



Application Note AN-RS-016

Trace Detection of Potassium Ferrocyanide in Table Salt

Protecting consumer safety with Misa

Potassium ferrocyanide (KFC) is an anti-caking compound added to table salt. Although KFC is a common non-toxic food additive, its spectroscopic response is representative of analogous cyanide compounds. Trace detection of other cyanides in food products is essential to the safety of consumers, as

they can be toxic at oral consumption levels as low as 20 $\mu\text{g/g}$. This application demonstrates rapid trace analysis of potassium ferrocyanide in table salt with Misa (Metrohm Instant SERS Analyzer), in a simple assay format with minimal use of laboratory reagents.

INTRODUCTION

Federal guidelines state that KFC levels may not exceed 13 $\mu\text{g/g}$ as an additive to table salt. This application note describes detection of trace amounts

of potassium ferrocyanate in table salt with Misa and Metrohm Raman's silver nanoparticle (Ag NP) solution.

CYANIDE IN THE SERS SPECTRUM

A standard SERS reference spectrum (Figure 1) for KFC (100 $\mu\text{g/g}$, Ag NPs) is dominated by a prominent peak at 2133 cm^{-1} corresponding to the CN stretching mode. The CN cyanide stretch is distinctive, as it is one of very few Raman peaks which appear above 1800

cm^{-1} . In a y-corrected spectrum, this strong, distinct cyanide peak appears for any cyanide moiety, whether benign KFC or toxic hydrogen cyanide gas, making SERS an excellent technology for cyanide detection.

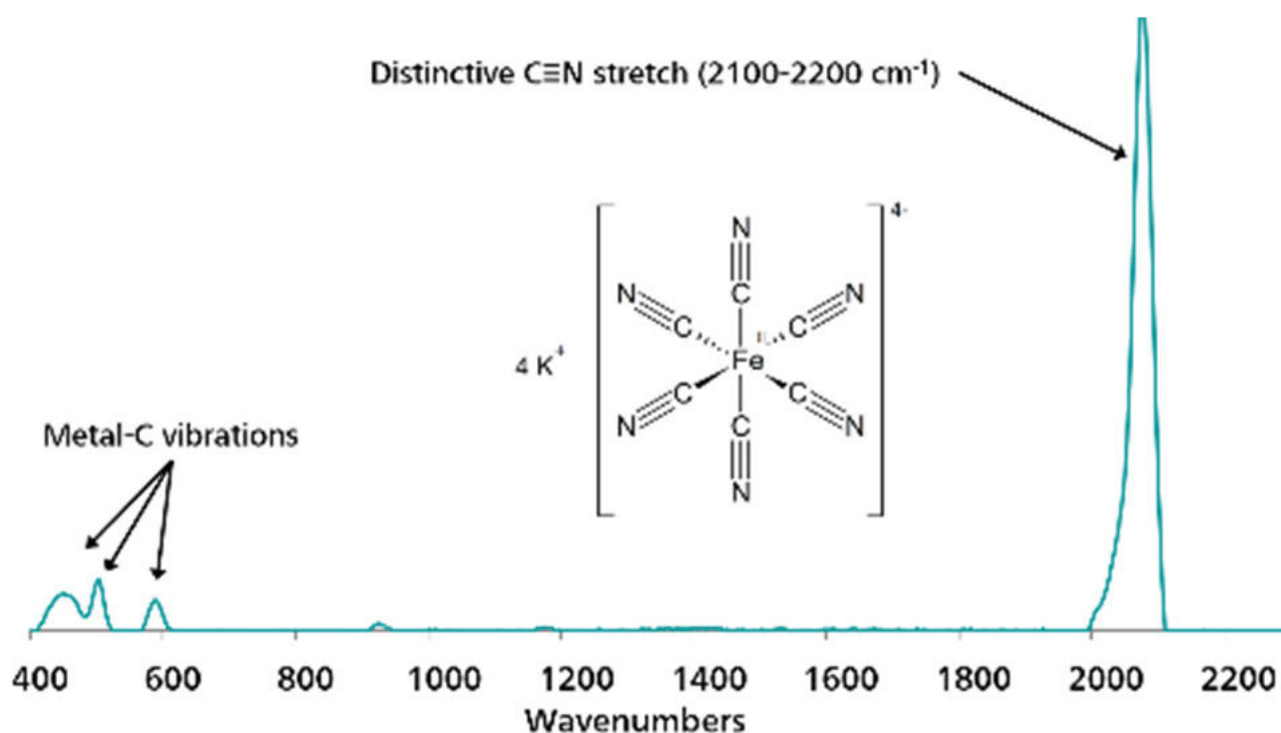


Figure 1. Representative SERS spectrum of cyanide.

