



Application Note AN-RS-029

Trace Detection of Malathion on Corn

Protecting consumer safety with Misa

Malathion is an insecticide widely used on a broad spectrum of plant species. Although classified as having low toxicity, ingestion or acute skin exposure can be dangerous to humans. Several studies have implicated chronic exposure to malathion in the development of certain cancers. Maximum residue limits for malathion have been enacted by the regulatory agencies of several countries: the US Food and Drug Administration sets maximum residue limits at $8 \mu\text{g}/\text{g}$ in foods, while the EU has a considerably

more stringent limit of $20 \text{ ng}/\text{g}$.

SERS is an accepted method for detection of malathion on fruit and vegetable surfaces. Misa (Metrohm Instant SERS Analyzer), which requires minimal laboratory chemicals and consumables and provides an extremely user-friendly interface, is an excellent SERS solution for trace detection of food adulterants. Misa is a formidable advancement in cost and convenience related to food safety testing and data analysis.

INTRODUCTION

This application note describes a simulated test procedure for detecting malathion sprayed on corn kernels. The assay is based on the acquisition of SERS-

specific spectra for malathion in acetone extracts using Misa and gold nanoparticles (Au NPs).

REFERENCE SPECTRUM AND LIBRARY CREATION

To establish a reference spectrum, a pure malathion standard at 100 µg/mL in ethanol was analyzed using

Au NPs. The unique SERS spectrum shown in **Figure 1** can be used to create a library entry for malathion.

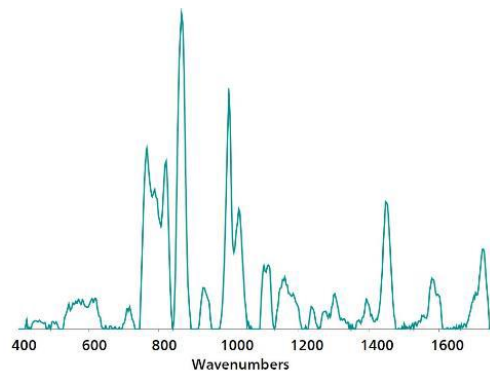


Figure 1. Standard Au NP SERS reference spectrum of malathion.

EXPERIMENT

A stock solution of malathion in acetone was sprayed onto field corn kernels to yield a range of test samples from 1–20 µg/g (concentrations calculated with respect to mass of corn kernels). 0.25 mL of acetone was added to samples in capped glass vials, which were swirled occasionally over a period of 10 minutes to facilitate extraction. At the end of this time, 100 µL of the acetone extract was transferred to a second glass vial and dried on a hot plate. The residue was resuspended in 450 µL of Au NPs and 50 µL of 0.5 mol/L NaCl, the vial was gently shaken to mix, and then immediately placed into the vial attachment on Misa for measurement.



Table 1. Experimental parameters

Instrument		Acquisition	
Firmware	0.9.33	Laser Power	5
Software	Misa Cal V1.0.15	Int. Time	10 s
Misa Vial Attachment	6.07505.040	Averages	10
ID Kit - Au NP	6.07506.440	Raster	ON

RESULTS

Overlaid baseline-corrected spectra acquired for malathion recovered from corn kernels reveal high-resolution malathion detection down to 5 $\mu\text{g/g}$ (Figure 2). Note at these low concentrations, SERS

spectra are very sensitive to any other SERS-active moieties present, including organic materials dissolved with the target compound and the SERS substrate itself.

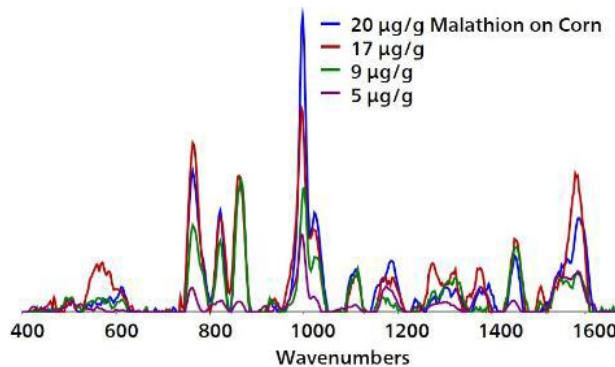


Figure 2. Overlaid baseline-corrected spectra acquired from Au NPs show detection of malathion on field corn kernels to 5 $\mu\text{g/g}$.

FIELD TEST PROTOCOL

Detection of malathion in the field

Place 2–3 pieces of corn in a vial and fill the vial halfway full with acetone, and allow to rest for 5 to 10 minutes, swirling occasionally. With a pipette, remove half of the solvent to a *clean, dry vial* and evaporate to

dryness on a hotplate. Fill this vial halfway full with Au NPs and add 2 drops of NaCl solution, then cap and shake the vial gently to mix. Insert into the vial attachment on Misa for measurement.

Table 2. Requirements for field test protocol

ID Kit - Au NP	6.07506.440
includes:	Gold nanoparticles (Au NP)
	Scoop
	Disposable pipettes
	2 mL glass vials
Reagents	
Acetone	
NaCl solution	3 g NaCl in 100 mL water
Test settings	Use ID Kit OP on MISA

CONCLUSION

Misa offers a facile, green, and economical method for sensitive detection of malathion sprayed on corn ears. Based on the unoptimized protocol reported in this application note, Misa provides sufficient sensitivity

for detecting malathion at concentrations that approximate or exceed the US maximum residue limit for sweet corn.

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CONFIGURATION



MISA Advanced

Metrohm Instant SERS Analyzer (MISA) est un système d'analyse portable hautement performant pour détecter ou identifier rapidement des traces de substances illicites, d'additifs et de contaminants alimentaires. MISA possède un spectrographe très efficace doté de la technologie ORS (Orbital Raster Scan) unique de Metrohm. Son encombrement est minimal et la durée de vie prolongée de la batterie en fait le système d'analyse idéal pour les tests sur site ou les applications de laboratoire mobiles. MISA propose divers accessoires laser de classe 1 pour des options d'échantillonnage flexibles. L'appareil d'analyse peut fonctionner via la connectivité Bluetooth ou USB.

Le module MISA Advanced est un ensemble complet qui permet à l'utilisateur d'effectuer des analyses SERS avec les solutions de nanoparticules de Metrohm et des bandelettes réactives P-SERS.

Le module MISA Advanced contient un embout de flacon MISA, un embout P-SERS, un standard de calibration ASTM, un câble USB mini, un bloc d'alimentation USB et le logiciel MISA Cal pour le fonctionnement de l'appareil MISA. Une mallette de protection robuste est également fournie pour ranger l'appareil et ses accessoires en toute sécurité.



Kit d'identification – Au NP

Le kit d'identification Au NP comprend les composants nécessaires à un utilisateur Mira/Misa pour une analyse SERS avec une solution d'or colloïdal. Le kit se compose d'une spatule à usage unique, d'une pipette compte-gouttes, d'un petit flacon d'échantillon et d'un flacon d'or colloïdal