

## IDENTIFICATION OF LOW-BOILING FRACTION OF PYROLYSIS OIL

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Composition of the low-boiling fraction of the pyrolysis oil obtained from continuous rectification has been determined by combination of capillary gas-liquid chromatography with other identification methods (catalytic hydrogenation, polymerization). In this way components have been identified which form overall 86.0 per cent (*m/m*) of the low-boiling fraction. The said pyrolysis oil fraction has been found to contain almost 50 per cent (*m/m*) of unsaturated components able of polymerization, especially methylindenes, methyl-, ethyl- and dimethylvinylbenzenes, divinylbenzenes and 1,2-dihydronaphthalene. Elution behaviour of all the identified isomeric methylindenes, divinylbenzenes and 1,2-dihydronaphthalene has been evaluated by determination of parameters of the equation  $I_{st, phase(2)} = k \cdot I_{st, phase(1)} + q$ . The Kováts elution indices of all the identified aromatic hydrocarbons have been determined with the use of a glass capillary column wetted with Carbowax 20M at 80°C.

The pyrolysis oil represents a varied mixture containing predominantly aromatic hydrocarbons. Identification of some fractions obtained from rectification of pyrolysis oils is dealt with in many papers which are summarized in ref.<sup>1</sup>. However, few communications have only been found dealing with identification of low-boiling fraction of pyrolysis oil.

Kuraš and Hála<sup>2</sup> carried out a type analysis of alkylbenzenes by means of mass spectrometry using the Fitzgerald method. In this way they also identified higher-boiling aromatic hydrocarbons in pyrolysis oil. Kugacheva<sup>3</sup> identified lower-boiling hydrocarbons of pyrolysis oil condensate by GLC using 1,2,3-tris-(2-cyanoethoxy)propane as stationary phase in a packed column. She identified especially *n*-alkenes, *n*-alkanes, dienes and 31 aromatic hydrocarbons boiling within the limits of 80 to 223°C at atmospheric pressure. Gallegos and coworkers<sup>4</sup> used capillary GLC with squalane column and mass spectrometry and identified 133 components (especially alkanes, alkenes, dienes and naphthenes) inclusive of aromatic hydrocarbons boiling up to 182°C at atmospheric pressure. Amal and Bahram<sup>5</sup> used similar experimental conditions to determine individual compound types (alkanes, alkenes, aromatic hydrocarbons) present in distillate from pyrolysis gasoline with distillation range 32 to 200°C. Bruk and coworkers<sup>6</sup> suggested a method for analysis of pyrolysis products by GLC using two packed columns out of which one is packed with modified alumina and the other with a carrier wetted with Benton 245; in this way some aromatic compounds were identified (alkylbenzenes and indene). Bajus and coworkers dealt with composition of pyrolysis products of *n*-heptane<sup>7</sup> and methylcyclohexane<sup>8</sup> (pyrolysis in the presence of steam) by combination of capillary GLC and MS, the main attention being focused on the components boiling below b.p. of styrene. The same authors dealt with similar problems in another communication<sup>9</sup>. Herrera and coworkers<sup>10</sup> identified the components present in a pyrolysis gasoline with the use of a capillary column wetted with

SE-30 and a temperature program from  $-25$  to  $200^{\circ}\text{C}$ , which made it possible to identify  $\text{C}_3$ — $\text{C}_5$  alkenes and dienes.

The low-boiling fraction of pyrolysis oil could represent, with respect to the presence of a number of unsaturated components (especially indene, but also its methyl- and dimethylhomologues<sup>11,12</sup>), a suitable raw material for preparation of resins or a suitable source of some aromatic hydrocarbons. Hence, the aim of the present communication is identification of all main components present in low-boiling fraction of pyrolysis oil (Závody ČSSP Litvínov) by application of chemical methods combined with capillary GLC.

