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by L. Sojak et al.

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CAPILLARY GAS CHROMATOGRAPHY OF THE PRODUCTS OF CATALYTIC DEHYDROGENIZATION OF N-TRIDECANE AND N-TETRADECANE

L. Sojak, J. Hrivnak, P. Skalak

Catalytic dehydrogenation of n-alkanes produces all theoretically possible straight chain alkenes having the same number of carbon atoms in the molecule as the n-alkane, and aside from small amounts of other components, alkylbenzenes. Gas chromatographic analysis of these complex higher molecular mixtures, particularly group separation of hydrocarbons (alkanes, alkenes, semisaturated compounds), is performed using selective stationary phases. Using this method Döring et al. (1) analyzed dehydrogenation products of n-tetradecane in a 120 m capillary column coated with Carbowax 20M by programming the column temperature from 100 to 200°C. They obtained separation of grouped n-tetradecenes from n-tetradecane, and individual separation of alkylbenzenes. Lebedev et al. (2) were separating dehydrocracking products of n-tetradecane in a 7 m packed column using polyphenylether as the stationary phase; separation of n-tetradecenes from n-tetradecane was only partial.

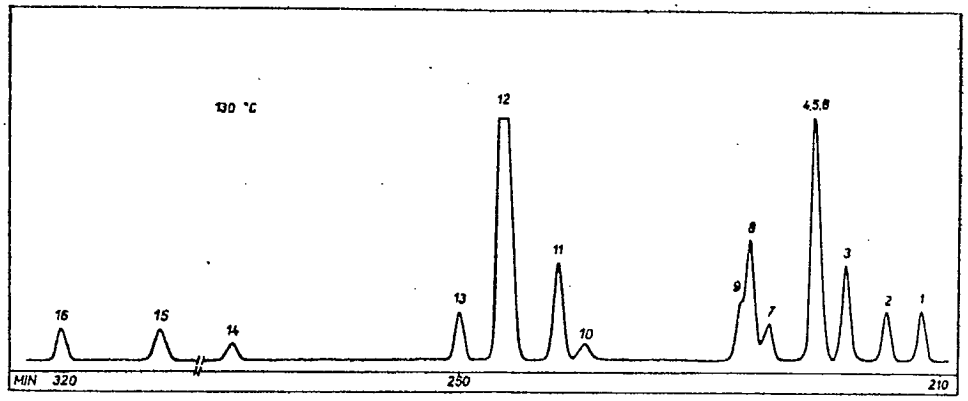
The location of the double bond of n-alkenes does not have to be important in their further technological processing. However in certain reactions, individual isomers of n-alkenes do not give various valuable end

products with equal ease (3). Hence the knowledge of individual composition of n-alkenes in reaction products is useful. In previous papers (4-6) we studied products of catalytic dehydrogenation of n-alkanes C_6-C_{12}. Using high efficiency capillary gas chromatography on squalane as the stationary phase, individual separation was obtained, and the main components of the dehydrogenation products were identified. The individual components were characterized through gas chromatography using retention indexes and their temperature increments. Unknown boiling points were calculated. The subject of this paper is the application of the above procedures to the products of catalytic dehydrogenation of n-tridecane and n-tetradecane.

EXPERIMENTAL SETUP

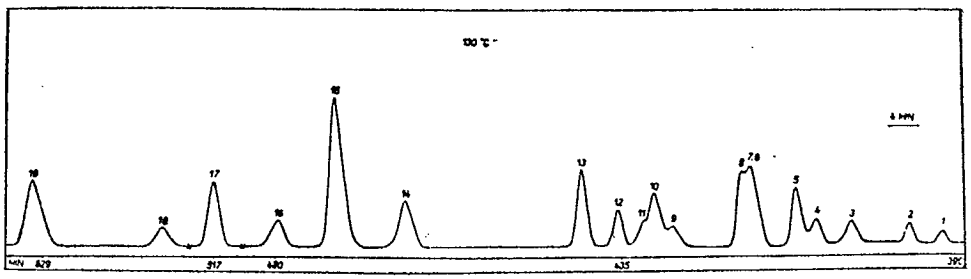
Carlo Erb GI 450 gas chromatography instrument with a flame ionization detector was used for analysis of the condensation products of dehydrogenation. A 200 m stainless steel capillary column of 0.2 mm inner diameter, impregnated with squalane as the stationary phase, was used. Working temperatures of the column were 86, 100, and 130°C. The inlet pressure of the hydrogen carrier gas was 3.0 kg/cm^2. Sample size was 0.2 to 0.5 μl diluted to 1 per cent concentration. Efficiency of the column, calculated for a chromatographic peak of trans-2-tridecene, was 350000 plates. Available standards were only n-alkanes

and 1-alkenes. For qualitative reasons, concentration of n-alkenes and alkylbenzenes in the samples of condensate was increased using a silica gel column.



