Application Note AN-RS-003

Identification of conventional organic solvents with handheld Raman spectrometers

This Application Note describes the fast and nondestructive handheld Raman spectroscopic identification and confirmation of commonly used organic solvents, which play an important role in many market segments. Measurements with the Mira M-1 handheld Raman spectrometer do not require sample preparation and allow instant results that identify the organic solvents unambiguously, putting traditional methods like HPLC, GC, and TLC into the shade.

INTRODUCTION

Solvents are liquids which have the ability to dissolve, suspend or extract other (solid) materials. They have a wide range of usage including the process, application, cleaning or separation of materials. Regarded as a matter of course, one often forgets the importance of solvents in daily life. In fact, without solvents, many of the products we use and rely on, from penicillin to industrial paint, would not perform to the standards we demand today.

Organic solvents play an important role in many beauty and cosmetic products, paints, fragrances, and synthesis reactions. Additionally one should not forget about the pharmaceutical industry, which can be considered as the most solvent-intensive field. Here solvents are used as reaction media, in separation and purification of synthesis products, to perform tablet coatings etc.

In this study, a fast alternative to the HPLC, GC, and TLC analysis of incoming solvents is shown. Further, the attractive features of handheld Raman spectroscopy, compared to other analytical techniques, are outlined.

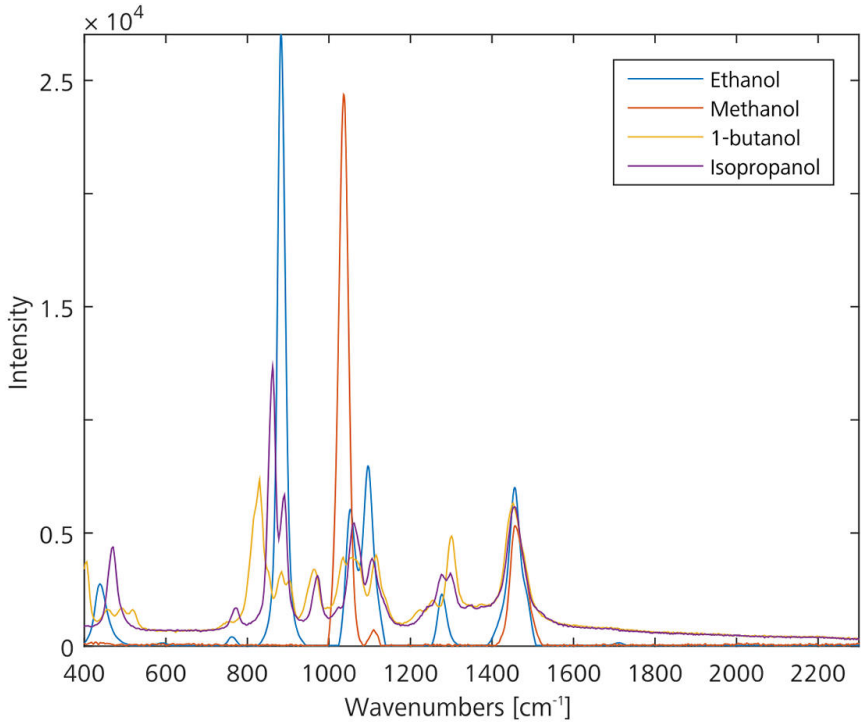

Figure 1. Overlaid Raman spectra of ethanol, methanol, 1-butanol, and isopropanol.

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. Spectra of solvents present in amber bottles were recorded with the point-and-shoot adapter, suitable for a long working distance (LWD). Solvents received in thick plastic containers were transferred to clear glass vials first before analyzing them with the vial holder attachment.

A collection of commonly used solvents such methanol, ethanol, isopropanol (IPA), tetrahydrofuran (THF), acetonitrile, dichloromethane (DCM), cyclohexane, xylene, dimethyl sulfoxide (DMSO) samples were used to build a specific library with the Mira Cal software.