## EXPERIMENTAL

### *Capillary Gas-Liquid Chromatography*

Liquid fraction from gasoline pyrolysis at  $820$ — $825^{\circ}\text{C}$  was submitted to continuous rectification at atmospheric pressure on a column with 30 t.p. efficiency to give a fraction boiling within the limits  $160$  to  $210^{\circ}\text{C}$  (yield  $6.5\%$  m/m with respect to the starting pyrolysis oil). Samples of this low-boiling fraction were analyzed on the columns 1 to 3 (Table I) installed in a Fractovap 4160 apparatus (Carlo Erba, Milan) equipped with an inlet splitter (the ratio was adjusted at the value  $1:100$ ), a flame ionization detector, and a temperature programmer. Conditions of the analysis:  $0.1\ \mu\text{l}$  injection, argon flow rate  $1.0\ \text{ml}\ \text{min}^{-1}$ , the temperature conditions are given in Table I. The increments of elution indices of methyl and vinyl groups were determined from elution data of styrene and isomeric methylvinylbenzenes determined with the column 1 at  $75^{\circ}\text{C}$ . The columns 1 and 2 were wetted by the mercury plug method<sup>13</sup> after modification of inner surface of the capillary (Table I); the column 3 was wetted in the same way after modification and silanization according to ref.<sup>14</sup>. The same conditions were also used for analysis of the samples of low-boiling fraction modified by polymerization or hydrogenation.

### Vacuum Rectification

The low-boiling fraction of pyrolysis oil ( $1\ 443\ \text{g}$ ) was rectified on a column of  $0.9\ \text{m}$  length packed with constantan spirals at  $4\ \text{kPa}$  pressure. Reflux ratio  $1:30$ . The fractions were taken within the limits of  $0.5$  to  $5^{\circ}$ ; some of them were submitted to catalytic (Pd/C) hydrogenation at atmospheric pressure of hydrogen for  $48\ \text{h}$ .

### Catalytic Hydrogenation at Higher Pressure of Hydrogen

A sample of  $20\ \text{g}$  of the low-boiling fraction of pyrolysis oil was diluted with  $450\ \text{ml}$  ethanol and submitted to catalytic (Pd/C) hydrogenation in an autoclave equipped with a vibration stirrer (Lampart, Hungary) at  $35$  to  $40^{\circ}\text{C}$  at  $980\ \text{kPa}$  hydrogen pressure. The catalyst was removed by filtration, ethanol was distilled off, and the sample was analyzed by capillary GLC.

### Polymerization

The unsaturated components present in the sample of low-boiling fraction of pyrolysis oil were polymerized by action of a  $7:3$  (m/m) mixture of  $93\%$   $\text{H}_2\text{SO}_4$  and  $85\%$   $\text{H}_3\text{PO}_4$  (ref.<sup>15</sup>). The non-

-polymerized portions were submitted to steam distillation, the distillate was extracted with ether, the extract was dried, ether was evaporated, and the residue was analyzed by capillary GLC.

#### Preparation of Chromatographic Standards

The analytical standards prepared as chemical individuals were identified by IR spectrometry (Unicam SP 200G, GB) and capillary GLC, the mixed standards were only analyzed by capillary GLC. The mixture containing 65% (*m/m*) 5-methylindane and 35% (*m/m*) 4-methylindane was prepared by chloromethylation of 1 mol indane (Urxovy závody, Valašské Meziříčí) with a mixture of formaldehyde and hydrochloric acid<sup>16</sup> and subsequent hydrogenolysis of the chloromethylindane mixture in acetic acid on a Pd/C catalyst at 980.6 kPa hydrogen pressure. After neutralization of acetic acid the product was extracted with benzene, benzene and indane were distilled off from the extract through a Vigreux column, the distillation residue was distilled without column and analyzed by capillary GLC. Yield of methylindanes: 35% related to indane. 1-Methylindane was prepared by hydrogenation of 1-methylindene (Serva Heidelberg, Germany) in ethanol on Pd/C catalyst at 196 kPa hydrogen pressure for 8 h. 3-Methylindene was prepared by reaction of 0.1 mol 1-indanone (Research Institute of Organic Syntheses, Pardubice - Rybitví) with 0.12 mol methylmagnesium iodide in anhydrous ether under nitrogen atmosphere. After decomposition of the organomagnesium salt, 1-methyl-1-indanol was separated in an organic layer with ether, ether was distilled off, and the residue was submitted to dehydration by distillation with a mixture of anhydrous sodium sulphate and sodium hydrogen sulphate at atmospheric pressure. Yield of methylindene fraction (b.p. 204–206°C) 27% with respect to 1-indanone. 1,4-Dihydro-