Obr. 1. Chromatogram delenia hlavných zložiek produktu katalytickej dehydrogenizácie n-tridekánu, teplota squalánovej kolóny 130 °C. 1-cis-6-tridecén, 2- cis-5-tridecén, 3- trans-6-tridecén, 4- trans-5-tridecén, 5- trans-4-tridecén, 6- cis-4-tridecén, 7- 1-tridecén, 8- trans-3-tridecén, 9- cis-3-tridecén,10- 1-n-propyl-2-n-butylbenzén, 11- trans-2--tridecén, 12- n-tridekán, 13- cis-2-tridecén, 14- 1-etyl-2-n-pentylbenzén, 15- n-heptylbenzén, 16- 1-metyl-2-n-hexylbenzén

Figure 1. Chromatogram showing the separation of the main components of n-tridecane catalytic dehydrogenation product; column temperature 130°C. 1- cis-6-tridecene, 2- cis--5-tridecene, 3- trans-6-tridecene, 4- trans-5-tridecene, 5- trans-4-tridecene, 6- cis-4-tridecene, 7- 1-tridecene, 8- trans-3-tridecene, 9- cis-3-tridecene, 10- 1-n-propyl--2-n-butylbenzene, 11- trans-2-tridecene, 12- n-tridecane, 13- cis-2-tridecene, 14- 1-ethyl-2-n-pentylbenzene, 15- n-heptylbenzene, 16- 1-methyl-2-n-hexylbenzene



Obr. 2. Chromatogram delenia hlavných zložiek produktu katalytickej dehydrogenizácie n-tetradekánu, teplota squalánovej kolóny 130 °C. 1- cis-7-tetradecén, 2- cis-6-tetradecén, 3- cis-5-tetradecén, 4- trans-7-tetradecén, 5- trans-6-tetradecén, 6- cis-4-tetradecén, 7- trans-5-tetradecén, 8- trans-4-tetradecén, 9- 1-tetradecén, 10- trans-3- tetradecén, 11- cis-3-tetradecén, 12- 1,2-di-n-butylbenzén, 13- 1-n-propyl-2-n-pentylbenzén, 14- trans--2-tetradecén, 15- n-tetradekán, 16- cis-2-tetradecén, 17- 1-etyl-2-n-hexylbenzén, 18- n-oktylbenzén, 19- 1-metyl-2-n-heptylbenzén

Figure 2. Chromatogram showing the separation of the main components of n-tetradecane catalytic dehydrogenation product; column temperature 130°C. 1- cis-7-tetradecene, 2- cis-6-tetradecene, 3- cis-5-tetradecene, 4- trans-7--tetradecene, 5- trans-6-tetradecene, 6- cis-4-tetradecene,

