# NYWGF RESEARCH - FINAL REPORT

**Funding for fiscal year:** 2025-26

## SECTION 1:

**Project title:** Expanding the range of rapid analysis approaches to semi-polar volatiles and non-volatile precursors in grapes

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**Co-PI Collaborators with contact info**:

**New Research ☐ Continued Research ☒**

**Amount Funded $** 91,281

## SECTION 2:

**Project Summary Impact Statement:** Targeted measurements of volatiles or volatile precursors in grapes are useful for grape and wine quality evaluation, but these analyses are often slow and prohibitively expensive for routine use. Over the last several years, the Sacks lab developed a new approach for increasing throughput and decreasing costs for routine volatile analyses by using sorbent sheets (SPMESH) coupled to direct-analysis in real time mass spectrometry (DART-MS).

In 2025, we evaluated a new commercial SPMESH-DART-MS prototype for target grape and wine analytes. We determined that by combining a new mesh design with the new system, we could achieve a 25-fold improvement in detection limit for 3-isobutyl-2-methoxypyrazine (IBMP, “green pepper”), approaching its sensory threshold. Similar improvements were observed for other analytes. Additionally, an optimized approach for analysis of C6 alcohols (“green”) and C8 alcohols (“moldy”) was developed using in-source derivatization. Finally, we established that the analytical performance of SPMESH-DART-MS was not diminished by using smaller sample volumes (1 vs. 5 mL), indicating the method will be compatible with a higher throughput 96-well format. In summary, this work has made progress towards establishing SPMESH-DART-MS as a routine high-throughput method for trace-level volatile analyses.

**Objectives:**

Objective 1: Using an improved turnkey commercial SPMESH-DART-MS system (Bruker EVOQ DART-TQ+), validate newly developed rapid assays for analysis of key grape aroma compounds

Objective 2: Fabricate and test 96-well plate SPMESH devices for rapid analyses of multiple volatile targets

**Materials & Methods:**

The SPMESH-DART-MS has been previously described and is shown in Figure 1. The work used a prototype Bruker EVOQ DART-TQ+ mass spectrometer.

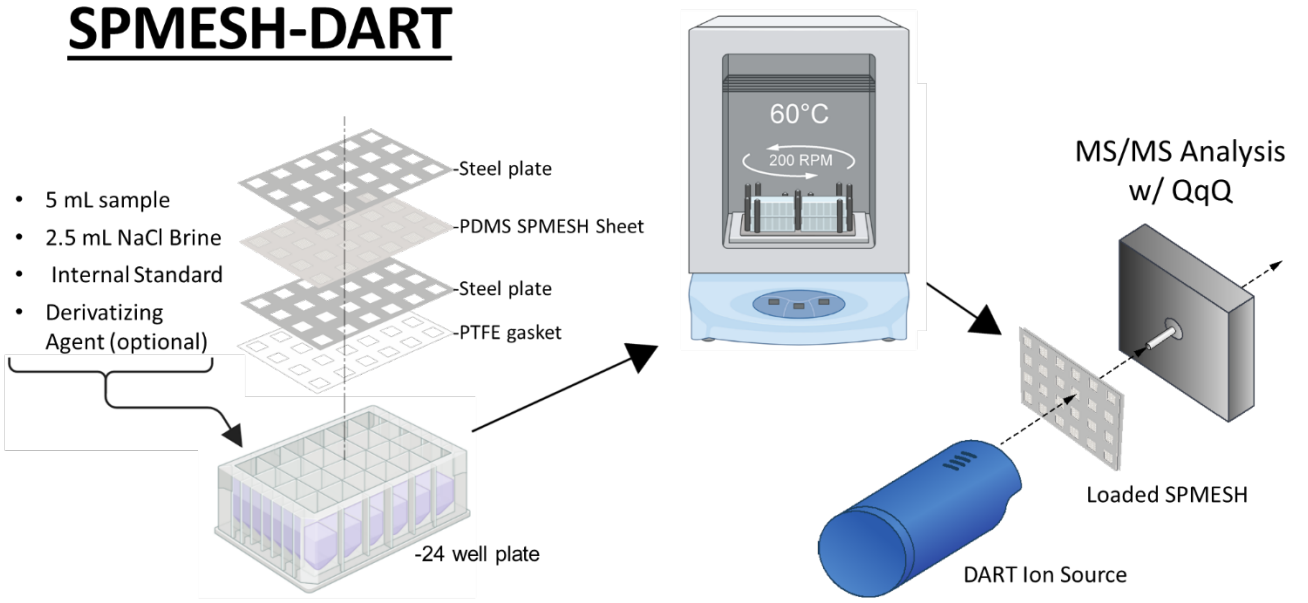



***Figure 1*** *– Overview of SPMESH-DART-MS approach. In this work we evaluated new SPMESH sheet mesh designs, selection of derivatizing agents for C6 and C8 alcohols, and the impact of sample volume.*