TABLE I

The capillary columns<sup>a</sup> used for analysis of the low-boiling fraction of pyrolysis oil

Column No	Length m	I.d. mm	Stationary phase phase	<i>b</i> <sup>b</sup>	<i>t</i> , °C
1	42	0.26	Carbowax 20 M (Carlo Erba)	86 000	70; after 20 min it was increased at a rate 0.5°C min <sup>-1</sup> up to 90, then at a rate 5°C min <sup>-1</sup> up to 125
2	45	0.26	Reoplex 400 (Applied Sci.)	112 000	the same program as with column No 1
3	34	0.25	OV-101 (Applied Sci.)	94 000	70; after 5 min it was increased at a rate 1°C min <sup>-1</sup> up to 110, then 2 min at 110

<sup>a</sup> Inner surface of the capillary before its wetting (in the case of column No 3 even before its deactivation by silanization) was modified by etching with methyl 1,1,2-trifluoroethyl ether in gas phase; <sup>b</sup> number of theoretical plates was determined for *k* = 3.4 in all the cases.

naphthalene was obtained by reduction of 0.2 mol naphthalene with 2.6 mol sodium in boiling ethanol according to ref.<sup>17</sup>; a part of the product was rearranged to 1,2-dihydronaphthalene (after removing ethanol by distillation) by boiling the distillation residue with 1-pentanol for 8 h (ref.<sup>17</sup>). Pentanol was removed by distillation from oil bath, the distillation residue was decomposed with water and extracted with benzene, and the extract was rid of benzene by distillation from oil bath. Ethyl-2,6-dimethylbenzene was prepared in the same way as 1-methylindane by hydrogenation of 2,6-dimethylvinylbenzene (ICN Pharmaceuticals, USA). Ethyl-2,5-dimethylbenzene was obtained by acetylation of 0.5 mol *p*-xylene (Lachema, Brno) with acetic anhydride in 1,1-dichloroethane in the presence of anhydrous aluminium chloride and by subsequent reduction with zinc amalgam<sup>18</sup>. Yield 73% ethyl-2,5-dimethylbenzene related to *p*-xylene. The same method was adopted and similar yields obtained in preparations of ethyl-2,4- and ethyl-3,4-dimethylbenzenes from *m*- and *o*-xylenes, respectively (the both xylenes Lachema, Brno). Ethyl-2,3-dimethylbenzene mixed with the corresponding 2,4-, 2,5-, and 2,6-isomers was prepared by chloromethylation of *o*-ethyltoluene (Urxovy závody, Valašské Meziříčí) and subsequent hydrogenolysis on Pd/C catalyst (in analogy to 4- and 5-methylindanes). 2-Methylindane was prepared according to ref.<sup>19</sup> from benzyl chloride, diethyl methylmalonate (Pfaltz Bauer, USA) and sodium, by subsequent saponification and decarboxylation of the product formed to  $\alpha$ -methylcinnamic acid, conversion of the acid to its chloride by thionyl chloride, cyclization of  $\alpha$ -methylcinnamoyl chloride to 2-methyl-1-indanone, and final reduction with zinc amalgam. Reduction of 2-methyl-1-indanone with sodium borohydride gave 2-methyl-1-indanol whose dehydration (heating with anhydrous sodium sulphate and sodium hydrogen sulphate) gave 2-methylindene (the fraction boiling within 206–206.5°C at atmospheric pressure). Similarly, 3,4-dimethylacetophenone was reduced to 1-(3,4-dimethylphenyl)ethanol whose dehydration gave 3,4-dimethylvinylbenzene. 6-Methylindene was obtained in similar way from 5-methyl-1-indanone which was prepared according to ref.<sup>20</sup> by acylation of toluene with 2-chloropropionyl chloride (BASF, Ludwigshafen, Germany) and cyclization of the product formed with concentrated sulphuric acid.