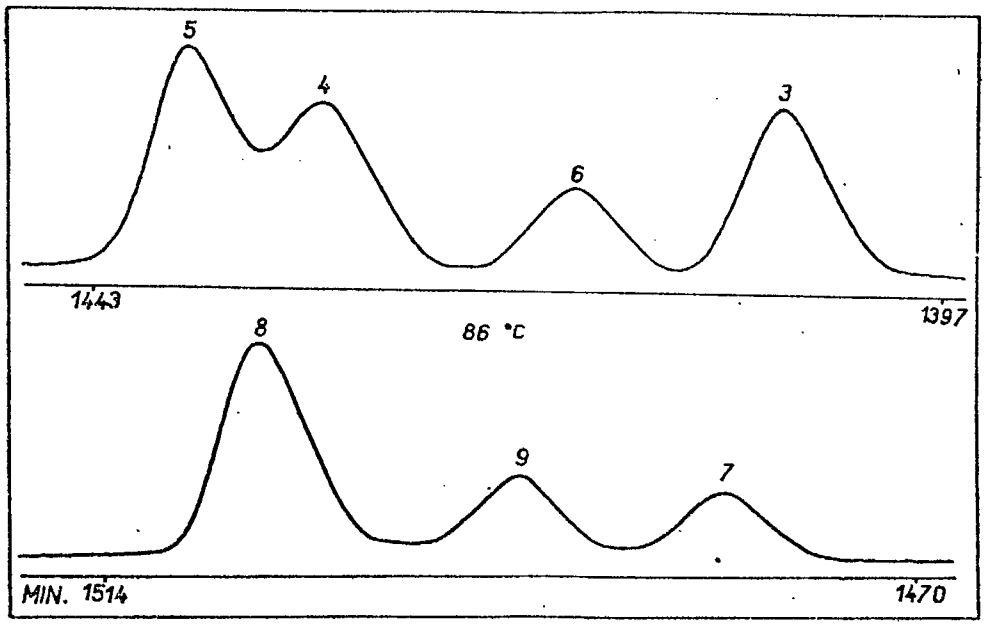
7- trans-5-tetradecene, 8- trans-4-tetradecene, 9- 1-tetradecene, 10- trans-3-tetradecene, 11- cis-3-tetradecene, 12- 1,2-di-n-butylbenzene, 13- 1-n-propyl-2-n-pentylbenzene, 14- trans-2-tetradecene, 15- n-tetradecane, 16- cis-2--tetradecene, 17- 1-ethyl-2-n-hexylbenzene, 18- n-octylbenzene, 19- 1-methyl-2-n-heptylbenzene

RESULTS AND DISCUSSION

As the carbon chain length increases, so do the difficulties in separation of a growing number of positional and configurational isomers (7). This problem of separation is further complicated by the presence of other components of dehydrogenation reaction product. High efficiency capillary gas chromatography is the most suitable way of individual analysis of the dehydrogenation products. High efficiency, which refers to the capillary column length, however contributes to longer analysis times.

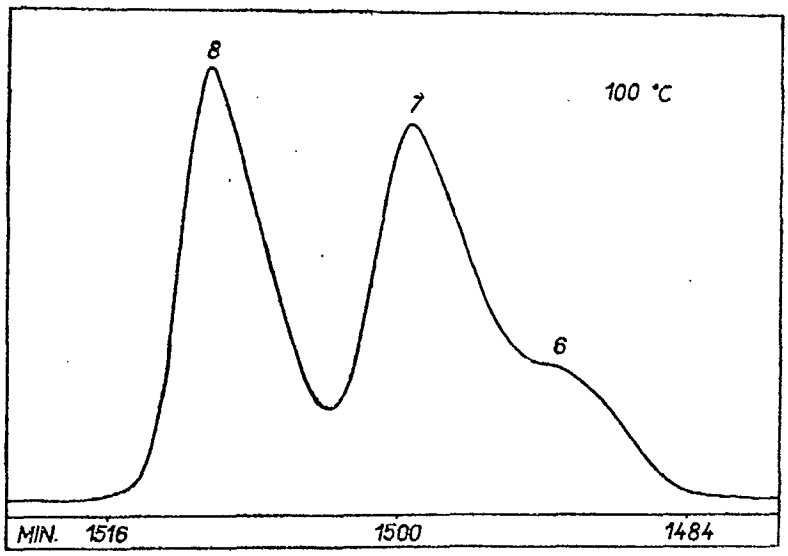
Chromatogram showing the separation of the main components of n-tridecane catalytic dehydrogenation product, at 130°C in the squalane impregnated column, is in figure 1. Chromatogram showing the separation of the main components of n-tetradecane dehydrogenation product, analyzed under the same conditions, is in figure 2. As can be seen from the figures, by separation of the dehydrogenation products at 130°C, separation of n-alkenes from n-alkanes and alkylbenzenes was obtained. Among individual n-alkenes, from 11 possible n-tridecenes only cis-4- + trans-4- + trans-5--tridecene did not separate, and from 13 possible n-tetradecenes cis-4- + trans-5-tetradecene did not separate.

In previous papers (4-7) we studied temperature dependence of retention data of n-alkenes and alkylbenzenes on squalane. We found that the temperature increment of retention indexes dI/dT of alkylbenzenes on squalane is 0.2 to 0.3 index units (i.u.) per 1°C, for n-alkenes dI/dT < 0.1 i.u./°C, and for corresponding cis-trans isomers the temperature increment is by 0.03 i.u. higher for the cis-isomer. Different temperature dependence of the retention indexes can be used in separation of the components of the product. Chromatogram showing the separation of the critical pairs of n-tridecenes is in figure 3. A comparison of figures 1 and 3 shows, that by change of the column temperature, separation of not separated or partially separated n-tridecenes was obtained. Separation of cis-4- and trans-5-tridecene at 100°C is in figure 4.



