ISSN 1070-3632, Russian Journal of General Chemistry, 2016, Vol. 86, No. 10, pp. 2325–2331. © Pleiades Publishing, Ltd., 2016.
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Synthesis and Antiradical Activity of Hybrid Antioxidants Based on Isobornylphenols

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Received March 14, 2016

Abstract—Alkylation of isobornylphenols with allylbenzene in the presence of homogeneous and heterogeneous catalysts of different nature has been studied. The maximum yield of phenols containing isobornyl and 1-phenylpropyl moieties has been achieved in the presence of catalyst FIBAN K-1 at 100°C and catalyst concentration of 10%. The inhibiting action of isobornylphenol derivatives has been studied in the model reaction of ethylbenzene oxidation. Hybrid antioxidants on the basis of isobornylphenols were found to actively interact with peroxide radicals and, therefore, they can be considered as promising additives for conservation of quality and increase in service life of different organic compounds and materials.

Keywords: isobornylphenols, alkylation, phenyl(alkyl)isobornylphenols, hybrid antioxidants, antiradical activity

DOI: 10.1134/S1070363216100133

Nowadays, sterically hindered phenols are widely used in polymer industry [1–4]. Thus, antioxidants of phenol type cover more than 50% of the world market of stabilizers for plastics and about 30% for resins and caoutchoucs [5]. Substituted phenols inhibit free-radical oxidation of organic substrates and improve working characteristics of articles. Effectiveness of alkylphenols strongly depends on the size and the position of the substituents in the aromatic ring [6]. Besides, the advantage of this class of compounds is a low toxicity as well as a good solubility in hydrocarbons and practical insolubility in water.

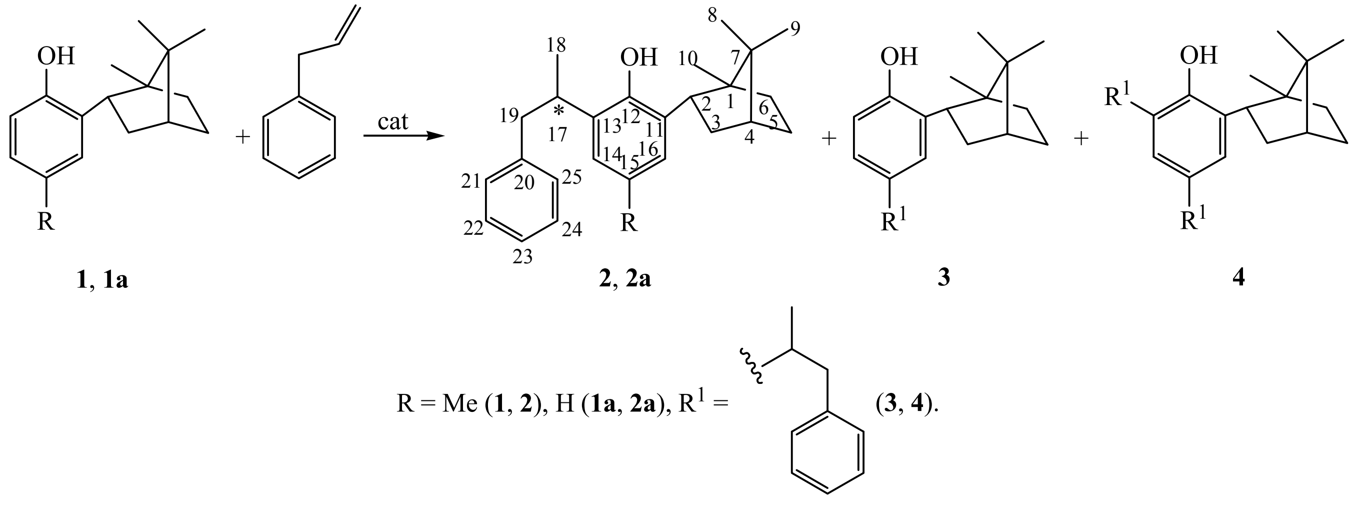
A promising direction in improving the properties of antioxidants is elaboration of polyfunctional stabilizers or hybrid structures on the basis of phenols. Introduction in the structure of phenols of bulky substituents of different nature may lead to a change of physico-chemical properties.

