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Copper (II) chloride / tetrabutylammonium bromide catalyzed oxidation of 2,6-diisopropylnaphthalene and 4,4'-diisopropylbiphenyl

Research Article

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Abstract: The oxidation processes of 2,6-diisopropylnaphthalene and 4,4'-diisopropylbiphenyl with oxygen in the presence of a catalyst, composed of copper(II) chloride and tetrabutylammonium bromide, were investigated. It was found that, in essence, only one isopropyl group undergoes oxidation, and obtained mixtures contained mainly peroxide, alcohol, ketone and only small amounts of hydroperoxide.

Keywords: *Catalytic oxidation • 2,6-Diisopropylnaphthalene • 4,4'-Diisopropylbiphenyl • Peroxides*

1. Introduction

The oxidation of isopropylaromatic hydrocarbons with oxygen in the liquid phase is one step in the synthesis of hydroxyaromatic compounds and acetone. Every year, more than 7 million tonnes of phenol is synthesized by the Hock method consisting of alkylation of benzene with propylene, oxidation of cumene to hydroperoxide and acid decomposition of hydroperoxide to phenol and acetone [1]. Due to the problem of acetone surplus on the market alternative methods of phenol synthesis are needed, which avoids the co-production of acetone. The methods of direct benzene oxidation to phenol using N_2O, H_2O_2 or O_2/H_2 were described [1-4].

Analogous industry scale syntheses based on the Hock method are compiled for hydroquinone, resorcinol, cresols and 2-naphthol [5,6]. In the literature, we can also find information about oxidation processes for 2,6-diisopropylnaphthalene (DIPN) [7,8] and 4,4'-disopropylbiphenyl (DIPB) [9,10] with oxygen to form hydroperoxides (Schemes 1 and 2). They are subsets of research on the synthesis of 2,6-dihydroxynaphthalene, 2-hydroxy-6-(1-methylethyl) naphthalene, 4,4'-dihydroxybiphenyl and 4-hydroxy-4'-(1-methylethyl)biphenyl, which can be used in the synthesis of liquid crystal polymers, epoxy resins, crop protection chemicals and pharmaceuticals [11].

Oxidation of DIPN to hydroperoxides was conducted mostly within the temperature range of 80-110°C, under a pressure of 0.1 to 0.5 MPa. The use of solvents like acetonitrile, heptane, methylisobutyl ketone or alkaline aqueous emulsion has been described [7]. For instance, oxidation of DIPN at 105°C in the presence of an azo initiator leads to hydroperoxides with a selectivity of 82% and a conversion of 50% [8]. Similar conditions have been applied to DIPB oxidation [9]. Uses of solvents, alkalis and surfactants have been described. Thus, non-catalytic oxidation of DIPB carried out at 110°C in tert-butylbenzene as the solvent leads to hydroperoxides with a selectivity of 90% and a conversion of 66% [10]. Recently, application of N-hydroxyphtalimide (NHPI) as a catalyst in oxidation reactions of DIPN and DIPB has been described [12-15]. Oxidation of DIPN in the presence of NHPI and AIBN followed by acid decomposition led to 2,6-dihydroxynaphthalene, with a yield of 90% [12,13]. On the other hand, oxidation of both compounds in the presence of a catalytic system consisting of NHPI and $Co(OAc)_2$ led to 2,6-di(1-hydroxy-1-methylethyl)naphthalene or 4,4'-di(1-hydroxy-1-methylethyl)biphenyl in high yields, 90%, from which

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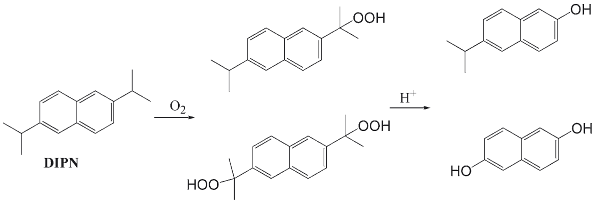
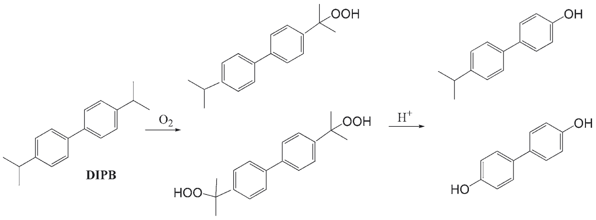


Scheme 1. 2,6-Dihydroxynaphthalene and 2-hydroxy-6-(1-methylethyl)naphthalene synthesis by the Hock method.



Scheme 2. 4,4’-Dihydroxybiphenyl and 4-hydroxy-4’-(1-methylethyl)biphenyl synthesis by the Hock method.

