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ADVANCED PREPARATIVE APPROACHES FOR THE COLLECTION AND STRUCTURE ELUCIDATION OF VOLATILE COMPONENTS FROM COMPLEX SAMPLES

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The collection of analytes from natural sources is the goal of each preparative system. Conventional GC analysis for preparative purpose presents different limitations: although wide-bore columns (0.53 mm I.D.) are commonly used an excess of on-column sample amounts could result in skewed peaks and decreased resolution. On the other hand, the collection of pure components requires the injection of lower amounts in order to avoid coelutions on the wide-bore column. The higher is the injection volume, the lower is the total time required to collect a specific compound, thus the highest injection volume should be always used. Aiming to improve the productivity of the system a multidimensional prep-GC instrument is presented with the goal to reduce the total collection time and to improve the purity of the components collected. A prep-MDGC system was successfully used for the collection of pure components ranging from 10 to 30% concentration, collected at the milligram level, to allow a further characterization by means of other techniques (NMR, FTIR, MS). The system consists of an SLB-5ms - Supelcowax 10 - SLB-IL59 ionic liquid stationary phase (0.53 I.D.) combination used in the three GC dimensions, in order to provide three distinct selectivities. A preparative station, connected at the 3rd GC column outlet, allowed the re-condensation of pure components in a tube. The demands for the collection analytes at concentrations <10%, would consist in an increased sample injection volume, but this option could lead to the exceed the GC liner capacity. To improve the capability of the system, an on-line 4D chromatographic system (prep LC-GC-GC-GC) instrument can be adopted enabling the injection of higher sample volumes, the reduction of collection times, while maintaining high levels of purity. The system can be operated in different configurations, based on the complexity of the sample, exploiting a front-end LC pre-separation (whenever required by the complexity of the sample) before the three GC dimensions. Different applications are reported describing the potentiality of such an approach to provide a tool for the identification of possible valuable molecules for industrial and biological evaluations.