Earlier, we have synthesized the hybrid phenols containing in one molecule a bulky isobornyl and *tert*-butyl or 1-phenylethyl fragments [7, 8]. The estimation of the antioxidant activity of isobornylphenols with *tert*-butyl substituents showed that the hybrid compounds have higher antioxidant activity than the liposoluble antioxidant BHT, but do not exceed antioxidant activity of trolox (water-soluble analog of tocopherol).

In the present work, alkylation of racemic 2-isobornyl-4-methylphenol **1** and 2-isobornylphenol **1a** with allylbenzene was studied using homogeneous [$(CH_3C_6H_4O)_3Al$, $TsOH{\cdot}H_2O$, $AlCl_3$] and heterogeneous (FIBAN K-1, Amberlyst 15, Amberlyst 36, Amberlyst 36 Dry and KSF) catalysts. The antiradical activity of the synthesized compounds was assessed.

Of practical interest for the synthesis of difficultly accessible *o*-substituted alkylphenols is the use of aluminum phenolate as the catalyst. Earlier we have shown that alkylation of phenol with camphene in the presence of aluminum phenolate occurs with high regioselectivity and good yield of *o*-isobornylphenols (87%) [9]. Unfortunately, in the reaction studied here this catalyst was ineffective (Table 1). When using the Lewis acid catalyst $AlCl_3$ the starting phenol is dealkylated. In the presence of $TsOH{\cdot}H_2O$ at 100–160°C the reaction does not take place either, while the increase in the temperature of the reaction mixture to 180°C promotes isomerization of the terpene moiety of the starting isobornylphenol and the yield of product **2** is small (30%) (Scheme 1).

Scheme 1.



Heterogeneous catalysts have such advantages over the homogeneous ones as the easy separation from the reaction mixture and the possibility of multiple reuse.

KSF clay is a solid Lewis acid containing sulfonic groups SO_3H^+ on the surface. This catalyst was shown to be effective in the synthesis of phenols containing the isobornyl and *tert*-butyl substituents in one molecule [8]. However, alkylation of phenol **1** with allylbenzene in the presence of KSF does not occur, either.

FIBAN K-1 is a polypropylene fiber with grafted copolymer of styrene and divinylbenzene, containing sulfonic groups SO_3H^+, and it is an effective catalyst for the synthesis of methyl-*tert*-alkyl ethers [10]. The use of FIBAN K-1 in the studied reaction leads to the formation of products of alkylation **2**, **2a**, **3**, and **4** with moderate conversion of the starting phenols **1** and **1a**. The effect of the catalyst concentration, the ratio of the reagents, and the temperature of the reaction mixture was studied. It was found that the increase in the catalyst amount from 5 to 10% caused the increase in the conversion of the starting phenol and the yield of the target product. The increase in the temperature to 140°C increases the rate of the reaction, but the conversion and the yield vary only slightly because of the processes of dealkylation of the starting isobornylphenol and isomerization of the terpene fragment. In the reaction of 2-isobornyl-4-methylphenol **1** with allylbenzene the maximum yield of product **2** (63–66%) was achieved at the temperature of 100°C and the amount of the catalyst of 10%. Variation of the molar ratio of the reagents (1 : 1 or 1 : 2) does not affect the yield of product **2**. Note that the use of excess of allylbenzene does not increase the conversion of the starting phenols. Performing the reaction at the ratio phenol : allylbenzene = 2 : 1 did not allow to increase the yield of the target product either. In this case, dealkylation of phenol **1** to cresol with subsequent alkylation of *p*-cresol with allylbenzene occurred; 10% of *p*-cresol and 21% of 2-phenylpropyl-4-methylphenol was obtained.

Alkylation of phenol **1a** gives regioisomers **2a** and **3**, as well as the disubstituted product **4**. Note that in the presence of sulfocationic catalysts FIBAN K-1 and Amberlyst 15, 36 and 36 Dry, the *p*-alkylated phenol **3** is formed in high yield. The largest yield (44%) of product **3** was achieved in the presence of catalyst FIBAN K-1 with equimolar ratio of the reagents. Using the excess of allylbenzene increases the yield of the products of dialkylation.

Polystyrene Amberlyst sulfonic acid resins effectively catalyze hydration and etherification of olefins, dehydration of alcohols, and alkylation of phenols [11, 12]. The amount of Amberlyst catalyst (10%) was chosen according to the literature data [13].

