ANALYSIS OF ESSENTIAL OILS, 
USING CAPILLARY CHROMATOGRAPHY*

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The complete analysis of essential oils, which, as a rule, represent extremely complex mixtures of compounds of different chemical natures, is a difficult methodological problem. The difficulties arising in the analysis of these very complex compositions are associated primarily with the simultaneous presence in the mixture of compounds with a broad range of boiling points, many of which form groups of isomers, close in properties and therefore difficult to separate. In addition, the simultaneous presence of representatives of at least three to five different classes of substances substantially complicates the separate identification of many components. These difficulties are aggravated by the large range of concentrations of the substances to be determined, as well as by the fact that impurities contained even in small amounts frequently are decisive in the problem being studied. An example is lemon oil, the aroma and quality of which are determined primarily by oxygen-containing components, in amounts of no more than 4%. These difficulties cannot always be overcome even by the method of gas-liquid chromatography.

Until recently the composition of essential oils was usually determined by gas-chromatographic analysis of narrow fraction obtained in the separation of natural compositions by chemical methods or by fractional distillation. Thus, in an analysis of geranium oil [1], it was preliminarily separated into hydrocarbon and oxygen-containing fractions, while the identification was carried out by comparing the chromatograms before and after treatment of the oil fraction with reagents that selectively remove individual groups of compounds. A chromatographic study of coriander oil was conducted in the same way [2]; Bulgarian fennel oil was preliminarily subjected to fractional distillation, followed by chromatography of the fractions obtained [3]. A combination of the treatment of citrus oils with chemical reagents with the use of gas chromatography, UV, IR, and mass spectrometry is described in [4].

However, particular successes in the use of gas chromatography have not given a complete solution of the basic analytical problem: a rapid, reliable, and sufficiently rigorous quantitative evaluation of all the components of essential oils. Therefore the efforts of researchers to develop a method of complete gas chromatographic analysis of essential oils without their preliminary separation into fractions [5-6] is quite natural. The low resolving power of filled columns has made it possible to obtain only "characteristic curves" of essential oils, i.e., chromatograms on which a substantial fraction of the components proved to be unresolved. But even such results frequently permitted extremely important conclusions to be drawn on the basis of the ratio of the basic components, not only on the composition of the oils, but also on the area of cultivation, stages of vegetation and agrobiological conditions of cultivation of the oil-bearing plants, as well as the falsification of rare and costly essential oils [7-10].

The purpose of this work was to use capillary chromatography in the analysis of essential oils. We selected isothermal capillary chromatography as the original and basic method. An analysis of the data available in the literature on the relative retention of the most frequently encountered components of essential oils (terpene hydrocarbons [11-13], oxygen-containing isoprenoid compounds [14, 15], phenol derivatives [16], etc.) indicated that only in rare cases are the differences in the retainable volumes less than 1.02-1.04. This means that for a complete analysis of essential oils it is sufficient to have columns with an efficiency of about 30,000-50,000 theoretical plates. Therefore, we preliminarily conducted a verification and refinement of the existing procedures of preparation of capillary columns with sufficiently high and reproducible efficiency.

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Dioctyl sebacate (DOS) and dinonyl phthalate, as liquid phases of moderate polarity, and polyethylene glycol and polypropylene glycol (PPG), as more polar, were selected as the most promising liquid phases. The objects of investigation were various samples of essential oils isolated from oil-bearing plants in the Moldavian SSR.