Differentiation of inorganic salts using Raman spectroscopy

This Application Note shows the ability of handheld Raman systems like the Mira M-1 to identify and differentiate between salts such as carbonates, phosphates, and sulfates. The main focus of this work was to evaluate the influence of the cationic part and the water of crystallization on the Raman-spectroscopic identification of the mentioned salts.

INTRODUCTION

Inorganic salts often have the same anionic part, while the cationic part differs. Many salts can be distinguished by the number of water molecules bond to the salt.

In this study, the influences on the Raman spectrum of the cationic part of the salt and the number of water molecules bound to its anionic part were investigated. Although the differences between the salts are very small, the spectra that were recorded using a handheld Raman spectrometer differed sufficiently to differentiate between them.

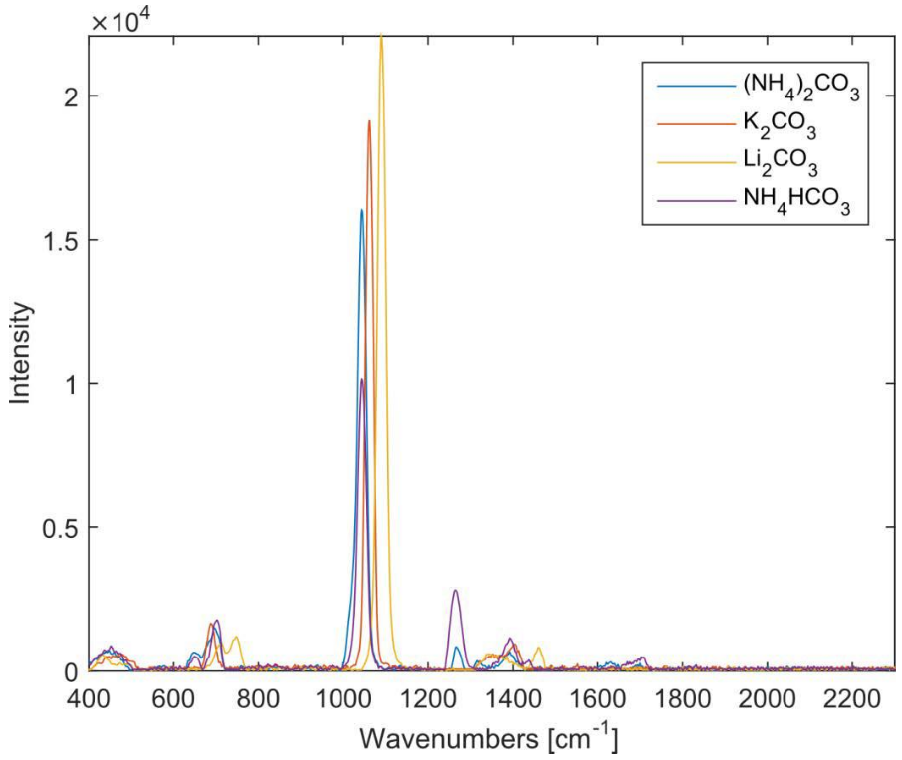

Figure 1. Overlaid Raman spectra of different carbonate salts.

EXPERIMENTAL

All spectra were measured using the Mira M-1 Raman spectrometer in auto-acquisition mode, i.e., integration times were determined automatically. A laser wavelength of 785 nm and the Orbital-Raster-Scan (ORS) technique were used. The samples were analyzed in small vials with the vial holder adapter. Three different anions that give are Raman-active were chosen: carbonate, phosphate, and sulfate. The cationic part of the salts was varied and the resulting spectral changes were investigated.

RESULTS AND DISCUSSION

Figure 2 shows how the carbonate peak in the Raman spectrum shifts depending on the counter cation. This shift is significant enough to use it to differentiate between the various carbonate salts (except for ammonium carbonate and ammonium bicarbonate, where the difference is very small).

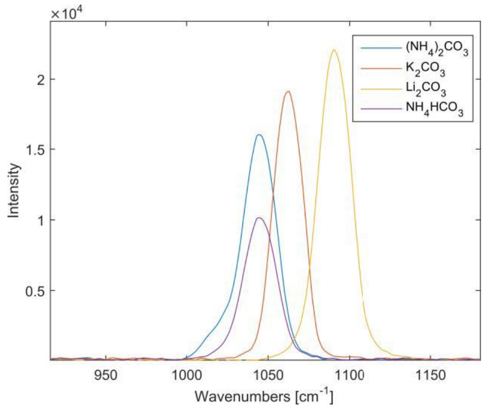

Figure 2. Main peaks of the various carbonates.

Similar results are also observed for various phosphates; the differences in the spectra are useful for their differentiation (see Figure 3) – despite the fact that there are some difficulties when trying to distinguish between diammonium phosphate and dipotassium phosphate (due to the similar ionic radius of potassium and ammonium).