In the presence of macroporous cationites Amberlyst 15, Amberlyst 36, and Amberlyst 36 Dry the reactions of phenols **1** and **1a** proceed with incomplete conversion (42–68%); besides, the products with the isomerized terpene fragment are formed. Products **2**, **2a**, **3,** and **4** are formed in the presence of all studied Amberlyst catalysts practically in equal amounts irrespective of the temperature of the reaction mixture and the ratio of the reagents. Therefore, it is impossible to choose the most effective catalyst.

Table 1. Alkylation of isobornylphenols with allylbenzene

Phenol	Catalyst	Phenol: allylbenzene	*T*, °C	Time, h	Conversion, %	Composition of the products of alkylation, %			
						2	**2a**	**3**	**4**
1	$(CH_3C_6H_4O)_3Al$ (10 wt %)	1 : 1	160	15	–	–	–	–	–
	$TsOH{\cdot}H_2O$ (26 mol %)	1 : 1	180	3	78	30	–	–	–
	$AlCl_3$ (10 wt %)	1 : 1	100	0.5	100	–	–	–	–
	KSF (100 wt %)	1 : 1	100	7	–	–	–	–	–
	FIBAN K-1 (5 wt %)	1 : 1	100	10	58	57	–	–	–
		1 : 2	100	10	56	60	–	–	–
		1 : 1	140	3	66	53	–	–	–
		1 : 2	140	3	62	55	–	–	–
		2:1	140	3	100[a]	35	–	–	–
	FIBAN K-1 (10 wt %)	1 : 1	100	10	72	66	–	–	–
		1 : 2	100	10	72	63	–	–	–
		1 : 1	140	3	73	60	–	–	–
	Amberlyst 15 (10 wt %)	1 : 1	100	6	68	52	–	–	–
		1 : 2	100	6	66	55	–	–	–
	Amberlyst 36 10 wt %	1 : 1	100	6	42	54	–	–	–
		1 : 1	140	6	56	52	–	–	–
		1 : 2	140	6	54	50	–	–	–
	Amberlyst 36 Dry (10 wt %)	1 : 1	100	6	60	54	–	–	–
		1 : 2	140	6	56	52	–	–	–
1a	$AlCl_3$ (10 wt %)	1 : 1	100	0.5	100	–	–	–	–
	FIBAN K-1 (5 wt %)	1 : 1	140	3	72	–	19	38	21
		1 : 2	140	3	75	–	16	33	25
	FIBAN K-1 (10 wt %)	1 : 1	100	6	74	–	19	44	17
		1 : 2	100	6	80	–	15	35	28
	Amberlyst 15 (10 wt %)	1 : 1	100	3	54	–	16	35	19
		1 : 2	100		66	–	15	40	24
	Amberlyst 36 (10 wt %)	1 : 1	140	6	54	–	12	40	9
		1 : 2	140	6	62	–	14	36	17

[a] Conversion calculated to allylbenzene.

The structure of the products was proved by NMR spectroscopy. In the ^1H and ^{13}C NMR spectra of phenols **2**, **2a,** and **3**, apart from the signals of terpenophenol moiety, the signals of phenylpropyl substituent are present.

Since the starting isobornylphenols **1** and **1a** are racemates, and in the course of the reaction an additional chiral center at C^{17} is formed, the products of alkylation **2**, **2a,** and **3** are present as mixtures of diastereomers in the ratio ~1 : 1. Unfortunately, we

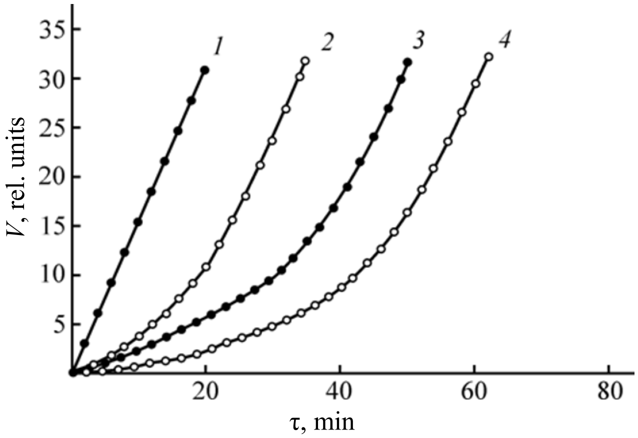


Fig. 1. Kinetic curves of oxygen absorption in the initiated oxidation of ethylbenzene in the (*1*) absence and (*2–4*) in the presence of antioxidant **2** in different concentrations (mol/L): (*2*) 3×10^{-5}, (*3*) 6×10^{-5}, (*4*) 7.6×10^{-5}. T = 333 K, $w_i = 5\times10^{-8}$ mol L^{-1} s^{-1}.

