



Application Note AN-RS-024

Trace Detection of Pyrimethanil in Wine

Protecting consumer safety with Misa

Pyrimethanil is a broad-spectrum fungicide. As grapevines are susceptible to fungal pathogens, large-scale viticulture operations apply pyrimethanil as part of a mixed treatment. Although chemical analysis of wines post-fermentation finds low to undetectable amounts of residue, pyrimethanil is a suspected human carcinogen and chronic exposure can result in multi-organ toxicity in some animal species. The US FDA and EU have established a maximum permissible level of 5 µg/mL pyrimethanil in finished wine products to balance potential health risks with a

sustainable wine industry.

Standard methods for detecting pyrimethanil in bottled wines include laboratory-based GC, LC, and immunoassays. Misa (Metrohm Instant SERS Analyzer) integrates detection, data processing, and results sharing into a user-friendly system for high-throughput, onsite testing. In this application, trace detection of pyrimethanil in wine with Misa requires few laboratory supplies and minimal sample processing, yet returns rapid results.

INTRODUCTION

This application note describes a procedure for trace detection of pyrimethanil in white wine. A very simple sample extraction process results in very sensitive

SERS detection of pyrimethanil with Misa and gold nanoparticles (Au NPs).

REFERENCE SPECTRUM AND LIBRARY CREATION

To establish a reference spectrum, pure pyrimethanil standard at a concentration of 10 µg/mL in ethanol was analyzed with Au NPs. The unique SERS spectrum

shown in **Figure 1** can be used to create a library entry for pyrimethanil.

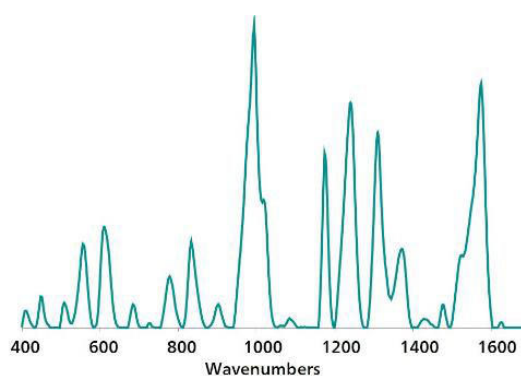


Figure 1. Unique standard reference Au NP SERS pyrimethanil spectrum.

EXPERIMENT

White wine was spiked with a stock solution of pyrimethanil in ethanol to provide a concentration range of test samples: 10, 5, and 1 µg/mL, 500 and 100 ng/mL. Chloroform (0.5 mL) was added to 1 mL of each sample concentration in a glass vial. This mixture was vigorously shaken and allowed to rest for at least 5 minutes to allow phase separation. Note that longer rest times improve results. Taking care to not disturb the lower chloroform layer, 200 µL of the top layer was transferred to a second vial and dried on a hot plate. The dried residue was resuspended in 450 µL of Au NP solution and 50 µL of 0.5 mol/L NaCl and shaken well to mix. This vial was inserted into the Misa vial attachment for measurement.



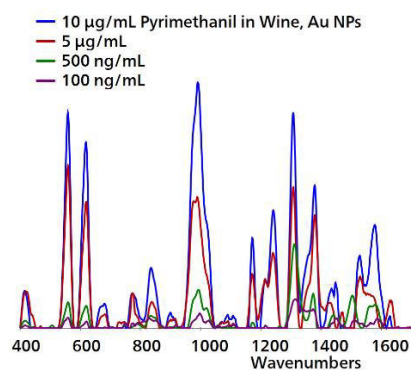
Table 1. Experimental parameters

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 Au NP SERS spectra acquired for the concentration range of test extracts demonstrates detection down to 100 ng/mL (Figure

2), a level significantly lower than the maximum permissible levels for pyrimethanil residue in wine.

**Figure 2.** Overlaid, baseline corrected, and background subtracted Au NP SERS spectra of pyrimethanil extracted from wine.

FIELD TEST PROTOCOL

Detection of pyrimethanil in the field

Fill a vial halfway with white wine. Using a pipette, add 10 drops of chloroform to this vial. Cap and shake very well to mix, and let rest for at least 5 minutes to allow layers to separate. Carefully remove a portion of the top layer with a clean pipette, and place 4 drops

of this solution into a *clean vial*. Evaporate the liquid on a hot plate. Fill this vial halfway full with Au NPs and add 1 drop of NaCl, then cap and shake. Insert into 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	
Chloroform	
NaCl solution	3 g NaCl in 100 mL water
Test settings	Use ID Kit OP on MISA

CONCLUSION

Misa provides a highly-sensitive, cost-effective solution for detecting pyrimethanil in wine. With Misa's portability, levels of pesticide residue can be

rapidly and reliably assessed in wineries during the production process, as well as in commercial storage, shipping, and receiving facilities.

CONTACT

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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 calibrage 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.