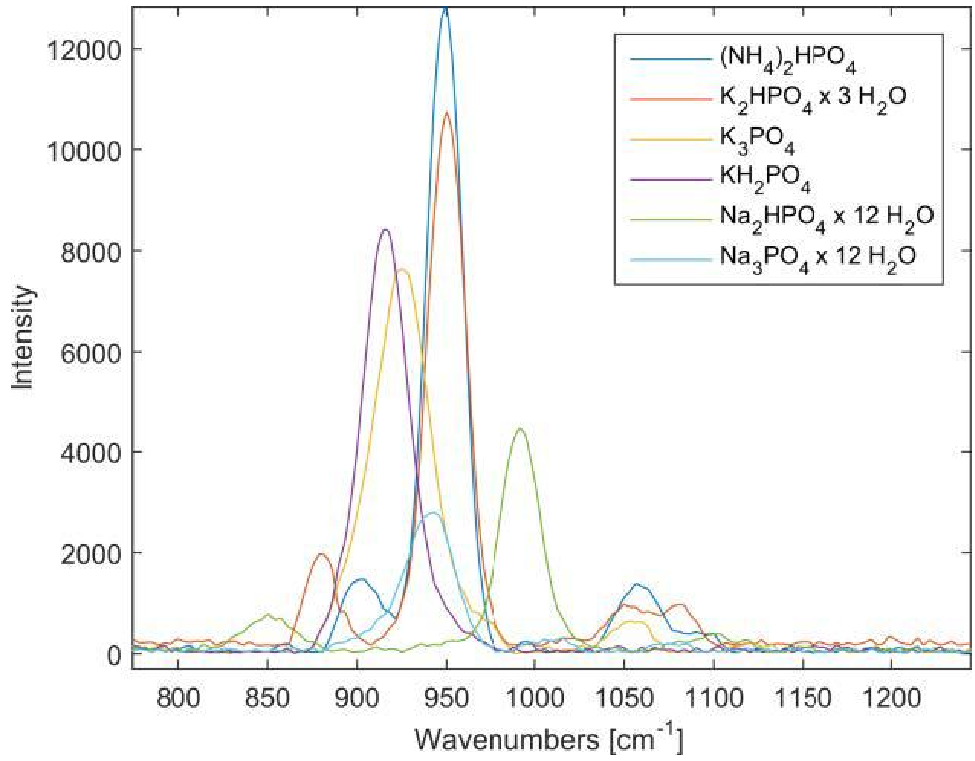

Figure 3. Main differences in the spectra of the phosphates.

The analyzed sulfates also differed from each other significantly, allowing their unambiguous identification with the handheld Mira M-1 (see Figure 4), although $CuSO_4$ x 7 H_2O, K_2SO_4, $SnSO_4$, and $ZnSO_4$ x 7 H_2O could not be identified unambiguously due to their similar spectra.

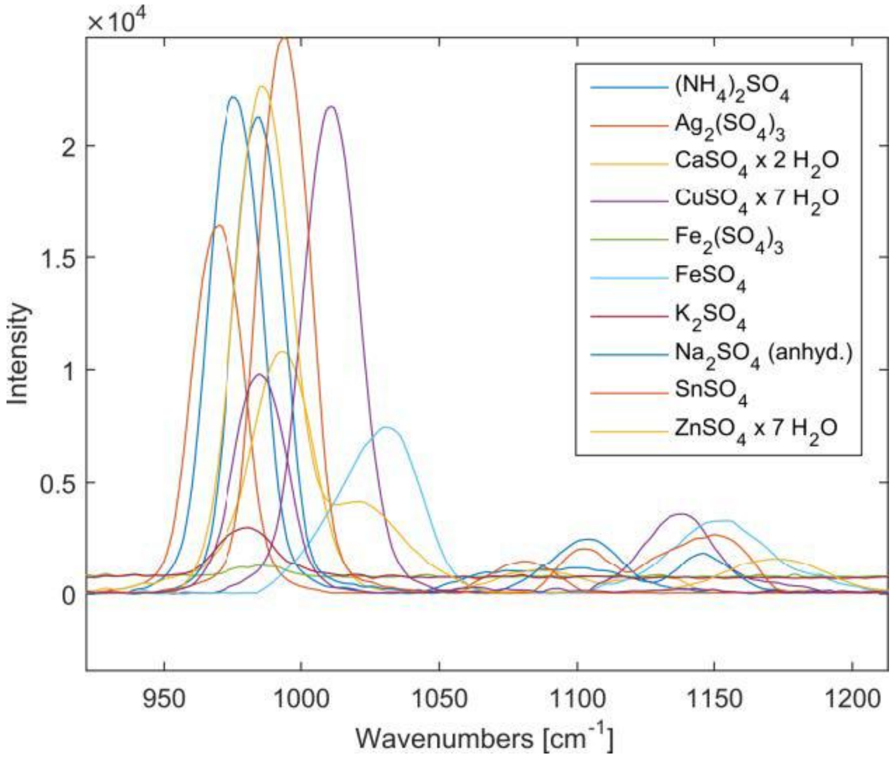

Figure 4. Main differences in the spectra of the sulfates.

CONCLUSIONS

Generally speaking, handheld Raman spectrometers, such as the Mira M-1, are very useful when it comes to inorganic material identification, even when dealing with salts having the same anionic part. Differences in the cationic parts of the salts help to unambiguously identify many salts with the Mira M-1 handheld Raman analyzer.

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CONFIGURATION

MIRA P Advanced

The Metrohm Instant Raman Analyzer (MIRA) P is a high-performance, handheld Raman spectrometer used for rapid, nondestructive determination and verification of different material types, such as Pharmaceutical APIs and excipients. Despite the small size of the instrument, the MIRA P has a ruggedized design and features a high-efficiency spectrograph design equipped with our unique Orbital-Raster-Scan (ORS) technology. The MIRA P is fully compliant with FDA 21 CFR Part 11 regulations.

The Advanced Package includes an attachment lens for analyzing materials directly or through containers (laser class 3b), as well as a vial holder attachment for analyzing samples contained in glass vials (laser class 1).