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Gas chromatography retention of isoprenoid hydrocarbons

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Isoprenoid hydrocarbons in petroleums, organic rock matter,
meteorites and other products are usually identified by means of a
combination of capillary gas chromatography and mass spectrometry [1].

Basic information on the nature of a substance is obtained from
mass spectra by a comparison of the mass-spectral characteristics of pure
compounds with the same substances present in a mixture. The identi-
fication of the components according to chromatographic values of reten-
tion in complex systems is not sufficiently reliable if the analysis is
carried out on one immobile phase in conditions of temperature program-
ming. The reliability of identification by gas chromatographic charac-
teristics is considerably increased if the separation is carried out on
several immobile phases in isothermal conditions. In principle,

isothermal gas chromatography is capable of more effectively separating substances with relatively similar retention values, given the proper selection of optimum conditions. The reproducibility of the relative retention values, for example, all the Kovats indexes, is considerably better in isothermal conditions than in programmed temperature conditions.

Successful use of several immobile phases for identification is possible only if the retention indexes of the examined substance on these phases are essentially different. As a result of the studies made, we succeeded in separating a number of immobile phases on which the isoprenoids exhibit very different chromatographic characteristics. The Table gives the Kovats indexes of some isoprenoid hydrocarbons obtained on apiezon L, SE-30 and diethylene glycol adipate joined with pentaerythrite at 170°C. The retention indexes were determined from the chromatograms of the Romashkinskii petroleum fraction, based on the experimental data of A. A. Petrov and his co-workers [1]. Chromatographic separation was carried out on copper capillary columns of different lengths with an efficiency of 20000-90000 theoretical plates. The average relative error in the determination of the Kovats indexes is ± 1 unit. The thickness of the film of the immobile phase varied with the length of the column. The immobile phase was applied to the short capillaries from the more concentrated solutions (10-15%). From the data provided in the Table it is seen that isoprenoid hydrocarbons change their position quite strongly in relation to the n-paraffins, depending on the nature of the immobile phase. Even on two non-polar phases apiezon L and SE-30 the isoprenoids, appearing as non-polar isoparaffins, have essentially different retention values. It can be noted that pristane and phytane separate best from n-C₁₇ and n-C₁₈ on polyethylene glycol adipate joined with pentaerythrite. On

this same phase norpristane (2, 6, 10-trimethylpentadecane) is close to $n\text{-C}_{16}$; on SE-30 pristane has retention values close to $n\text{-C}_{17}$. The isoprenoid hydrocarbons can be identified quite reliably by comparing the retention indexes of the components of the mixture analyzed with the standard index values on three immobile phases. The quantitative content of the examined substances in a mixture can also be refined by juxtaposing areas of peaks from three chromatograms. In the case of small quantities of isoprenoids and in the presence of considerable overlapping of the peaks it is wise to conduct an analysis on mixed immobile phases, gradually varying their composition. The Kovats indexes of isoprenoid hydrocarbons, obtained on a mixture of apiezon L and diethylene glycol adipate joined with pentaerythrite (1:1) are given in the Table. As expected, the retention values on a binary immobile phase are intermediary according to the composition of this phase.

Table
Retention indexes of isoprenoid hydrocarbons at 170°C

Component	diethylene glycol adipate, joined with pentaerythrite	apiezon L	SE-30	DEGA joined with PE + apiezon L (1:1), 175°C
2, 6, 10 - trimethylundecane	1233,3	1259,8	1273,5	1245,5
2, 6, 10 - trimethylododecane	1348,0	1365,4	1377,0	1354,3
2, 6, 10 - trimethyltridecane	1426,1	1448,3	1462,0	1435,0
2, 6, 10 - trimethyltetradecane	-	1539,8	1553,8	-
2, 6, 10 - trimethylpentadecane	1605,5	1632,0	1648,0	1619,2
2, 6, 10, 14 - tetramethylpentadecane	1650,0	1686,6	1708,4	1671,8
2, 6, 10, 14 - tetramethylhexadecane	1762,5	1790,6	1812,0	1773,5

* Diethylene glycol adipate, joined with pentaerythrite.

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