Obr. 3. Delenie kritických dvojíc n-tridecénov pri teplote 86 °C. 3- trans-6-tridecén, 4- trans-5-tridecén, 5- trans-4-tridecén, 6- cis-4-tridecén, 7- 1-tridecén, 8- trans-3-tridecén, 9- cis-3-tridecén

Figure 3. Separation the critical pairs of n-tridecenes at 86°C. 3- trans-6-tridecene, 4- trans-5-tridecene, 5- trans--4-tridecene, 6- cis-4-tridecene, 7- 1-tridecene, 8- trans--3-tridecene, 9- cis-3-tridecene



Obr. 4. Delenie cis-4-tetradecénu (6), trans-5-tetradecénu (7) a trans-4-tetradecénu (8) pri teplote 100 °C.

Figure 4. Separation of cis-4-tetradecene (6), trans-5-tetradecene (7), and trans-4-tetradecene at 100°C.

Individual n-alkenes were identified on the basis of a comparison of the measured and the published retention indexes I^{SQ} (7). Retention indexes of n-tridecenes and n-tetradecenes were measured on the same squalane column as the tabulated retention data (7), however with an approximately one year time lapse (the column was used). A comparison of the two sets of measurements shows a certain systematic deviation. The presently measured retention index values of n-alkenes are by 0.6 i.u. higher on the average than the previously published values. The measured and the published retention index values of n-tridecenes on squalane at 130°C I^{SQ}_{130} are presented in table 1. Other columns of the table contain a comparison of the I^{SQ}_{86} data (7) with data obtained recently abroad (8). Good agreement of

Table 1

Retention indexes and boiling points of components identified in n-tridecane catalytic dehydrogenation products

Tabuľka 1

Elučné indexy a body varu zložiek identifikovaných v produktoch katalytickej dehydrogenizácie *n*-tridekánu

1. Zložka	bod varu, °C 2.	b. v. (8) 3.	I^{SQ}_{86} (7)	I^{SQ}_{86} (8)	I^{SQ}_{130} (7)	I^{SQ}_{130}
a cis-6-tridecén	231,7	231,9	1270,2	1270,2	1273,4	1274,1
b cis-5-tridecén	231,9	232,1	1272,8	1273,7	1275,9	1276,6
c cis-4-tridecén	232,3	232,3	1277,7	1277,0	1280,7	1281,0
d trans-6-tridecén	232,4	232,3	1276,7	1277,3	1278,7	1279,1
e trans-5-tridecén	232,6	232,4	1279,0	1279,2	1280,7	1281,0
f trans-4-tridecén	232,8	232,7	1279,6	1280,1	1280,7	1281,0
g 1-tridecén	232,8	232,8	1282,8	1282,2	1283,8	1284,5
h cis-3-tridecén	233,2	233,4	1283,7	1283,8	1285,6	1286,3
i trans-3-tridecén	233,3	233,3	1284,9	1284,5	1285,1	1285,5
j trans-2-tridecén	234,8	234,8	1296,9	1296,1	1296,9	1297,5
k n-tridekán	235,4	–	1300,0	1300,0	1300,0	1300,0
l cis-2-tridecén	235,9	235,7	1301,0	1300,6	1302,7	1303,7
m 1-n-propyl-2-n-butylbenzén	239,9	–	–	–	1292,3	1296,6
o 1-etyl-2-n-pentylbenzén	242,7	–	–	–	1313,8	1319,3
p n-heptylbenzén	246,0	–	–	–	1340,2	1345,2
q 1-metyl-2-n-hexylbenzén	246,7	–	–	–	1344,8	1349,8

1. component, 2. boiling point, 3. b.p.
a- cis-6-tridecene, b- cis-5-tridecene, c- cis-4-tridecene, d- trans-6-tridecene, e- trans-5-tridecene, f- trans-4-tridecene, g- 1-tridecene, h- cis-3-tridecene, i- trans-3--tridecene, j- trans-2-tridecene, k- n-tridecane, l- cis-2--tridecene, m- 1-n-propyl-2-n-butylbenzene, n- 1-ethyl-2-n--pentylbenzene, o- n-heptylbenzene, p-1-methyl-2-n-hexylbenzene

Table 2

Retention indexes and boiling points of components identified in n-tetradecane catalytic dehydrogenation products.