RESULTS AND DISCUSSION

Raman offers high selectivity for solvents with double bonds, triple bonds or aromatic functional groups. Commonly used aliphatic hydrocarbon solvents such as hexane and heptane could be easily identified and confirmed based on the highly differentiating spectral correlation value. An overlay of commonly used chlorinated solvents is shown in Figure 2.

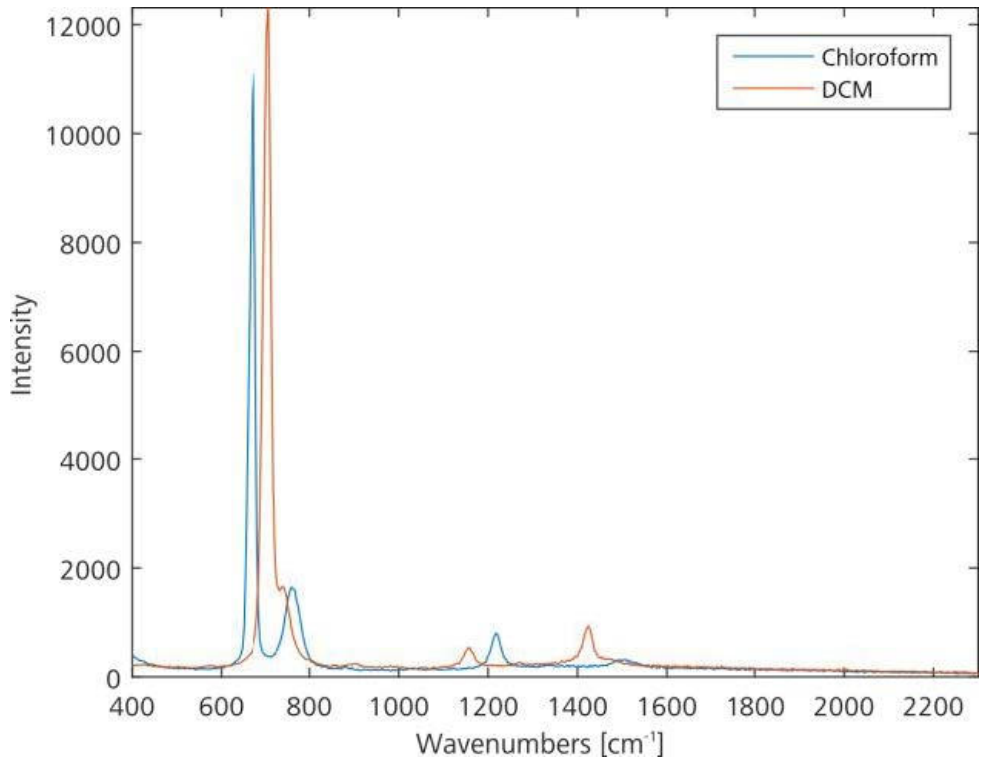

Figure 2. Overlay of chloroform and DCM, two widely used, chlorinated, solvents.

Figure 2 demonstrates that there are enough differences in the spectra of chloroform and DCM for a proper differentiation. When correlating the chloroform spectra to the DCM spectra, a spectral correlation of 0.081 is obtained, indicating the specifity and the unambiguous identification of these two solvents.

Figure 3 summarizes, how the Mira (with the ORS technique) enables unambiguous identification of all the common solvents used in the chemical and pharmaceutical industries. All nine solvents are correctly identified with spectral correlation values greater than 0.98, while the spectral correlation values for non-matching solvents were below 0.4.

