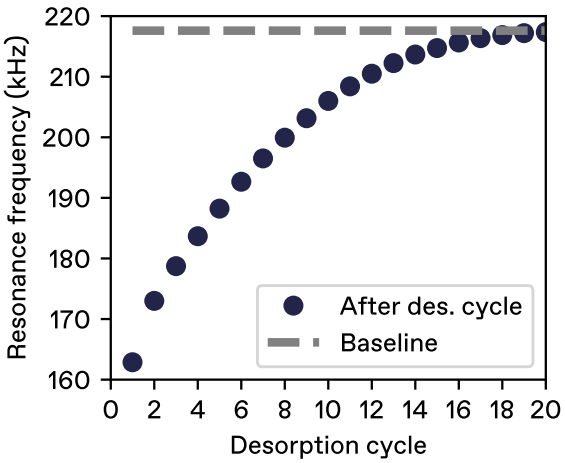


Figure 2: The resonance frequency of the EMILIE™ sampling and sensing chip is recorded after each desorption cycle. The resonance frequency increases until all the theobromine has been desorbed and a frequency plateau is reached.

Assuming an approximately homogeneous mass distribution on the resonator surface, the mass of theobromine present on the surface of the EMILIE™ nanomechanical sampling and sensing chip can be calculated from the recorded resonance frequency [1]. The estimated theobromine mass on the surface of the sensor as a function of the number of heating cycles applied is shown in Figure 3. The evolution of the NEMS-FTIR-TD spectrum is shown as a heat map in Figure 4.

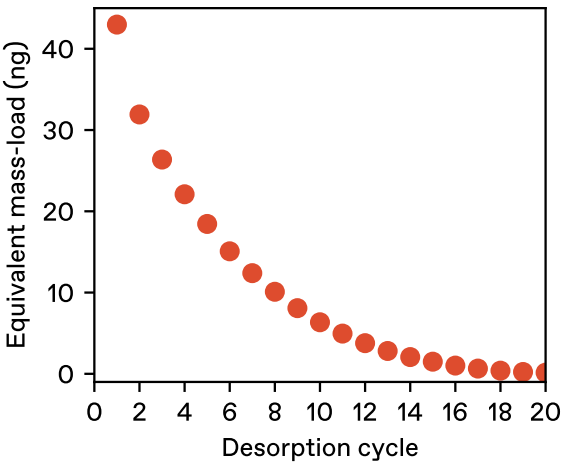


Figure 3: The equivalent mass-load of theobromine can be evaluated from the EMILIE™ sampling and sensing chip's resonance frequency. The theobromine mass present on the surface of the chip decreases with each desorption cycle until all the theobromine has been desorbed.

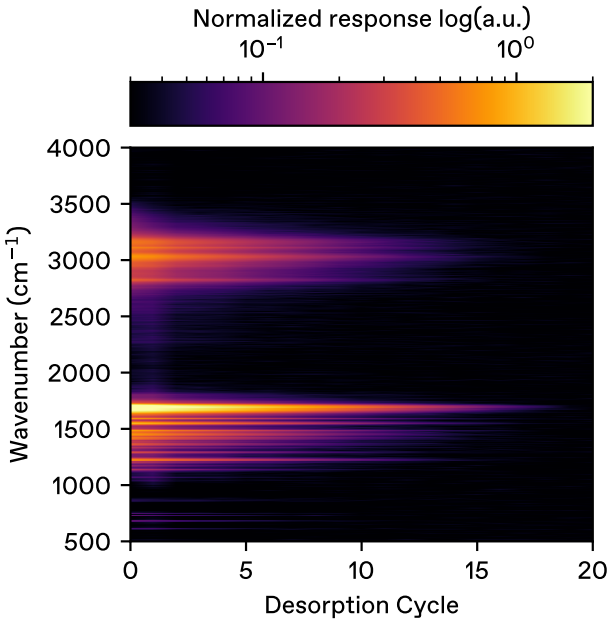


Figure 4: A heat map shows the spectral changes observed in theobromine's NEMS-FTIR spectrum with each desorption cycle until all theobromine has been desorbed from the sensing surface.

These results demonstrate the potential of NEMS-FTIR-TD for the combined desorption and spectroscopic analysis. This opens the possibility of separating and analysing complex mixtures in-situ with picogram sensitivity.

4 BENEFITS OF EMILIE™

- Picogram sensitivity
- in-situ thermal desorption & separation
- No sample transfer

REFERENCES

[1] Niklas Luhmann, Robert G. West, Josiane P. Lafleur, and Silvan Schmid. Nanoelectromechanical infrared spectroscopy with in situ separation by thermal desorption: Nems-ir-td. ACS Sensors, 8:1462–1470, 4 2023.