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PL10 ^{13}C -NMR as a tool for identification of individual components in natural mixtures

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The structural and quantitative analysis of complex mixtures such as essential oils or extracts, is commonly carried out by GC/KI, GC/MS, GC/IRFT, HPLC/MS or a combination of these techniques. In a different approach, ^{13}C -NMR spectroscopy could be used for the non separative and non destructive identification of the individual components of complex mixtures (essential oils, petroleum distillates, vegetable oils, biomass pyrolysis liquids). Following the pioneering work of Formáček and Kubeczka, we applied this methodology to identify the main components of essential oils and extracts. We developed an experimental procedure, based on computer-aided analysis of the ^{13}C -NMR spectrum of the mixture. We compared the chemical shift of each carbon in the experimental spectrum with the spectra of pure compounds listed in our spectral library. In order to obtain reproducible chemical shift values we began simultaneously: i) to define the best experimental and acquisition conditions to record NMR spectra (solvent, concentration, pulse width, ...) and ii) to create our ^{13}C -NMR spectral library. First, we checked the experimental procedure and the software with synthetic mixtures of terpenic derivatives. Then we analysed a large number of essential oils and solvent extracts. In all cases, this method allowed a good identification of sesquiterpenes as well as diterpenes present in essential oils (up to 24 compounds, lower limit: 0.5%) including structurally close molecules (such as stereoisomers) and compounds which exhibit insufficiently resolved mass spectral patterns or co-eluate on GC or thermolabile compounds.

In this presentation we will describe the interest of this methodology for the unambiguous identification of terpenes (including unusual sesquiterpenes and diterpenes) present in essential oils as well as diterpene acids and triterpenic derivatives present in oleoresins and in solvent extracts.

Further work is in progress to carry out: i) quantitative analysis of the components with or without internal reference, ii) complete and unambiguous analysis of some complex essential oils by GC-MS and Carbon-13 NMR spectroscopy, iii) enantiomeric differentiation of the major components using chiral shift reagents.



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He is Professor of Organic Chemistry and Spectroscopy at the University of Corsica. He has been researcher of the CNRS during 10 years (1971-1981) and received a grant of the NATO-NIH to stay at the Rice University (Houston, Texas) (1974-1975). Since 1992 he is *Professeur de 1ère classe* of the University of Corsica, where he has also occupied several positions, such as: Dean of the Faculty of Sciences and President of the Scientific Council, Responsible of the International Relationships, etc.

At present, he is Head of the Research Centre on "Biodiversity in Islands and in the Mediterranean". His main research topics are: methodology of NMR analysis, analysis of natural complex mixtures, aromatic plants, essential oils, extracts, bio-oils. He has more than 70 papers published in international journals and has participated in many international congresses with conferences and communications.

His research group has had active cooperation with other universities: Marseille, Montpellier, Sassari, Cagliari et Pise (Interreg), Barcelona (Picasso), Coimbra, Eindhovein, Abidjan, Conakry, Cotonou and Hanoi.