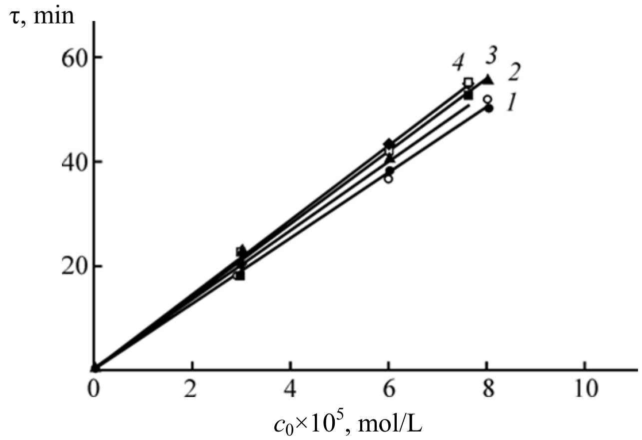


Fig. 2. Dependence of the induction period in the initiated oxidation of ethylbenzene on the initial concentration of additives: (*1*) **2a**, **6**; (*2*) **7**; (*3*) **3**; (*4*) **2**, **5**. T = 333 K, w_i = 5×10^{-8} mol L^{-1} s^{-1}.

failed to isolate individual diastereomers by column chromatography or crystallization.

We have assessed the antiradical activity of phenols **2**, **2a**, **3** with respect to the earlier synthesized compounds **5–7** [7], containing the 1-phenylethyl fragment (Scheme 2).

We used the model reaction of initiated oxidation of ethylbenzene, in which free radicals propagating the oxidation chains are generated with a constant rate. As an example, in Fig. 1 the kinetic curves of the absorption of oxygen in the presence of antioxidant **2** are shown. As can be seen, the oxygen absorption rate notably decreases in the presence of **2**, and a clearly expressed induction period (τ) is observed, whose value increases with the initial concentration c_0 (Fig. 2). Similar data were obtained for all studied samples.

From the kinetic curves of the oxygen absorption in the presence of isobornylphenols **2–3** and **5–7** the induction period (τ) was determined using the procedure described in [14], and the initial rates of oxidation. From the slope of the linear dependence of τ on c_0 (Fig. 2) the stoichiometric inhibition coefficient (f) was calculated by Eq. (1).

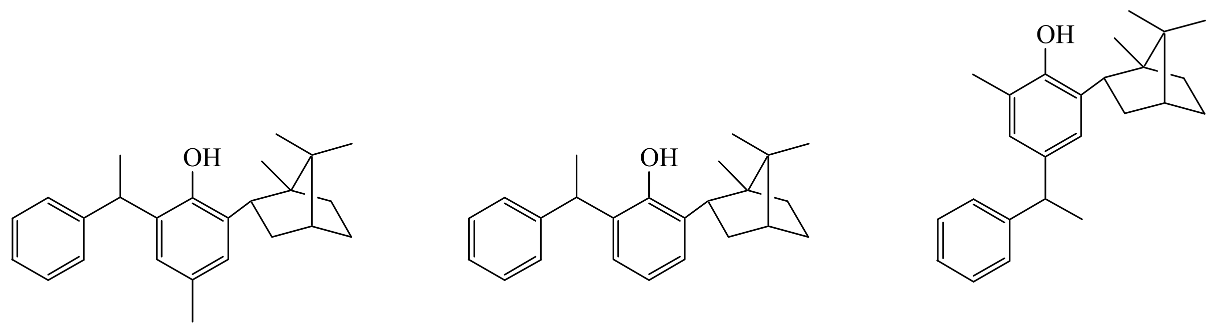
$$f = \tau w_i/c_0, \qquad (1)$$

where w_i denotes the rate of initiation. It can be seen from Table 2 that the values of f for isobornylphenols are close to 2. This is in good accordance with the mechanism of action of phenolic antioxidants, according to which one molecule of phenol causes the decay of two free radicals.

To calculate the rate constants of the reaction of antioxidants with peroxide radicals the values of the initial rates of oxidation were represented in the coordinates of Eq. (2) [15]:

$$w_0/w - w/w_0 = fk_7c_0/k_6^{0.5}w_i^{0.5}, \qquad (2)$$

Scheme 2.



