



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 µg/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.

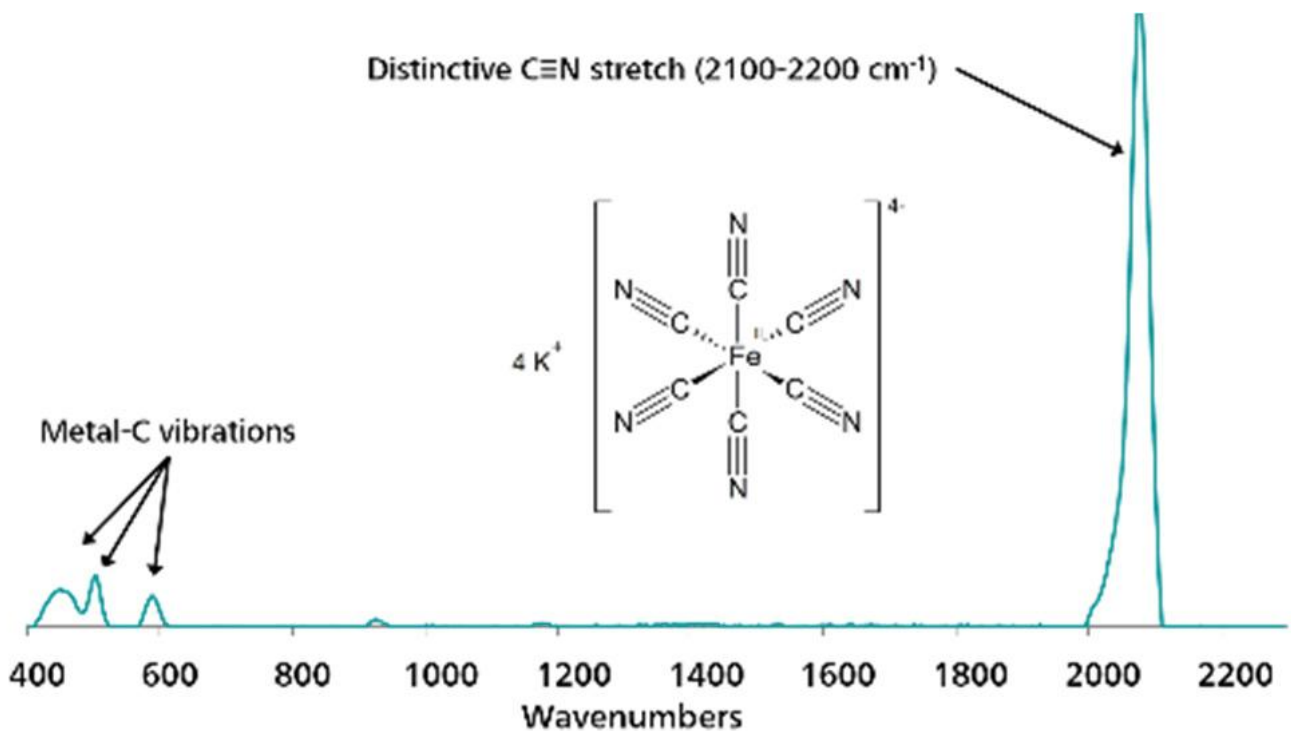


Figure 1. Representative SERS spectrum of cyanide.

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 µg/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.

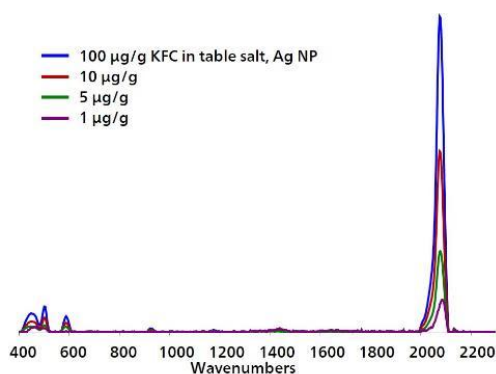


Figure 2. Overlaid, baselined, background-subtracted SERS spectra of KFC in table salt with Misa and Ag NPs.

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 $\mu\text{g/g}$ 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) 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 NP

The ID Kit - Ag NP contains the components a Mira / Misa user requires to perform a SERS analysis using silver colloidal solution. The kit contains a disposable spatula, dropper, sample vials, and a bottle of silver colloid.