The other chromatographic standards were commercial samples: 2,4- and 2,5-dimethylvinylbenzenes, 2-methylvinylbenzene, 3- and 4-methylvinylbenzenes, 1,2,4,5- and 1,2,3,5-tetramethylbenzenes (all from ICN Pharmaceuticals, USA), indene, 1,2,3,4-tetrahydronaphthalene, indane, 1,2,4- and 1,3,5-trimethylbenzenes (all from Urxovy závody, Valašské Meziříčí), 3- and 4-methylvinylbenzenes, isopropylbenzene, 1,3- and 1,4-divinylbenzenes (all from Kaučuk, Kralupy), 1,3- and 1,4-diethylbenzene and 1,2,3,4-tetramethylbenzene (all from Aldrich Europe, Belgium).

## RESULTS AND DISCUSSION

The hydrogenation and polymerization experiments with subsequent capillary GLC proved that the low-boiling fraction of pyrolysis oil obtained by continuous rectification at atmospheric pressure contains 49.0% (*m/m*) of unsaturated compounds, 5.9% (*m/m*) of naphthalene and 12.7% (*m/m*) of alkanes; the remaining 32.4% is formed by aromatic hydrocarbons C<sub>8</sub> to C<sub>10</sub>. With respect to boiling range of sample and changes in content of aromatic hydrocarbons after hydrogenation of several selected fractions (Table II) it can be stated that components of highest concentrations are methylindenes (total 24.9% (*m/m*)) followed by dimethylvinylbenzenes (total 11.96% (*m/m*)), 1,2-dihydronaphthalene (3.3% (*m/m*)), and ethylvinylbenzenes (2.5% (*m/m*)). The presence of alkane hydrocarbons is due to technological arrangement used for obtaining liquid products from the pyrolysis mixture and to boiling

TABLE II

Mass balance of some components after hydrogenation of fractions from rectification of low-boiling fraction of pyrolysis oil. Content of both the unsaturated and the corresponding saturated components was determined as relative value with respect to naphthalene content in the fraction in % ( $m/m$ ).

The reaction in question	Fraction boiling from 105.7 to 106.3°C/4 kPa			Fraction boiling from 106.3 to 108.6°C/4 kPa		
	before hydrog.	after hydrog.	rel. error, %	before hydrog.	after hydrog.	rel. error, %
2-methylindene → 2-methylindane	2.97	3.04	+2.3	0.76	0.78	+3.1
4- and 7-methylindene + 1,2-dihydronaphthalene → → 4-methylindane + tetrahydronaphthalene	7.45	7.68	+3.1	1.86	1.79	-3.8
5- and 6-methylindene → 5-methylindane	1.21	1.25	+2.9	0.11	0.11	0.0
1-methylindene → 1-methylindane	0.58	0.60	+3.0	—	—	—

range (the highest concentrations are those of *n*-undecane and *n*-dodecane). The hydrocarbons identified in the low-boiling fraction of pyrolysis oil are summarized in Table III.

