IRD 3 Application Note

Minor Components in Coriander Oil

Introduction

Coriander oil, used extensively in seasoning and perfumes, is obtained by the stream distillation of the partially dried ripe fruits of Coriander sativum L. The major areas of cultivation are the USSR, Poland and Hungary, as well as many additional countries with temperate climates. The oil is pale yellow with the odor of linalool, its major components (60-80%). Other components are mainly terpene hydrocarbons such as alpha pinene, gamma terpinene, camphor and para cymene. There are many other minor components. To illustrate the utility of combined GC/IRD/MSD in the area of essential oil analysis, two of these minor components of Russian coriander oil were examined and their identities confirmed at a high confidence level. Also several residual contaminants were found.

Product Overview

The IRD 3 is designed from the chromatographer's point-of-view and is the only analytical infrared instrument that seamlessly combines the separating power of the Gas Chromatography with the molecular identification of FTIR.

- Dedicated FTIR for use with GC
- Low maintenance and easy to use
- Small footprint
- Software interfaces with GC control software
- Seamless integration with MS

The IRD 3 is the perfect tool for the chromatographer looking to obtain more information about unknown samples. Using a heater light pipe flow cell, the sample is kept in a vapor state while interacting with IR. This allows the molecules to freely rotate in a low energy environment. Keeping the molecular geometry in tack during analysis provides unique and highly reproducible spectra.

Parameters and Results

The examination of Russian coriander oil, see Figure 1, by combined GC/IRD/MSD was performed using the series configuration where all of the column effluent is sent first through the flow cell of the IRD and then a portion of the flow cell effluent, ca. 15%, is sent to the MSD. This arrangement gives similar chromatographic sensitivities. A more thorough discussion of these operational considerations is documented elsewhere. The first ten minutes of the coriander oil chromatograms are shown in Figure 2 along with the peak assignments. The differences in signal size in the MSD's total ion chromatogram are apparent. By having both MS and IR data it is much easier to identify their components. The early components appear to be solvent residues from the oil preparation. Aromatic C-H absorption is observed in the IR

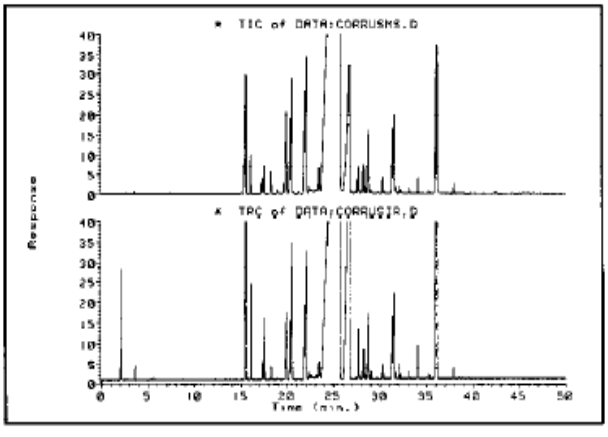

Figure 1. TIC and TRC of Russian Coriander Oil

spectra of the peak at 7.4 minutes while a strong m/z 91 ion is observed in the mass spectrum, strongly suggesting the presence of toluene. Oxygen, nitrogen, water and ethanol all have ions less than m/z 33 (the lowest mass limit used here) and hence are not seen on the Total Ion Chromatogram (TIC).

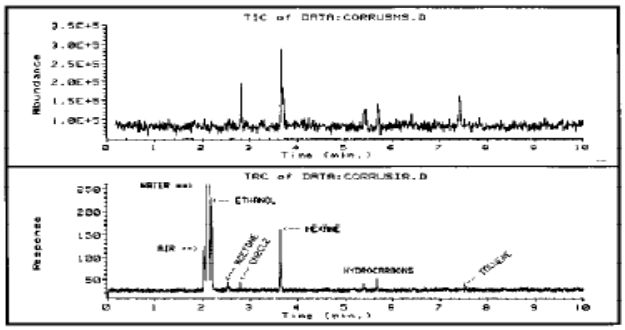

Figure 2. TIC and TRC of the first ten minutes

The major components of Russian coriander oil are noted on the chromatograms in Figure 3 and in Table 1. to illustrate the qualitative capabilities of the combined IRD/MSD, peak 3 at 17.3 minutes and peak 6 at 19 minutes were chosen. These peaks were identified by Chialva and Gabri. It can be seen in Figure 4 that the TIC/Total Response Chromatogram (TRC) response of peak 3 is fairly similar but the IR response is relatively weak for peak 6, however a good infrared spectrum was obtained.

