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Title: Gas Chromatography-Mass Spectrometry Paired with Total Vaporization Solid-Phase Microextraction as a Forensic Tool

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Author Questionnaire

1. **Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **NO**

2. **Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **NO**

3. **Interview statements:** Considering the COVID-19-imposed mask-wearing and social distancing recommendations, which interview statement filming option is the most appropriate for your group? **Please select one**.

   ☒ Interviewees wear masks until videographer steps away (≥6 ft/2 m) and begins filming, then the interviewee removes the mask for line delivery only. When take is captured, the interviewee puts the mask back on. Statements can be filmed outside if weather permits.

4. **Filming location:** Will the filming need to take place in multiple locations? **No**

Current Protocol Length

Number of Steps: 8
Number of Shots: 18

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **John Goodpaster:** The TV-SPME protocol is significant because it can analyze a wide range of samples including drugs, racing fuels, explosive materials, and polycyclic aromatic hydrocarbons. No filtration is needed because only volatile analytes will vaporize. So, dirty samples or suspensions can be analyzed. Various sample matrices can be used including organic or aqueous solvents.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.2. **John Goodpaster:** The main advantage of the TV-SPME technique is its high sensitivity. TV-SPME demonstrates better sensitivity than standard liquid injection, headspace SPME, and immersion SPME. TV-SPME also has the benefit of utilizing large sample volumes without any changes to the GC instrumentation.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

OPTIONAL:

1.3. **Kymeri Davis:** TV-SPME is a very simple technique to perform. The main difficulty comes from ensuring the proper volume has been delivered. The use of small manual or electronic syringes can help, as well as taking your time while sampling. It can also be difficult to ensure the fiber maintains its coating, so fibers must be monitored closely for degradation.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.4. **Kymeri Davis:** Visual demonstration is crucial because both both manual and robotic manipulations are required to be successful.

1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Protocol

2. General TV-SPME sample preparation and GC-MS analysis

2.1. To begin, extract or dissolve the solid sample in enough solvent to reach the desired concentration **[1]**. After ensuring that the sample is fully dissolved, calculate the volume needed to fully vaporize it at the chosen temperature **[2]**.

2.1.1. WIDE: Establishing shot of talent dissolving the sample.

2.1.2. Talent calculating the volume. *Video Editor: Show equation 3 here.*

2.2. Transfer this sample volume into a headspace vial **[1]** and secure the cap **[2]**. If derivatizing the sample, prepare the proper derivatization agent by placing approximately 1 milliliter of the agent into a headspace vial **[3-TXT]**. *Videographer: This step is important!*

2.2.1. Talent transferring the sample to a headspace vial.

2.2.2. Talent securing the cap.

2.2.3. Talent placing derivatization agent into a headspace vial. **TEXT: CAUTION: Derivatization agents are toxic and should be handled in a fume hood**

2.3. Set the proper incubation and extraction temperature, ensuring total vaporization, sufficient sample extraction, and complete derivatization **[1]**.

2.3.1. Talent setting the temperatures.

2.4. Select GC-MS parameters based on the class of the compounds of interest **[1]**. Ensure that the proper inlet liner is in the GC inlet **[2]** and that the SPME fiber has been properly conditioned and is in good working order before beginning the analysis **[3]**.

2.4.1. Talent setting the GC-MS parameters.

2.4.2. Proper inlet liner.

2.4.3. SPME fiber.

3. Gamma hydroxy butyrate (GHB) and gamma-butyrolactone (GBL) sample preparation

3.1. Prepare a sample of gamma hydroxy butyrate or gamma-butyrolactone in water with a concentration of less than 1 part per million **[1]**. Transfer 1 microliter of the sample to a 20-milliliter headspace vial **[2]** and cap the vial immediately **[3]**.

3.1.1. Talent preparing a sample of GHB or GBL.

3.1.2. Talent transferring the sample to a headspace vial.

3.1.3. Talent capping the vial.

3.2. Place 1 milliliter of BSTFA with 1% trimethylchlorosilane into a separate 20-milliliter headspace vial **[1-TXT]** and cap it **[2]**.

3.2.1. Talent placing BSTFA into a headspace vial. **TEXT: CAUTION: BSTFA is toxic and should be handled in a fume hood**

3.2.2. Talent capping the vial.

4. GC-MS parameters and setup for GHB and GBL in water

4.1. Create a GC-MS method by setting the initial oven temperature to 60 degrees Celsius for 1 minute, the oven program to 15 degrees per minute, the final oven temperature to 280 degrees for 1 minute, the flow rate to 2.5 milliliters per minute, and the inlet and transfer line temperatures to 250 and 280 degrees, respectively **[1]**.

4.1.1. SCREEN: Talent creating the GC-MS method. NOTE: Not uploaded, so please use videographer footage. *Videographer: Please film the screen for this shot as a backup.*

4.2. Ensure a narrow SPME inlet liner has been placed inside the GC inlet **[1]** and that the PDMS-DVB SPME fiber has been properly conditioned and is in good working order **[2]**, then run the GC-MS on the sample **[3]**.

4.2.1. SPME inlet liner inside the GC inlet.

4.2.2. Properly conditioned PDMS-DVB SPME fiber.

4.2.3. Talent running the sample.

Results

5. Results: Total vaporization solid phase microextraction (TV-SPME) of GHB and GBL

5.1. A GBL volume study was performed to demonstrate the sensitivity of TV-SPME compared to headspace and immersion SPME. Overall, sample volumes that allowed for TV-SPME demonstrated more sensitivity than headspace or immersion SPME for GBL in water **[1]**. A comparison of the chromatograms for each method is shown here **[2]**

5.1.1. LAB MEDIA: Figure 2.

5.1.2. LAB MEDIA: Figure 3.

5.2. Samples of wine spiked with an effective dose of GHB and GBL were analyzed. These samples also show the interconversion of GBL and GHB **[1-TXT]**. When TV-SPME is performed properly, a sharp, abundant peak is observed **[2]**.

5.2.1. LAB MEDIA: Figures 4 and 5. Video Editor: Label 4 "**8 mg/mL GHB in wine**" and 5 "**10 mg/mL GBL in wine**".

5.2.2. LAB MEDIA: Figure 6.

5.3. TV-SPME has high sensitivity, therefore proper concentrations should be used as to not overload the column. Peak asymmetry occurs when high concentrations are present. In these cases, diluting the sample or using a split injection can improve peak shape **[1]**.

5.3.1. LAB MEDIA: Figure 7.

Conclusion

6. Conclusion Interview Statements

6.1. **John Goodpaster:** TV-SPME could be paired with liquid chromatography, which would dramatically expand the range of detectable analytes.

6.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

6.2. **Kymeri Davis:** This technique is being developed for different fiber geometries such as hollow fibers, or “inside out SPME”, and other configurations.

6.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Acknowledgements Title Card

6.3. This research was supported by the National Institute of Justice (Award No. 2015-DN-BX-K058 & 2018-75-CX-0035). The opinions, findings, and conclusions expressed here are those of the author and do not necessarily reflect those of the funding organizations.