

Articles

Anal. Chem. 1994, 66, 425-430

Multidimensional Gas Chromatography Coupled with Infrared and Mass Spectrometry for Analysis of Eucalyptus Essential Oils

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A parallel cryogenic trapping multidimensional gas chromatography/Fourier transform infrared spectrometry/mass spectrometry combination (MDGC/FT-IR/MS) is evaluated for analysis of essential oils. As a demonstration of the value of this analytical approach, analysis of several essential oil samples are reported. The method developed to differentiate these oils is quite general and can be applied equally well to analysis of various other complex mixtures. By use of a multiport manual valve system in conjunction with multiple parallel cryogenic traps, it is demonstrated that three-stage or higher order gas chromatographic separations (GCⁿ) are practical, providing the expected improvements in separation efficiency needed for accurate qualitative analysis. Chromatographic and spectroscopic data obtained by analysis of two authentic and one known adulterated *Eucalyptus australasicus* oil samples are utilized as benchmarks for analysis of a fourth sample which is suspected of being adulterated and a fifth sample from an unknown source. It is concluded that the presence of either camphor or a combination of α -thujene, decane, sabinene, β -phellandrene, and γ -terpinene is indicative of eucalyptus oil adulteration.

Gas chromatography has been the analytical test of choice for separation and identification of essential oil components for many years. Traditionally, oil analysis has been performed mainly with flame ionization detection (FID) or with mass spectral detection.¹ However, there are several potential difficulties with both of these methods. When FID detectors are employed, retention times from separation on two different polarity columns are used to identify various components, requiring that retention times of authentic standards must be known for chromatography on both columns for each component of interest. Furthermore, because FID detection is nonspecific, it is difficult to deal with component overlap.

Mass spectrometry (MS) offers many advantages over FID detection with respect to specificity. Unfortunately, because most essential oils contain significant numbers of compounds which are isomers of one another, their positive identification using only mass spectral detection is often precluded. A well-known method of overcoming this difficulty is to incorporate infrared (IR) spectroscopy in the analytical system. Use of this complementary detector, in addition to mass spectrometry, greatly enhances the reliability of component identification.²⁻⁴ However, the accuracy of the identification step is still very much dependent upon the purity of the compounds resulting from a chromatographic separation. With mixtures consisting of many components as an essential oil, it is virtually certain that components will be incompletely separated using a single stage of separation.⁵ An appealing potential solution to this dilemma is the use of multidimensional gas chromatography (MDGC),^{6,7} as reported here. With MDGC, it should be possible to conveniently achieve much greater separation efficiency than is practical for a single-stage separation. When this methodology is coupled with infrared and mass spectral detection, the accuracy of qualitative analysis is limited by the quality and size of spectral databases available, rather than by an inability to obtain spectra of pure oil mixture components.^{8,9}

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