Trace Detection of Diphenylamine in Baby Food

Protecting consumer safety with Misa

Diphenylamine (DPA) is used as a dye fixative and antioxidant in industrial applications and as a produce preservative in agricultural operations. Despite its low toxicity to humans and the environment, there is controversy about its application on fruits and vegetables post-harvest to ensure long-term storage. Food safety advocates are concerned that daily ingestion of DPA, particularly in foods meant for babies, could have negative effects on children's health. To mitigate potentially toxic effects of DPA, both the US and EU stipulate a maximum residue limit (MRL) of 5 μg/g for whole pears and a stringent MRL of 10 ng/g for all processed baby foods. Misa (Metrohm Instant SERS Analyzer) provides a user-friendly and cost-effective alternative to traditional analytical methods used for detecting DPA in foods, such as GC-MS and GC-NPD. With Misa, trace level detection of DPA in infant food is achieved quickly and easily in a format that is easily adapted to diverse sample matrices.

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INTRODUCTION

This application note describes a simulated test procedure for detecting DPA in pureed pears marketed as food for infants. The assay is based on the acquisition of SERS-specific spectra for diphenylamine in chloroform extracts using Misa and gold nanoparticles (Au NPs).

REFERENCE MATERIAL AND LIBRARY CREATION

To establish a reference spectrum, a pure DPA standard (100 µg/g in ethanol) was analyzed using Au NPs. The unique SERS spectrum shown in **Figure 1** can be used to create a library entry for DPA.
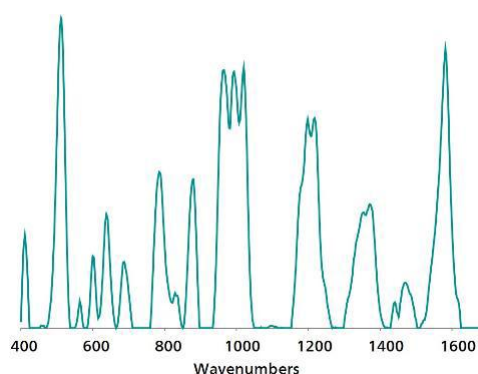

Figure 1. Standard gold nanoparticle SERS reference spectrum of diphenylamine.

EXPERIMENT

Serial dilutions of a stock solution of DPA in ethanol were spiked into purchased pureed pears sold as baby food to provide a range of test samples: 100, 50, 25, 10, 5, 2.5, and 1 µg/g. Samples were mixed with a vortex, then 0.5 mL chloroform was added, and samples were mixed again for 1 minute. After allowing phase separation for 10 minutes, 100 µL of the bottom chloroform layer was siphoned with a pipette and transferred to a glass vial for evaporative heating. The extract residue was suspended in 450 µL of Au NPs and 50 µL of 0.5 mol/L NaCl. Each vial was vigorously shaken and immediately placed into the vial attachment on Misa for measurement.
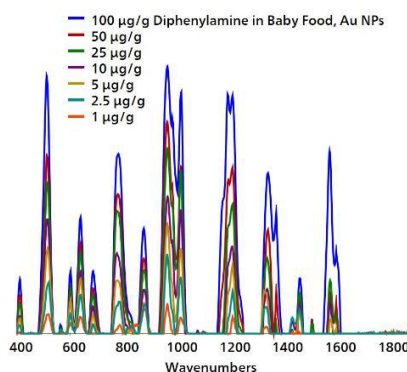
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Table 1. Experimental parameters

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

RESULTS

Overlaid of baseline-corrected spectra acquired for each tested concentration shows high-resolution DPA detection down to 1 µg/g (Figure 2).

Figure 2. Gold NP SERS spectral profile for diphenylamine in pureed pears, demonstrating detection down to 1 µg/g.

FIELD TEST PROTOCOL
Detection of diphenylamine in the field

Add baby food to glass vial until approximately 1/4 full. Using a pipette, add chloroform to the same vial until halfway full. Shake mixture vigorously for 1 minute, and then rest for 10 minutes to permit separation. Using a pipette, carefully remove half of the *bottom layer*, avoiding the top layer. Transfer this solution into a *clean glass vial* and heat until dry on a hotplate (5 minutes). Using clean pipettes, add Au NPs to vial until halfway full, then add 2 drops of NaCl solution. Cap vial and shake to mix, then allow to rest for 5 minutes before inserting into the vial attachment on Misa for testing.

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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

With successful, sensitive detection of diphenylamine in baby food, Misa is demonstrated to be a reliable QC tool for monitoring food safety, especially safeguarding the health of children. Misa is a portable and fully contained system, ideal for on-site testing and uniquely tailored for operation in a wide range of different environments.

Additionally, operators in postharvest produce treatment facilities who are at risk of high-level occupational exposure, for instance, would benefit greatly from a mobile monitoring system for such chemical hazards.

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CONFIGURATION

MISA Advanced

Metrohm Instant SERS Analyzer (MISA) 是一款高性能、便携式分析系,可快速/定非法物、食品添加和微量食品染物。MISA 的特点是配了 Metrohm 独特的道光栅描 (ORS) 技的高效光。其空需求最小和并且池寿命有所延,是或移室用的理想。MISA 提供各 1 激光附件,可活取。分析可通 BlueTooth 或 USB 接行。MISA Advanced 套件是一个完整套件,其作用是用能用 Metrohms 米粒溶液和 P-SERS 条行 SERS 分析。MISA Advanced 套件包含了一个 MISA 小管附件、一个 P-SERS-附件、一个 ASTM 校正准件、一个 USB 迷、一个 USB 供元和用于行 MISA 器的 MISA Cal 件。随供了一个用来安全保管器和附件的固保箱。

ID – Au NP

ID 套件 - Au NP 包含了 Mira/Misa 用使用体金溶液行 SERS 分析所需的件。套件包含了一个一次性抹刀、一个移液管、品小瓶和一个含金体的瓶子。

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