5 **6** **7**

where w_0 and w are the rates of oxidation in the absence and in the presence of the additives, respectively, k_6 is the rate constant for quadratic recombination of peroxide radicals ($k_6 = 1.9\times10^7$ mol L^{-1} s^{-1} [16]). From the slope of straight lines in Fig. 3, parameter fk_7 was calculated (Table 2).

As evident from the data of Table 2, the studied compounds are active antioxidants, their k_7 constant being dependent on the number, nature and location of the substituents with respect to the OH group of the phenol. Constant k_7 increases with the number of alkyl substituents and with the phenylpropyl substituent in place of the phenylethyl group. For dialkyl-substituted phenols, introduction of the bulky phenylpropyl substituent to the *p*-position to the OH group in **3**, as compared to the *o*-substituted analog **2a** results in two-fold increase in k_7 constant. The k_7 constant is known to sharply decrease with the introduction of two bulky (for example, *tert*-butyl) substituents in the *o*-position of the phenol molecule. This occurs because bulky substituents create steric hindrances to the reaction of phenol with peroxide radicals [17].

Therefore, we have shown that the maximum yield of product **2** (63–66%) can be achieved in the presence of FIBAN K-1 catalyst in concentration of 10%. In alkylation of 2-isobornylphenol in the presence of sulfocationic catalysts FIBAN K-1 and Amberlyst, the *p*-alkylated phenol **3** is formed in high yield. The obtained hybrid antioxidants, along with low toxicity and good solubility in hydrocarbons, actively react with peroxide radicals and can be considered as promising additives for preservation of quality and increase in the durability of various organic compounds and materials.

EXPERIMENTAL

Catalysts Amberlyst 15 (Fluka), Amberlyst 36 (Aldrich) and Amberlyst 36 Dry were crushed and calcined at 120°C for 1 h. Montmorillonite KSF (Acros Organics) with the content of 8–12% of free H_2SO_4, $AlCl_3$, and $TsOH\cdot H_2O$ were used without preliminary treatment. FIBAN K-1 catalyst was provided by our colleagues from Institute of Physical Organic Chemistry of the Belarus National Academy of Sciences.

IR spectra were recorded on a Shimadzu IR Prestige 21 spectrometer in KBr. Melting points were determined on a Sanyo Gallenkamp MDP350 unit and are uncorrected. ^{1}H and ^{13}C NMR spectra were registered on a Bruker Avance II 300 spectrometer with working frequencies 300.17 and 75.48 MHz from $CDCl_3$ solutions. Chemical shifts are given relative to the signals of the solvent, the signals were assigned using ^{13}C JMOD spectra.

Table 2. Stoichiometric inhibition coefficients (f) and rate constants of the reaction of antioxidants with peroxide radicals of ethylbenzene (k_7)

Compound	**2**	**2a**	**3**	**5**	**6**	**7**
$fk_7\times10^{-4}$, L mol^{-1} s^{-1}	20.0	6.6	15.3	17.4	5.2	1.6
f	2.15	1.9	2.1	2.15	1.9	2.0
$k_7\times10^{-4}$, L mol^{-1} s^{-1}	9.3	3.5	7.1	8.1	2.7	8.0

The reactions were monitored by TLC on Sorbfil plates (eluent cyclohexane–Et_2O, 50 : 1). The plates were developed by treatment with the solution of 15 g $KMnO_4$, 300 mL H_2O, and 0.5 mL of conc. H_2SO_4. The products were separated by column chromatography on silica Alfa Aesar (70/230μ).

The inhibiting activity of the hybrid compounds was studied by volumetric method in the model reaction of oxidation of ethylbenzene initiated by azobisisobutyronitrile. The kinetics of oxygen absorption during oxidation was registered by the use of highly sensitive volumetric device at 333 K and the rate of initiation $w_i = 5\times10^{-8}$ mol L^{-1} s^{-1}. Ethylbenzene with the dissolved initiator was preliminarily maintained at