The best separation was achieved with the capillary columns wetted with polar stationary phases Reoplex 400 (143 peaks detected) and Carbowax 20M (123 peaks detected), whereas the column wetted with OV-101 (stationary phase of low selectivity) gave imperfect separation especially in the field of unsaturated components; another complication consisted in simultaneous elution of alkanes and aromatics. The column with Reoplex 400 gives better separation of isomeric methylindenes from 1,2-dihydronaphthalene and isomeric divinylbenzenes (Fig. 1), whereas Carbowax 20M appears to be a more selective phase for mutual separation of the present tri- and tetraalkylbenzenes from dimethyl- and ethylvinylbenzenes (Fig. 2).

From the results of the analysis it follows that 1,4-dihydronaphthalene is not present among the unsaturated components (1,4-dihydronaphthalene is eluted between 1,2-dihydronaphthalene and naphthalene from the columns wetted with the both stationary phases); 3-methylindene is present in traces only. The absence of 1,4-dihydronaphthalene could obviously be explained by its low thermostability: it is quantitatively rearranged to 1,2-dihydronaphthalene at temperatures as low as 140–150°C. In contrast to analogous fractions from coal tar, high concentrations of 1,2,3,4- and 1,2,3,5-tetramethylbenzenes were found with all the used capillary columns. Two selected fractions of vacuum rectification of the low-boiling fraction

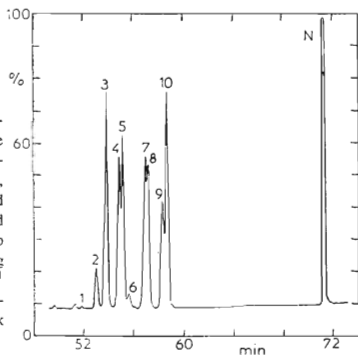


FIG. 1

Separation of isomeric methylindenes, divinylbenzenes, and 1,2-dihydronaphthalene on a column wetted with Reoplex 400. Experimental conditions: glass capillary column, 45 m length, 0.26 mm inner diameter, wetted with Reoplex 400;  $t_c = 70^\circ\text{C}$  was increased after 20 min at a rate  $0.5^\circ\text{C min}^{-1}$  up to  $90^\circ\text{C}$  and, immediately after reaching this temperature, at a rate  $5^\circ\text{C min}^{-1}$  up to  $125^\circ\text{C}$ . Numbers of the peaks correspond to those in Table V. The peak N — naphthalene

of pyrolysis oil were submitted to hydrogenation at atmospheric pressure of hydrogen to provide complementary identification of some unsaturated components. Comparison of concentrations of 4-methylindane, tetrahydronaphthalene, and two isomeric methylindenes (one of which is eluted together with 1,2-dihydronaphthalene from the capillary column wetted with Carbowax 20M) in the non-hydrogenated and hydrogenated fractions (Table II) revealed that from the two main isomeric methylindenes 4-methylindane only is formed: total content of the components 119 and 120 (Table III and Fig. 2) in the sample before the hydrogenation (Table II) corresponds within experimental error to total content of 4-methylindane plus tetrahydronaphthalene in the same sample after hydrogenation. Therefrom it follows that elution of 1,2-dihydronaphthalene is accompanied most probably by that of 4-methylindene which is less polar than 7-methylindene (judged from magnitude of the dipole moments) and should be less retained by a polar stationary phase. Similar method served for identification of 2,3- and 3,5-dimethylvinylbenzenes (in a fraction boiling within 95 to 101°C/4 kPa) whose hydrogenation gave the corres-