For Objective 1, multiple SPMESH mesh sheets differing in aperture hole size and spacing were prepared at the Cornell NanoScale Facility (CNF). The new SPMESH sheets were used to extract 3-isobutyl-2-methoxypyrazine (IBMP, “bell pepper”) and methyl anthranilate (MA, “Concord grape”) prior to DART-MS analyses. Detection limits were then determined.

To optimize conditions for C6 alcohols (“green”) and C8 alcohols (“moldy”), five nitrogenous bases were either to added samples prior to SPMESH extraction, or sprayed onto the SPMESH sheet. Analyses were then performed by DART-MS, and figures of merit (e.g. detection limits, reproducibility, linear range) were determined for a dilution series of standards.

For Objective 2, due to limitations with the Bruker software, we could not use 96 well plates. Instead, we used 24 well plates, and filled the wells with varying volumes (1-5 mL) of solutions containing target analytes. The plates were then analyzed using the optimized SPMESH-DART-MS system.

**Results/Outcomes/Next Steps:**

In 2025, a commercial prototype of a turnkey SPMESH-DART-MS system from an industry collaborator (Bruker) was evaluated. This new unit was selected based on feedback from the Sacks lab which demonstrated that the DART to MS coupling resulted in unacceptable signal losses.

Upon receiving the new instrument, we evaluated the detection limits for IBMP and MA. In the evaluation, we used the original SPMESH sheet design described in previous publications with novel mesh designs. We observed that using larger circular apertures resulted in order-of-magnitude improvements in detection limits for IBMP, and 5-fold improvements for MA (Figure 2).

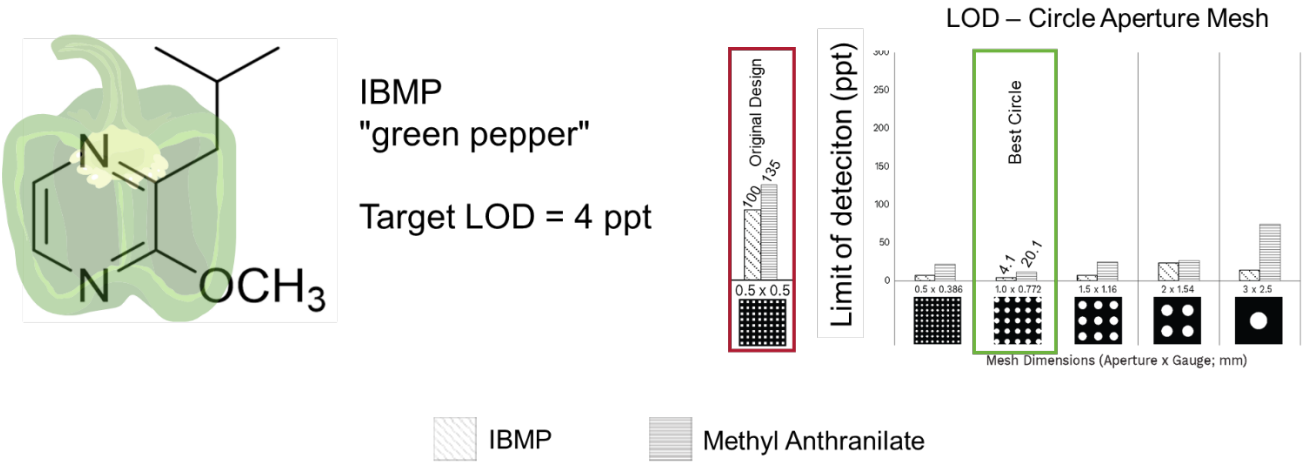
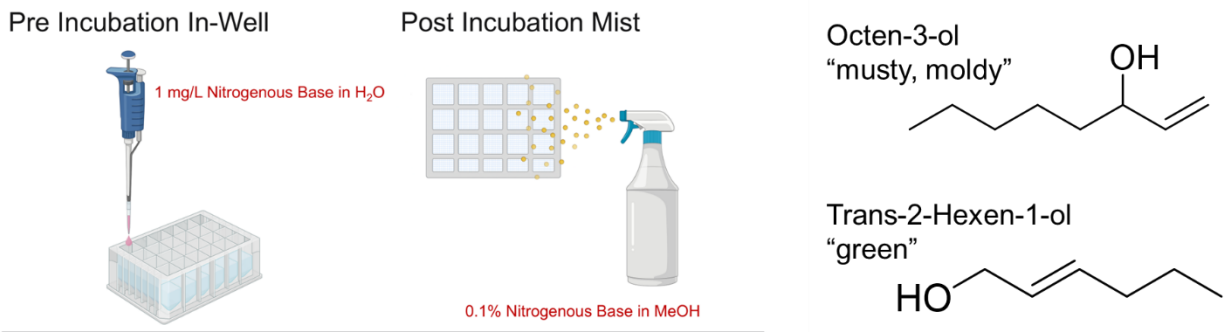