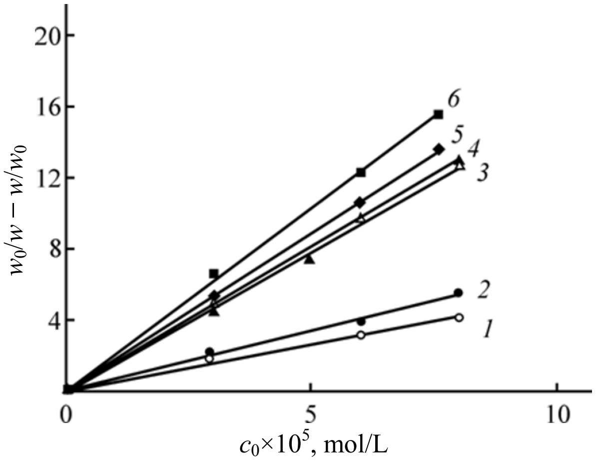


Fig. 3. Dependence of the initial rate of inhibited oxidation of ethylbenzene on the initial concentration of additives in the coordinates of Eq. (2): (*1*) **6**, (*2*) **2a**, (*3*) **3**, (*4*) **7**, (*5*) **5**, (*6*) **2**. $T = 333$ K, $w_i = 5\times10^{-8}$ mol L^{-1} s^{-1}.

a desired temperature, then the additive of antioxidant was introduced, and the kinetics of oxygen absorption was registered.

Alkylation of isobornylphenols with allylbenzene. Two-necked flask equipped with a condenser and a thermometer was charged with the calculated amounts of 2-isobornyl-4-methyl-phenol or 2-isobornylphenol and allylbenzene. Homogeneous [$TsOH{\cdot}H_2O$, $(TolO)_3Al$, $AlCl_3$] and heterogeneous sulfocationic catalysts (FIBAN K-1, Amberlyst 15, Amberlyst 36, Amberlyst 36 Dry and KSF) were used. The reagents and the catalyst were charged simultaneously. Reaction conditions are given in Table 1. The reaction mixture was separated by column chromatography (Silica gel 70/230 μ, gradient elution with cyclohexane-Et_2O with increasing fraction of the latter).

4-Methyl-2-(1-phenylpropan-2-yl)-6-(1,7,7-trimethylbicyclo[2.2.1]hept- *exo*-2-yl)phenol (2). Colorless powder with mp 95–98°C. IR spectrum, ν, cm^{-1}: 3522, 2953, 2924, 1454, 1458 (CH_2, CH_3), 1600 (C=C), 864 (C-H$_{Ar}$). ^{1}H NMR spectrum, δ, ppm: 0.59 and 0.77 s (3H, CH_3^{10}), 0.84 and 0.86 s (3H, CH_3^{18}), 0.90 and 0.92 s (3H, CH_3^{8}), 0.97 s (3H, CH_3^{9}), 1.35–1.39 m (2H, H^5, H^6), 1.52–1.66 m (2H, H^3, H^6), 1.80–1.95 m (2H, H^4, H^5), 2.03–2.16 m (2H, H^{19}), 2.18–2.27 m (1H, H^3), 2.34 s (3H, CH_3^{para}), 2.99-3.09 m (1H, H^2), 3.97–4.05 m (1H, H^{17}), 4.49 and 4.51 s (1H, OH), 6.95 and 6.98 s (1H, H^{16}), 7.02 s (1H, H^{14}), 7.23–7.33 m (5H, H$^{21–25}$). ^{13}C NMR spectrum, δ, ppm: 12.03 and 12.22 (C^{10}), 12.68 and 12.75 (C^9), 20.26 and 20.34 (C^8), 21.33 and 21.48 (C^{para}, C^{18}), 27.56 (C^5), 28.04 (C^{19}), 29.55 (C^3), 33.98 (C^6), 45.59 and 45.72 (C^{17}), 46.86 (C^2), 47.16 (C^4), 48.10 (C^7), 49.66 and 49.75 (C^1), 125.65 and 126.06 (C^{14}), 126.07 (C^{23}), 126.45 and 126.66 (C^{16}), 128.01 and 128.14 (C^{22,24}), 128.40 (C^{11}), 128.61 and 128.65 (C^{21,25}), 129.58 and 129.88 (C^{13}), 130.08 and 130.18 (C^{20}), 144.16 (C^{15}), 150.48 and 150.64 (C^{12}). Found, %: C 86.13; H 9.45. $C_{26}H_{34}O$. Calculated, %: C 86.4; H 9.61.