The infrared and mass spectra of peak 3 are shown in Figure 4. The IR spectrum was searched against the 2000 entry Robertet Flavor and Fragrance Vapor Phase IR Library and the mass spectrum was searched against the 43000 entry NBS Library. Sabinene was the only common hit from both hit lists. Similarly, the spectra from peak 6 are shown in Figure 5. The combined library search indicated the compound to be alpha phellandrene. This is indeed exceptional considering that the IR spectrum represents ca. 3 nanograms and the mass spectrum ca ½ nanogram. The ASAP Analytical IRD 3 has the capability to view infrared spectra and up to four selected wavelength chromatograms in real time. Figure 6 is an example of a selected wavelength chromatogram (SWC) that shows the infrared absorption from 1748 to 1761 cm-1 indicating that those components are esters. The peak at 36.0 minutes is indeed an ester, geranylacetate. Further IRD/MSD work on coriander oil will utilize the power of selected wavelength chromatography to help identify the minor components.

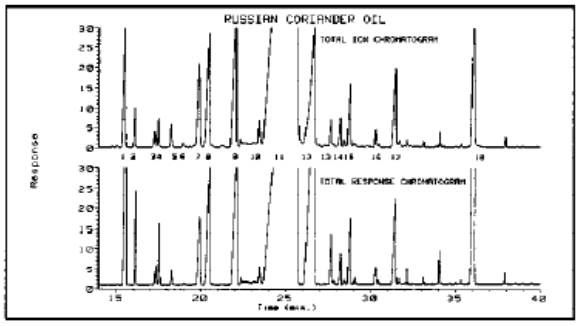

Figure 3. Major peaks of Russian Coriander Oil

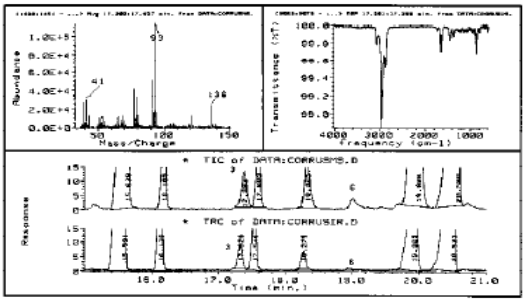

Figure 4. Expanded portion of chromatograms with mass and infrared spectra of peak at 17.3 minutes

Conclusion

The combined IRD/MSD system has been shown to be a powerful tool in the low level identification of trace components in coriander oil. The combined instrument provides a higher confidence results than either IR or MS technique alone.

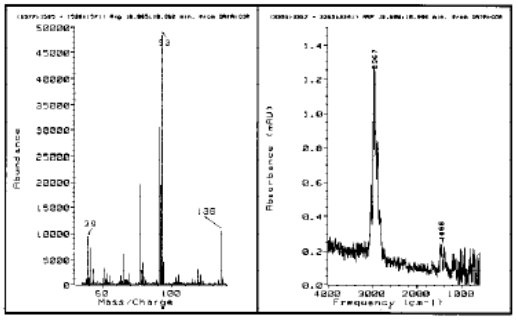

Figure 5. Mass spectrum and infrared spectrum of peak at 19.0 minutes

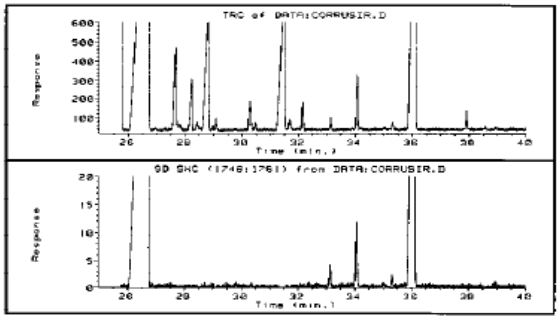

Figure 6. Later portion of TRC and second difference SWC for the Ester region