Tabuľka 2

Elučné indexy a body varu zložiek identifikovaných v produktoch katalytickej dehydrogenizácie *n*-tetradekánu

1. Zložka	2. bod varu, °C	I^{SQ}_{130} (7)	I^{SQ}_{130}
a cis-7-tetradecén	249,4	1369,5	1370,1
b cis-6-tetradecén	249,6	1371,3	1371,8
c cis-5-tetradecén	250,0	1374,6	1374,9
d trans-7-tetradecén	250,2	1376,4	1376,8
e trans-6-tetradecén	250,4	1377,5	1377,8
f cis-4-tetradecén	250,5	1379,9	1380,3
g trans-5-tetradecén	250,8	1379,9	1380,3
h trans-4-tetradecén	250,9	1380,4	1380,8
i 1-tetradecén	251,1	1384,0	1384,3
j cis-3-tetradecén	251,3	1385,0	1386,2
k trans-3-tetradecén	251,4	1384,9	1385,4
l trans-2-tetradecén	253,0	1397,0	1397,6
m n-tetradekán	253,6	1400,0	1400,0
n cis-2-tetradecén	253,9	1402,8	1403,6
o 1,2-di-n-butylbenzén	257,5	1381,7	1386,6
p 1-n-propyl-2-n-pentylbenzén	257,7	1383,7	1388,6
q 1-etyl-2-n-hexylbenzén	260,9	1409,9	1414,7
r n-oktylbenzén	264,4	1439,9	1444,3
s 1-metyl-2-n-heptylbenzén	265,0	1443,0	1448,4

1. component, 2. boiling point
a- cis-7-tetradecene, b- cis-6-tetradecene, c- cis-5-tetradecene, d- trans-7-tetradecene, e- trans-6-tetradecene, f- cis--4-tetradecene, g- trans-5-tetradecene, h- trans-4-tetradecene, i- 1-tetradecene, j- cis-3-tetradecene, k- trans-3-tetradecene, l- trans-2-tetradecene, m- n-tetradecane, n- cis-2-tetradecene, o- 1,2-di-n-butylbenzene, p- 1-n-propyl-2-n-pentylbenzene, q-1-ethyl--2-n-hexylbenzene, r- n-oktylbenzene, s- 1-methyl-2-n-heptylbenzene

the data, on the average within 0.5 i.u., confirms the identification of n-tridecenes. The measured and the published (7) retention indexes of n-tetradecenes on squalane at 130°C are presented in table 2.

The boiling points of n-tridecenes and n-tetradecenes were calculated from their retention indexes within a ±1°C repeatability (7). The method is based on association constants of boiling points and retention indexes k_p ($k_p = dI/dT_b$), and the dependence of these constants on the number of carbon atoms, the isomer structure, and the column temperature (9). Tables 1 and 2 contain the calculated boiling points (only the boiling points of 1-alkenes were known from literature). The boiling points of n-tridecenes (tab. 1) are compared with values obtained recently by classical methods. The agreement, on the average within 0.1°C, is very good and validates the proposed gas chromatography method of boiling point calculation for n-alkenes.

The distribution of aromatics in dehydrogenation products of n-alkanes C_6-C_{12} (4-6) was used in identification of aromatic hydrocarbons in dehydrogenation products of n-alkanes C_{13}-C_{14}. The process of creation of 1-n-propyl--2-n-butylbenzene, 1-ethyl-2-n-pentylbenzene, 1-methyl-2-n--hexylbenzene and n-heptylbenzene in n-tridecane dehydrogenation, and 1,2-di-n-butylbenzene, 1-n-propyl -2-n-pentylbenzene, 1-ethyl-2-n-hexylbenzene, 1-methyl-2-n-heptylbenzene and n-octylbenzene in n-tetradecane dehydrogenation