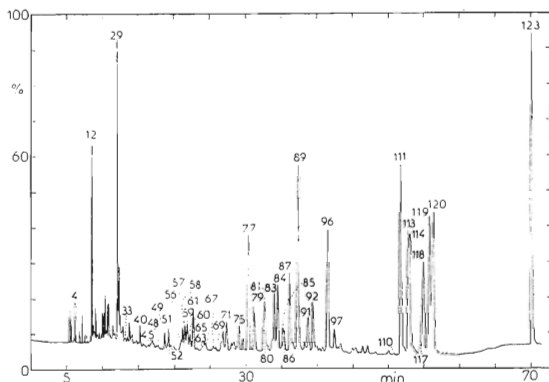


FIG. 2

Analysis of low-boiling fraction of pyrolysis oil on a column wetted with Carbowax 20M. Experimental conditions: glass capillary column, 42 m length, 0.26 mm inner diameter, wetted with Carbowax 20M,  $t_c = 70^\circ\text{C}$  was increased after 20 min at a rate  $0.5^\circ\text{C min}^{-1}$  up to  $90^\circ\text{C}$  and immediately after reaching this temperature, at a rate  $5^\circ\text{C min}^{-1}$  up to  $125^\circ\text{C}$ . Numbers of peaks, correspond to those in Table III

TABLE III

Composition of low-boiling fraction of pyrolysis oil and the Kováts elution indices of the identified aromatic hydrocarbons for the column No 1 at temperature 80°C

Component No <sup>a</sup>	Component name	Content % (m/m)	Elution index
1 to 3	isodecanes	0.7	—
4	n-decane	0.2	—
5 to 11	isoundecanes	0.4	—
12	n-undecane	1.1	—
13 to 28	isododecanes	6.5	—
29	n-dodecane	3.74	—
33	1,3,5-trimethylbenzene	0.4	1 219
35	vinylbenzene	0.6	1 237
40	1,2,4-trimethylbenzene	0.4	1 260
45	1,3-diethylbenzene	0.2	1 281
48	1,4-diethylbenzene	0.1	1 289
49	methyl-4-n-propylbenzene	0.1	1 290
51	ethyl-3,5-dimethylbenzene	0.3	1 299
52	isopropylbenzene	0.1	1 305
56	2-methylvinylbenzene	0.2	1 320
57	ethyl-2,4-dimethylbenzene	0.5	1 323
58	3- and 4-methylvinylbenzenes	0.6	1 325
59	ethyl-2,5-dimethylbenzene	0.8	1 328
60	indane	0.5	1 331
61	ethyl-3,4-dimethylbenzene	1.0	1 335
63	2-methylindane	0.1	1 345
65	ethyl-2,6-dimethylbenzene	0.3	1 349
67	1-methylindane	0.2	1 357
69	2,6-dimethylvinylbenzene	0.3	1 362
71	ethyl-2,3-dimethylbenzene	0.8	1 369
75	1,2,4,5-tetramethylbenzene	0.8	1 380
77	1,2,3,5-tetramethylbenzene	3.8	1 389
79	3-ethylvinylbenzene	1.8	1 393
80	4-ethylvinylbenzene	0.7	1 400
81	2,5-dimethylvinylbenzene	1.7	1 402
83	2,4-dimethylvinylbenzene	1.8	1 410
84	2,3-dimethylvinylbenzene	2.3	1 412
85	5-methylindane	0.7	1 419
86	unknown unsaturated component	0.5	—
87	indene	2.5	1 424
89	1,2,3,4-tetramethylbenzene	5.0	1 430
91	4-methylindane	1.2	1 438
92	3,5-dimethylvinylbenzene	1.8	1 440
96	3,4-dimethylvinylbenzene	4.1	1 451
97	tetrahydronaphthalene	1.0	1 457



TABLE III  
(Continued)

Component No <sup>a</sup>	Component name	Content % (m/m)	Elution index
110	3-methylindene	0.1	1 493
111	1-methylindene + 1,3-divinylbenzene	7.2	1 498
113	5-methylindene	4.0	1 506
114	6-methylindene	4.0	1 506
117	1,4-divinylbenzene	0.3	1 510
118	2-methylindene	3.5	1 514
119	4-methylindene + 1,2-dihydronaphthalene	6.5	1 518
120	7-methylindene	5.1	1 520
123	naphthalene	5.9	1 617

<sup>a</sup> Numbers of the components agree with those in Fig. 2.