***Figure 2*** *– SPMESH-DART-MS of 3-methyl-2-isobutylmethoxypyrazine (IBMP, "green pepper" aroma) with new turnkey system, comparing different mesh geometries. A new design with larger circular holes (apertures) in place of previous square design resulted in 25-fold improvements in detection limits. Improvements were also observed for methyl anthranilate (MA, "Concord grape" aroma).*

We also used the new instrument to optimize conditions for analysis of C6 ("green") and C8 ("mushroom") alcohols. In 2024, we had determined that adding pyridine or quinoline to samples would enhance sensitivity. In 2025, we evaluated a broader range of related nitrogenous bases, and evaluated adding the base directly to the sample or misting the SPMESH sheet post-extraction (Fig 2).




| Compound | Derivatizing Agent | Application Style | LOD (ppb) | $R^2$ |
|---|---|---|---|---|
| 1-Octen-3-ol | Quinoline | Spray | 2.71 | 0.93 |
| 1-Octen-3-ol | Quinoline | In well | 3.39 | 0.83 |
| 1-Octen-3-ol | Pyridine | Spray | 4.03 | 0.92 |
| 1-Octen-3-ol | Quinuclidine | Spray | 21.7 | 0.58 |
| 1-Octen-3-ol | Pyridine | In well | 26.74 | 0.45 |
| trans-2-Hexenol | Quinoline | In Well | 127.49 | 0.93 |
| trans-2-Hexenol | Quinoline | Spray | 132.33 | 0.69 |
| trans-2-Hexenol | Quinuclidine | Spray | 157.2 | 0.88 |
| trans-2-Hexenol | Pyridine | In Well | 237 | 0.75 |
| trans-2-Hexenol | Pyridine | In Well | 237.21 | 0.75 |
| trans-2-Hexenol | Pyridine | Spray | 328.52 | 0.62 |
| trans-2-Hexenol | 4-DMAP | Spray | 358.39 | 0.71 |

***Figure 3*** *– Optimization of SPMESH-DART-MS measurement conditions for trans-2-hexenol and octen-3-ol using in-source derivatization. Best detection limits were observed with quinoline, either sprayed on the mesh sheet or added to the multiwell plate.*

To evaluate the effect of switching to a 96-well plate format and smaller sample volumes, multiwell plates were loaded with varying amounts of target analytes in different sample volumes (1-5 mL). A blank was also included. We observed no significant effect of sample volume on signal for any analyte. Representative data is shown for methyl anthranilate (MA). This indicates that smaller format 96 well plates (maximum sample volume = 1 mL) should be compatible with SPMESH-DART-MS without a loss of analytical performance (Figure 4).