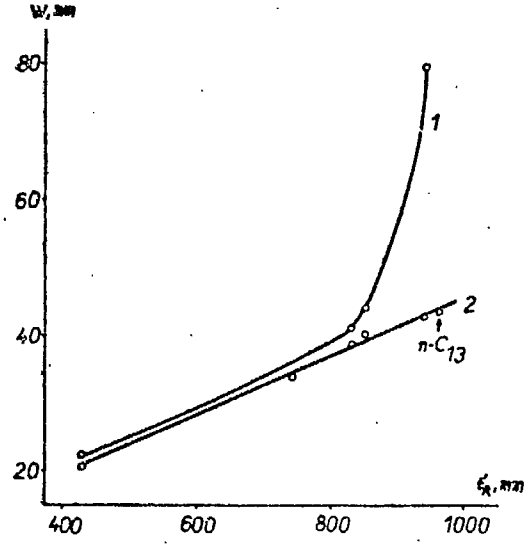
corresponds to the known process of creation of aromatic hydrocarbons. Identification of alkylbenzenes was confirmed on the basis of the dependence of methylene group contribution to the retention index $I^{SQ}_{CH_2}$ on the number of carbon atoms within a homologous series - n-alkyl-, 1-methyl--2-n-alkyl-, 1-ethyl-2-n-alkylbenzenes, 1-n-propyl-2-n-alkyl-, 1-n-butyl-2-n-alkylbenzenes (5).

A comparison of the measured retention indexes of alkylbenzenes C_{13}-C_{14} I^{SQ}_{130} with values measured previously (unpublished results) shows a considerably higher systematic deviation than for n-alkenes. The values are approximately by 5 i.u. higher. The data are presented in tables 1 and 2. The characteristic changes in the retention indexes of alkylbenzenes and n-alkenes on the column, in the course of its use, can be explained by changes in activity coefficients, as a consequence of changes in the squalane stationary phase structure (an increase in polarity of the phase). These results also point to problems in the use of published retention data as identification devices. They emphasize the need to standardize gas chromatography retention data.

Only the boiling points of n-heptylbenzene 246.0 and n-octylbenzene 264.4°C are known from among the identified alkylbenzenes. The boiling points of all alkylbenzenes among the products studied were calculated from the measured retention indexes of C_{13}-C_{14} alkylbenzenes, using retention indexes and boiling points of C_6-C_{12} alkylbenzenes,

on the basis of correlations resulting from free enthalpies (5). These boiling points are presented in tables 1 and 2.

We referred to a change in selectivity of the squalane column during its long term use at raised temperatures, and the related problems in qualitative characterization of alkylbenzenes and n-alkenes. In unconcentrated samples of the dehydrogenation products (with high n-alkane contents) we found another qualitative effect. A linear relationship holds between the peak width w and reduced retention time t_R $w = a + b\ t_R$. Appropriate measurements taken during the separation of n-tridecane dehydrogenation products



Obr. 5. Závislosť šírky elučných vín od elučného času pre zložky eluujúce pred n-tridekánom. Rozdeľovanie zmesi zložiek, 1- s vysokým obsahom n-tridekánu, 2- rovnakých koncentrácií

Figure 5. The dependence of peak widths on retention times for components eluting before n-tridecane.
1- high n-tridecane concentration, 2- equal concentration

showed considerable widening of trans-2-tridecene peak eluting closely preceeding n-tridecane (the concentration ratio of these components was approximately 1:50). Figure 5 shows the dependence of peak widths on retention times for components eluting before n-tridecane. Inspite of an approximately equal concentration of these components, a nonlinear change in width of the last trans-2-tridecene peak can be seen (almost double). It seemed that more components elute under this peak. On the other side, the width of the cis-2-tridecene peak eluting closely following the n-tridecane peak became narrower. Similar events were described by Berezkin et al. (10) in a qualitative study of microadditives in packed columns, and their mechanism was explained by Deans (11). Capillary columns in comparison with packed columns have relatively low sample capacity, and this is probably the reason for the appearance of these effects already under the given concentration ratios.

Catalytic dehydrogenation of n-tridecane produces mainly n-alkenes with a double inner bond. The ratio of positional isomer quantities 1-:2- is approximately 1:4. The ratio of quantities of individual inner positional isomers is approximately the same (increases slightly as the double bond is shifted towards the end of the molecule). For the n-tetradecenes the ratio of quantities of positional isomers 7-:6- is 1:2. The ratio for the geometric cis-:trans- isomers positionally corresponding to the double bond is approximately 1:2.

CONCLUSION

Using high efficiency capillary gas chromatography on squalane in an analysis in 86 to 130°C temperature range, individual separation of the main components of n-tridecane and n-tetradecane catalytic dehydrogenation products - all straight chain alkenes, the characteristic alkylbenzenes, and the n-alkanes - was obtained. Identification of the product components was made using gas chromatography methods. Individual components were characterized by retention indexes and their calculated boiling points. Accompanying qualitative effects were briefly discussed - a change in squalane column selectivity and a nonlinearity between peak width and retention time.

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