



Application Note AN-RS-015

Trace Detection of Melamine in Dairy Products

Protecting consumer safety with Misa

The illicit addition of melamine to milk due to its apparent enhancement of protein content in foods attracted worldwide attention in 2008. It was discovered at this time that melamine was being deliberately added to raw milk at collecting stations in rural China. Thousands of young children and infants that consumed formula produced from melamine-tainted milk experienced kidney damage and death. As a result, both daily intake limits and increased monitoring of melamine in dairy products were

established globally.

Misa (Metrohm Instant SERS Analyzer) provides quick, easy, and robust detection of melamine in a complex food matrix. As a direct test with no additional reagents, Misa's assay format requires minimal user training, in contrast to standard analytical tests for detecting melamine, including capillary electrophoresis, GC-MS, LC-MS, and immune-based assays.

INTRODUCTION

Melamine is used in the production of industrial materials such as kitchenware, building materials, paints, and paper products.

Melamine's high nitrogen content can enhance the apparent protein content of animal feed and foods for human consumption, notably in dairy products. Unfortunately, this provides motivation for food adulteration for enhanced profits at the expense of human health.

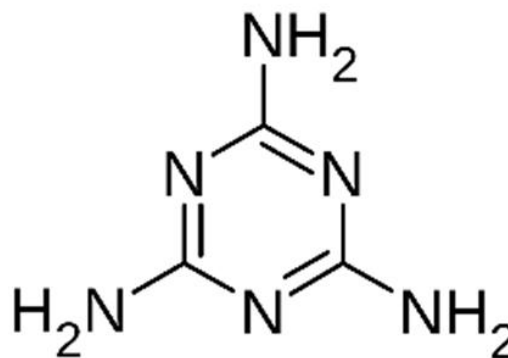


Figure 1. Melamine's nitrogen-rich structure.

REFERENCE MATERIAL AND LIBRARY CREATION

To establish a standard SERS reference spectrum for melamine, a pure standard is analyzed on silver (Ag) P-SERS strips. The unique spectrum for melamine as shown in Figure 2 can be used to create a library entry for the analyte.

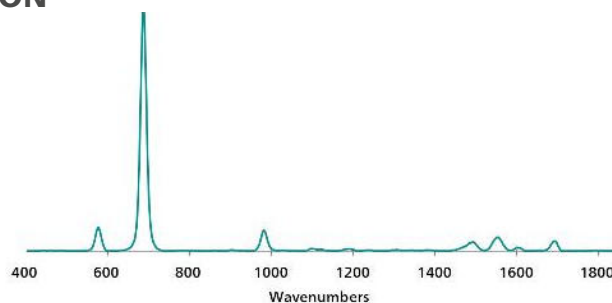


Figure 2. Standard Ag P-SERS reference spectrum of melamine.

EXPERIMENT

Light cream bought from a local grocery was spiked with melamine and tested directly to simulate an authentic scenario of a tainted dairy product. A stock solution of melamine dissolved in methanol was added to light cream to yield samples ranging from

1–500 µg/mL melamine concentration.

10 µL of each sample was pipetted directly onto Ag P-SERS strips, dried briefly, and inserted into the P-SERS attachment on Misa for analysis.

Table 1. Experimental Parameters

| Instrument | | Acquisition | |
|------------------------|------------------|-------------|-----|
| Firmware | 0.9.33 | Laser Power | 5 |
| Software | Misa Cal V1.0.15 | Int. Time | 5 s |
| Misa P-SERS Attachment | 6.07505.030 | Averages | 5 |
| ID Kit - Ag P-SERS | 6.07506.470 | Raster | ON |

RESULTS

The overlaid spectra in **Figure 3** demonstrate reliable detection of melamine in light cream with Ag P-SERS

down to 5 µg/mL, based on the most prominent peak of melamine at 685 cm⁻¹.

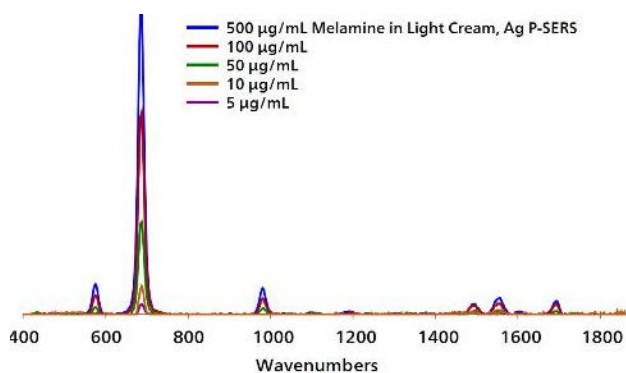


Figure 3. Baselined, background-subtracted spectra of melamine in light cream at various concentrations.

FIELD TEST PROTOCOL

Detection of melamine in the field

Using a pipette, add 1 drop of raw dairy product onto the colored portion of a Silver P-SERS strip. Insert into

P-SERS attachment on Misa for measurement.

Table 2. Requirements for Field Test Protocol

| | |
|--------------------|-----------------------|
| ID Kit - Ag P-SERS | 6.07506.470 |
| includes: | Silver P-SERS |
| | Scoop |
| | Disposable pipettes |
| | 2 mL glass vials |
| Reagents | None |
| Test settings | Use ID Kit OP on Misa |

CONCLUSION

The detection of melamine in a distinctly challenging dairy product matrix has been demonstrated using Misa Ag P-SERS substrates.

Misa provides rapid, high-throughput, and cost-effective on-site identification of food contaminants and adulterants.

CONTACT

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CONFIGURATION



MISA Advanced

Metrohm Instant SERS Analyzer (MISA) is a high performance, portable analyzer system used for rapid, trace level detection / identification of illicit materials, food additives and food contaminants. MISA features a high-efficiency spectrograph equipped with Metrohm's unique Orbital-Raster-Scan (ORS) technology. It has a minimal footprint and extended battery life, perfect for on-site testing or mobile laboratory applications. MISA offers various Laser Class 1 attachments for flexible sampling options. Analyzer operation is available through BlueTooth or USB connectivity.

The MISA Advanced package is a complete package that allows the user to perform SERS analyses using Metrohm's nanoparticle solutions and P-SERS strips.

The MISA Advanced package includes a MISA Vial Attachment, a P-SERS Attachment, a ASTM Calibration Standard, a USB Mini Cable, a USB Power Supply and MISA Cal software for operating the MISA instrument. A ruggedized protective case is also provided to securely store the instrument and accessories.



ID Kit – Ag P-SERS

The ID Kit - Ag P-SERS contains the components a Mira / Misa user requires to perform a SERS analysis using silver P-SERS strips. The kit contains a disposable spatula, dropper, sample vials and 2 silver P-SERS strips.