Lib / Smpl	MeOH	EtOH	IPA	THF	ACN	DCM	CYH	XYL	DMSO
MeOH	1.00	0.01	0.07	0.07	0.01	0.00	0.13	0.09	0.08
EtOH	0.01	1.00	0.04	0.23	0.07	0.00	0.00	0.00	0.01
IPA	0.07	0.04	1.00	0.15	0.05	0.01	0.01	0.05	0.00
THF	0.07	0.23	0.15	1.00	0.44	0.00	0.01	0.00	0.00
ACN	0.01	0.07	0.05	0.44	1.00	0.00	0.00	0.02	0.00
DCM	0.00	0.00	0.01	0.00	0.00	1.00	0.00	0.16	0.03
CYH	0.13	0.00	0.01	0.01	0.00	0.00	1.00	0.08	0.00
XYL	0.09	0.00	0.05	0.00	0.02	0.16	0.08	1.00	0.05
DMSO	0.08	0.01	0.00	0.00	0.00	0.03	0.00	0.05	1.00

Figure 3. Correlation values showing the selectivity results of the Mira M-1

This shows that the spectral region used for the spectral identification of the solvents is perfectly suited for developing a specific solvents library and using it for identification.

Additionally, many other, structurally similar, solvents were tested, e.g., hexane and heptane, toluene and xylene. The Mira M-1 is capable of differentiating between structurally similar solvent molecules and shows a high selectivity. Figure 4 shows an overlay of hexane and heptane.

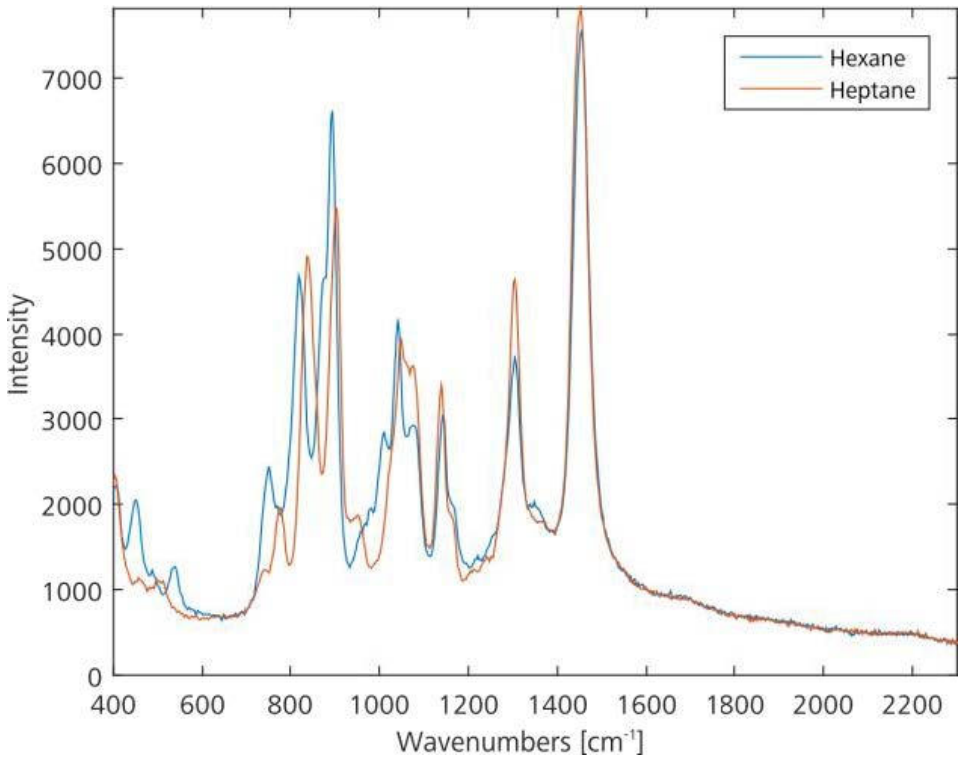

Figure 4. Ovarlay of hexane and heptane, showing the similarity in the spectra.

CONCLUSIONS

In this work, the Mira M-1 handheld Raman spectrometer is shown to be a reliable technique for the identification and confirmation of solvents used in a variety of industries. Measurements with the Mira usually take a few seconds and allow quick and confirmative information on the various solvents. In this way, Mira could be easily implemented at the receiving area for a fast identification of solvents, or even for solvent quality monitoring.

CONTACT

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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).