ponding ethyldimethylbenzenes which were identified on the basis of published elution data<sup>21</sup>. Theoretical elution indices of the two said dimethylvinylbenzenes were determined on the basis of the increments for methyl and vinyl groups found from elution data of benzene, vinylbenzene and its methyl homologues (Table-IV), and two distinct peaks of unsaturated components eluted in this region were assigned thereto. The best agreement was achieved in the case of 2,3-dimethylvinylbenzene whose theoretical elution index (calculated by addition of increment of 2-methyl group to experimental elution index of 3-methylvinylbenzene) is 1 416, the corresponding experimental value being 1 418. A substantially worse agreement was obtained in the case of 3,5-dimethylvinylbenzene which was assigned to the peak having the elution index 1 450. The elution index value 1 516 predicted theoretically by addition of increment of vinyl group to experimental elution index of *m*-xylene is considerably different, the same being true of the elution index 1 421 predicted by addition of 3-methyl group increment to experimental elution index of 3-methylvinylbenzene. This fact can be explained by a previous finding<sup>22</sup> that the increments of elution indices of alkyl groups observed with columns wetted with polar stationary phases do not fully reflect mutual polar interactions of these groups in the resulting system, which results in considerable worsening of agreement between theoretical and experimental elution indices as compared with non-polar stationary phases.

The separation on the capillary column wetted with Reoplex 400 enabled a more precise determination of content of 1,3-divinylbenzene, 4-methylindene, 1,2-dihydro-

naphthalene, and 1-methylindene; the elution data and content of the individual isomers are given in Table V. Linear relation between elution behaviour of the analyzed aromatic hydrocarbons on the two stationary phases (Carbowax 20M and Reoplex 400) can be expressed as:  $I_{\text{REO}} = 1.360 \cdot I_{\text{CWX}} + 578.7$  (correlation coefficient  $r = 0.9960$  for 10 measurements), where  $I_{\text{REO}}$  and  $I_{\text{CWX}}$  are the Kováts elution indices for the columns wetted with Reoplex 400 and Carbowax 20M, respectively. The content of hydrocarbons in low-boiling fraction of pyrolysis oil given in Tables III and

TABLE IV

Elution increments of methyl and vinyl groups bound to aromatic nucleus found on capillary column wetted with Carbowax 20M at temperature 75°C

Aromatic hydrocarbon	Elution index	$\Delta I$	Way of calculation
Benzene	836	—	—
<i>m</i> -Xylene	1 119	—	—
Vinylbenzene	1 232	396	$I_{\text{PhCH}=\text{CH}_2} - I_{\text{PhH}}$
2-Methylvinylbenzene	1 322	90	$I_{2-\text{MePhCH}=\text{CH}_2} - I_{\text{PhCH}=\text{CH}_2}$
3- and 4-Methylvinylbenzenes	1 327	95	$I_{3-\text{MePhCH}=\text{CH}_2} - I_{\text{PhCH}=\text{CH}_2}$

TABLE V

Elution indices of some unsaturated aromatic hydrocarbons found on a capillary column wetted with Reoplex 400 at 80°C and content of these hydrocarbons in low-boiling fraction

Component No <sup>a</sup>	Aromatic hydrocarbon	Elution index	Content, % (m/m)
1	3-methylindene	1 525	0.1
2	1,3-divinylbenzene	1 528	2.1
3	1-methylindene	1 531	5.2
4	5-methylindene	1 534	3.7
5	6-methylindene	1 535	5.3
6	1,4-divinylbenzene	1 535.5	0.3
7	2-methylindene	1 539	3.5
8	1,2-dihydronaphthalene	1 540	3.3
9	4-methylindene	1 543	2.2
10	7-methylindene	1 544	5.1

Numbers of the components agree with those in Fig. 1.

V was determined from relative surface area of the individual corresponding peaks; the response factor was neglected with respect to structural similarity of the compounds present.

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