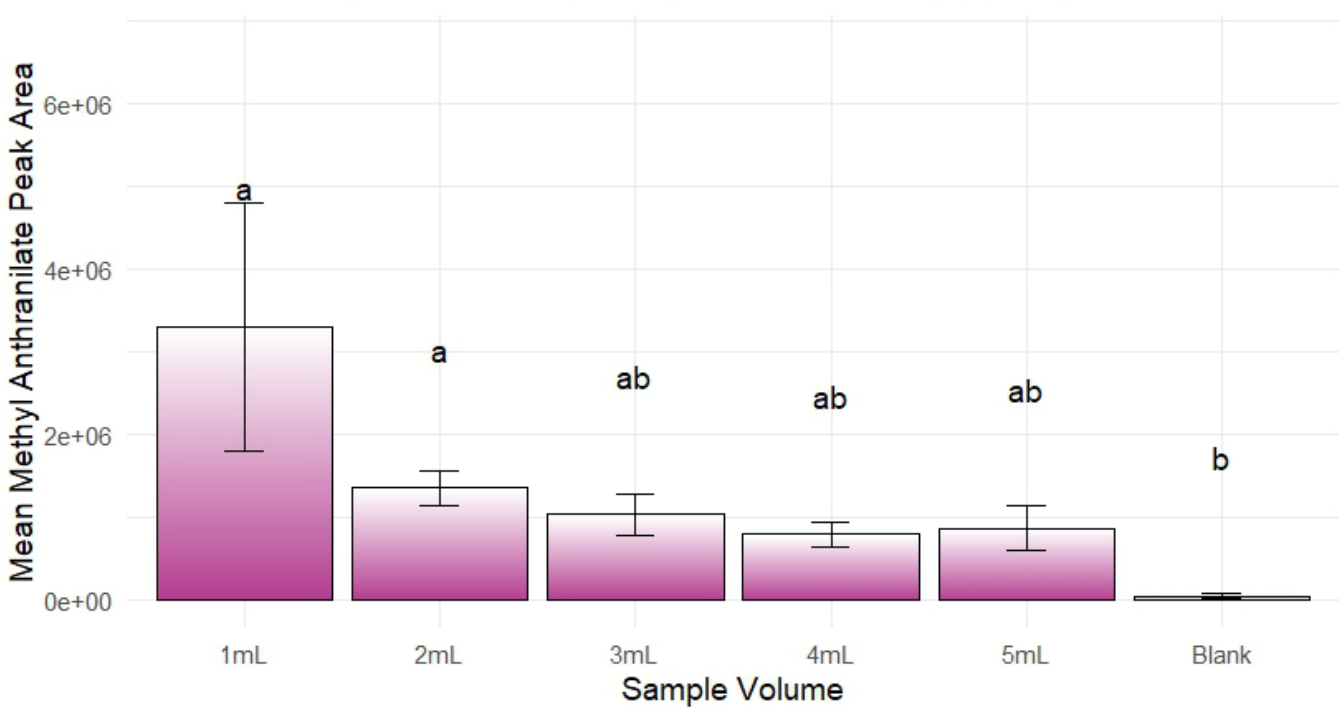



***Figure 4*** *–SPMESH-DART-MS response for methyl anthranilate (MA, "Concord grape") as a function of sample volume. The lack of a significant effect indicates that 96-well plates with 1 mL sample volume will not have a loss of analytical performance as compared to the current 24-well, 5 mL format.*

**Technology Transfer Plan:** Results from this work are currently in preparation for publication in the Journal of Agricultural and Food Chemistry, and we are in discussion of a major winery with operations in New York (E&J Gallo) and with an instrument manufacturer (Bruker) to further commercialize the SPMESH-DART-MS approach. We also filed a provisional patent on work related to SPMESH extraction:

Bates TL, Kalenak AP, Sacks GL. "Manifold for selective sorbent cartridges to facilitate parallel extraction of headspace volatile compounds". **Jun 2025** [Provisional Patent USPTO 63/820,424]

## SECTION 3:

**Project summary and objectives:** Measurement of trace-levels of odorants or their precursors in grapes and wines are useful to winemakers, but existing analytical approaches are prohibitively expensive. We evaluated a new commercial system – based on innovations in our lab – for rapid inexpensive odorant analyses. Using this system, we also investigated new approaches to measurements of odorants in grapes, wines, and fermenting must.

**Importance of research to the NY wine industry:** Analysis of many components of juices and wines, such as acids, sugars and alcohol is routine. However, analysis of most odorants in grapes or wines is prohibitively expensive for many wineries. Decreasing the cost of these volatile analyses will allow for their more frequent usage by winemakers and grapegrowers in decision making.

**Project Results/next steps:** We have demonstrated that the new turnkey SPMESH-DART-MS system coupled with a new mesh design can achieve acceptable detection limits for target grape and wine analytes. Switching to a higher throughput 96-well design will not compromise detection limits. We are currently working with a major winery and instrument company to translate these results so they can be used for routine analysis of samples at commercial wineries.