EXPERIMENT

To prepare spiked samples, regular table salt was doped with potassium hexacyanoferrate(II) trihydrate dissolved in water to generate a concentration range of test samples: 10,000, 1,000, 100, 10, 1 $\mu\text{g/g}$, and 100 ng/g . Following the addition of analyte, samples were air-dried.

For SERS analysis, 100 mg of each sample was dissolved in 0.5 mL water. A 100 μL aliquot of the sample was mixed thoroughly with 800 μL Ag NPs and 50 μL 50 mmol/L NaCl in a glass vial. The sample vial was inserted into the vial attachment on Misa for testing.

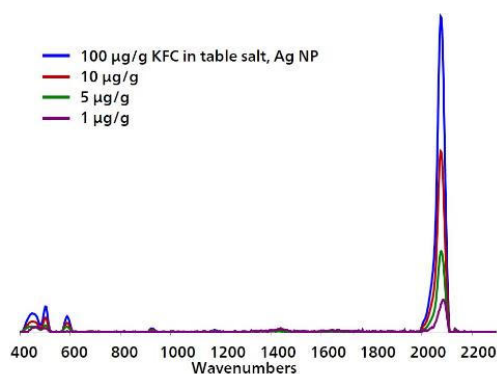


Table 1. Experimental Parameters

Instrument		Acquisition	
Firmware	0.9.33	Laser Power	5
Software	Misa Cal V1.0.15	Int. Time	1 s
Misa Vial Attachment	6.07505.040	Averages	10
ID Kit - Ag NP	6.07506.450	Raster	ON

RESULTS

Figure 2 displays overlaid Ag NP SERS spectra acquired for different concentrations of KFC, demonstrating detection down to 1 µg/g.

**Figure 2.** Overlaid, baselined, background-subtracted SERS spectra of KFC in table salt with Misa and Ag NPs.

FIELD TEST PROTOCOL

Detection of potassium ferrocyanide in the field

Using the large end of the scoop, add 3–4 scoops of sample to a 2 mL vial. Add water to the vial until ~1/4 full. Cap and shake the vial gently to dissolve solids, then let the sample rest for 2 minutes. Fill a clean vial

halfway full with Ag NPs. Using pipettes, add 2 drops of sample solution and 1 drop of NaCl solution to Ag NPs, then cap and shake the vial gently to mix. Insert into vial attachment on Misa for measurement.

Table 2. Requirements for Field Test Protocol

ID Kit - Ag NP	6.07506.450
includes:	Silver nanoparticles (Ag NP)
	Scoop
	Disposable pipettes
	2 mL glass vials
Reagents	
Water	
NaCl solution	3 g NaCl in 100 mL water
Test settings	Use ID Kit OP on Misa

CONCLUSION

Misa's rapid, facile trace detection of potassium ferrocyanide in table salt down to 1 µg/g is an order of magnitude below permissible levels. The simple procedure described herein requires minimal sample

preparation and is ideally suited for on-site testing. Given the similarity of SERS cyanide spectra, the results of this study suggest that Misa may be used as a robust cyanide sensor.

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CONFIGURATION



MISA Advanced

Metrohm Instant SERS Analyzer (MISA) ist ein hochleistungsfähiges, tragbares Analysensystem zur schnellen Detektion / Identifizierung von illegalen Substanzen, Lebensmittelzusatzstoffen und Lebensmittelverunreinigungen im Spurenbereich. MISA verfügt über einen hocheffizienten Spektrographen, der mit der einzigartigen Orbital-Raster-Scan (ORS)-Technologie von Metrohm ausgestattet ist. Er hat einen minimalen Platzbedarf und eine verlängerte Batterielebensdauer, perfekt für Tests vor Ort oder mobile Laboranwendungen. MISA bietet verschiedene Laser-Klasse-1-Aufsätze für flexible Probenahmeoptionen. Der Betrieb des Analysators ist über BlueTooth oder USB-Konnektivität möglich.

Das MISA Advanced-Paket ist ein Komplettpaket, das es dem Anwender ermöglicht, SERS-Analysen mit Metrohms Nanopartikellösungen und P-SERS-Streifen durchzuführen.

Das MISA Advanced-Paket enthält einen MISA Vial-Aufsatz, einen P-SERS-Aufsatz, einen ASTM-Kalibrierstandard, ein USB-Minikabel, ein USB-Netzteil und die MISA Cal-Software zum Betrieb des MISA-Geräts. Ein robuster Schutzkoffer zur sicheren Aufbewahrung des Instruments und des Zubehörs wird ebenfalls mitgeliefert.



ID Kit - Silbernanopartikel (Ag NP)

Das ID Kit - Ag NP enthält die Komponenten, die ein Mira/Misa-Benutzer benötigt, um eine SERS-Analyse mit kolloidaler Silberlösung durchzuführen. Der Kit enthält einen Einwegspatel, eine Tropfpipette, Probenfläschchen und eine Flasche mit Silber-Kolloid.