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Temperature and Flow Rate Controlled Serially Coupled Gas Chromatographic Capillary Column Systems with Tunable Polarity

Summary of Ph. D. Thesis (Eötvös Loránd University, Budapest, 2000)

Combination of the highly efficient chromatographic techniques with spectrometric methods enables rapid progress in the investigation of important multi-component complex mixtures. By means of these so called hyphenated techniques (GC-AES, GC-MS, GC-FTIR, etc.), reliable identification of a component has become much easier, so the solution of an analytical problem is often based on information provided by the spectrometer, and possibilities provided by the separation step are neglected. The information provided by spectrometry alone might, however, be insufficient to solve an analytical task – for example when too many components coelute with the component of interest, or when the component to be determined is hidden by another eluting in much greater amount. These problems can be solved if the selectivity of the chromatographic system is enhanced with regard to the components of interest.

In gas chromatographic systems with open tubular columns five basic stationary phases can be distinguished regarding polarity: poly(dimethylsiloxane), poly(phenylmethylsiloxane), poly-(trifluoropropylmethylsiloxane), poly(biscyanopropylsiloxane), and high molecular weight poly(ethylene glycol). Interim polarities (selectivities) can be set by the technique of polarity (selectivity) tuning.

With open tubular columns, the polarity tuning can be realized by three basic methods:

(i) Synthesis of a new polymeric phase by using, in a predetermined ratio, the appropriate monomers which contain the functional groups needed to obtain the desired polarity.
(ii) Mixing of two or more stationary phases in an appropriate ratio.
(iii) Coupling in series of two or more columns containing different stationary phases (creating a so called tandem system). To set the appropriate polarity, the length, phase ratio, temperature of the columns, and pressure drop across them can be altered.

For most chromatographic laboratories, possibility (iii) seems feasible, because in this case polarity tuning can be achieved by use of commercially available columns. A more sophisticated method of polarity tuning is when the two columns coupled in series are left unchanged and the desired polarity is set by independent modification of temperature and/or of pressure drops across the constituent columns ("on-line" polarity tuning).

The on-line-type tandem system can be used to solve the following tasks: (*a*) setting up an arbitrary column polarity within the limits determined by the constituent columns; (*b*) checking peak purity, (*c*) optimization of separation of a given mixture; (*d*) identification of components with reliability higher than is possible with single column systems.

In this thesis, techniques of polarity (selectivity) tuning in gas chromatography using two capillary columns coupled in series (so called "tandem" system) were described. The investigation covers theoretical and practical aspects of tandem systems tunable by modification of temperature and/or gas flow rate of the constituent columns. Possibilities of mathematical modeling, optimization of separation, reliable component identification, as well as experimental realization of tandem systems were studied in detail.