2-(1-Phenylpropan-2-yl)-6-(1,7,7-trimethylbicyclo[2.2.1]hept-*exo*-2-yl)phenol (**2a**). Colorless oil. IR spectrum, ν, cm^{-1}: 3604, 3531, 2953, 2933, 2873, 1598, 1444, 702. ^{1}H NMR spectrum, δ, ppm: 0.62 and 0.80 s (3H, CH_3^{10}), 0.86 and 0.89 s (3H, CH_3^{18}), 0.93 and 0.95 s (3H, CH_3^{9}), 1.00 s (3H, CH_3^{8}), 1.30–1.49 m (2H, H^{5,6}), 1.56–1.72 m (2H, H^{3,6}), 1.81–1.99 m (2H, H^{4,5}), 2.09–2.18 m (2H, H^{19}), 2.23–2.27 m (1H, H^3), 3.04-3.14 m (1H, H^2), 4.02–4.11 m (1H, H^{17}), 4.71 and 4.73 s (1H, OH), 6.98 t (1H, J = 7.5 Hz, H^{15}), 7.18–7.37 m (7H, H$^{14,16,21–25}$). ^{13}C NMR spectrum, δ, ppm: 12.05 and 12.24 (C^{10}), 12.66 and 12.73 (C^9), 20.23 and 20.34 (C^8), 21.33 and 21.47 (C^{18}), 27.58 (C^5), 28.07 (C^{19}), 34.03 and 34.08 (C^3), 39.90 and 39.98 (C^6), 45.60 and 45.66 (C^{17}), 46.88 (C^2), 47.18 (C^4), 48.09 and 48.13 (C^7), 49.69 and 49.79 (C^1), 119.68 (C^{15}), 125.15 and 125.54 (C^{14}), 126.04 (C^{16}), 126.54 (C^{23}), 128.02 and 128.15 (C^{22,24}), 128.65 and 128.70 (C^{21,25}), 130.18 and 130.22 (C^{13}), 130.35 (C^{11}), 144.03 and 144.09 (C^{20}), 152.93 (C^{12}).

4-(1-Phenylpropan-2-yl)-2-(1,7,7-trimethylbicyclo[2.2.1]hept-*exo*-2-yl)phenol (**3**). Light-brown oil. IR spectrum, ν, cm^{-1}: 3535 (OH), 2953, 2929, 2876, 1604, 1501, 1454, 1381, 702. ^{1}H NMR spectrum, δ, ppm: 0.82 and 0.83 s (3H, CH_3^{10}), 0.90 s (3H, CH_3^{9}), 0.92 s (3H, CH_3^{8}), 0.94–0.98 m (3H, J = 7.5 Hz, CH_3^{18}), 1.41–1.55 m (2H, H^{5,6}), 1.64–1.75 m (2H, H^{3,6}), 1.90–1.99 m (2H, H^{4,5}), 2.08–2.12 m (2H, H^{19}), 2.14–2.29 m (1H, H^3), 3.17 t (1H, J = 8.7 Hz, H^2), 3.79 t (1H, J = 7.8 Hz, H^{17}), 4.81 s (1H, OH), 6.71 d (1H, J = 8.1 Hz, H^{14}), 6.92–6.97 m (1H, H^{13}), 7.24–7.33 m (6H, H$^{16,21–25}$). ^{13}C NMR spectrum, δ, ppm: 12.48 and 12.51 (C^{10}), 12.80 and 12.84 (C^9), 20.17 and 20.21 (C^8), 21.51 (C^{18}), 27.59 (C^5), 28.67 (C^{19}), 34.13 and 34.18 (C^3), 40.12 and 40.16 (C^6), 45.57 and 45.67 (C^{17}), 48.02 (C^7), 49.77 (C^1), 52.69 and 52.82 (C^{2,4}), 114.82 and 114.85 (C^{13}), 125.83 (C^{14,16}), 127.88 and 128.03 (C$^{22–24}$), 128.30 (C^{21,25}), 129.32 and 129.34 (C^{11}), 136.57 and 136.73 (C^{15}), 145.78 (C^{20}), 152.84 (C^{12}).

ACKNOWLEDGMENTS

This work was performed with the support from the Program “UMNIK” (no. 4799GU1/2014), partially financed by the Program of Ural Branch of Russian Academy of Sciences (no. 15-6-3-6). Spectral measurements were performed with the use of equipment of the Center for Joint Usage “Chemistry” of the Institute of Chemistry of Komi Scientific Center, Ural Branch, Russian Academy of Sciences.

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