2,6-dihydroxynaphthalene and 4,4’-dihydroxybiphenyl were synthesized in a simultaneous process of oxidation with hydrogen peroxide and acid decomposition [14,15].

The present paper deals with investigations concerning the oxidation of hydrocarbons, DIPN and DIPB, in the presence of a catalytic system consisting of copper(II) chloride and tetrabutylammonium bromide (TBAB). This catalyst was previously used in the oxidation of aromatic hydrocarbons with one isopropyl group, such as cumene [16,17] and 2-isopropylnaphtalene [18], and products contained considerable amounts of alcohol, ketone and peroxide, and only small amounts of hydroperoxide were obtained. The aim of this research was to determine whether oxidation of DIPN and DIPB in the presence of Cu(II)/TBAB catalyst can be used in the synthesis of corresponding peroxides, mono- and di-alcohols and ketones, which can potentially be used as initiators, flavour compounds and monomers for polymers with special abilities.

2. Experimental Procedure

2,6-Diisopropylnaphthalene (DIPN) > 99%, m.p. 66.8°C; 4,4’-diisopropylbiphenyl (DIPB) > 99%, m.p. 66°C. The catalysts ($CuCl_2{\bullet}2H_2O$, TBAB) and solvents are commercially available.

A Waters Alliance 2690 HPLC equipped with an autosampler and UV detector (Waters photodiode array) was used. Column Nova-Pak Silica 60 Å 4 μm (150 mm×3.9 mm; Waters) was applied with a gradient elution of a mixture of hexane and 2-propanol as a mobile phase [19,20]. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Varian Unity Inova-300 spectrometer. The melting points were determined in capillary tubes on SMP 3 (Stuart Scientific) apparatus.

2.1. Catalytic oxidation of DIPN and DIPB with oxygen

DIPN and DIPB were oxidized in a glass reactor (ø 2.8 cm; H 6.5 cm) supplied with a mechanical stirrer (1800 rpm), bubbler, thermometer, reflux condenser and heating jacket. The hydrocarbon was placed in the reactor and heated up to reaction temperature. Subsequently, oxygen was passed through and catalyst was added. The content of peroxides bis(1-methyl-1-(2-(6-isopropylnaphthyl))ethyl) peroxide (1P) and bis(1-methyl-1-(4-(4’-isopropylbiphenyl))ethyl) peroxide (2P) was determined iodometrically according to the method described by Zawadiak *et al*. [21], and the contents of 2-(1-hydroperoxy-1-methylethyl)-6-(1-methylethyl) naphthalene (1MHP), 2-(1-hydroxy-1-methylethyl)-6-(1-methylethyl) naphthalene (1MH), 1-(6-(1-methylethyl) naphthalene-2-yl)ethanone (1MAc), 2,6-bis(1-hydroxy-1-methylethyl) naphthalene (1DH), 4-(1-hydroperoxy-1-methylethyl)-4’-(1-methylethyl)biphenyl (2MHP), 4-(1-hydroxy-1-methylethyl)-4’-(1-methylethyl)biphenyl (2MH), 1-(4’-(1-methylethyl)biphenyl-4-yl)ethanone (2MAc), 2,6-bis(1-hydroxy-1-methylethyl) biphenyl (2DH) were determined by HPLC according to previously elaborated methods [19,20].

2.2. Peroxides separation

The peroxides 1P and 2P were isolated by crystallization of 5 g of oxidation product with a corresponding amount of ethanol. The contents of the peroxides were determined iodometrically [21]. Obtained results are collected in Table 2.

Peroxides 1P and 2P were also purified by column chromatography (Kieselgel 60 / Merck 0.063 ÷ 0.200 mm). During purification of peroxide 1P, by column chromatography, traces of bisperoxide 2,6-bis(1-methyl-1-(1-methyl-1-(6-isopropylnaphthyl-2-yl)ethyl) ethyldioxy) naphthalene were isolated. ^1H and ^{13}C NMR analyses of purified peroxides were done.

***Bis(1-methyl-1-(2-(6-isopropylnaphthyl))ethyl) peroxide* 1P**

5 g of DIPN oxidation product (80°C, 10 h, Cu(II) 6.7 mmol mol^{-1} DIPN, TBAB 0.27 mmol mol^{-1} DIPN) containing 25% of 1P (1.25 g; 2.75 mmol) and 19 cm^3 (15 g) of EtOH were quickly boiled. The hot mixture was decanted from green precipitate of catalyst and cooled. Precipitated product was filtered off and dried. Crude 1P (0.58 g) with purity of 80% (0.46g, 1.01 mmol) was

obtained (crystallization yield 37%). Crude 1P (0.18 g) was purified by column chromatography (hexane/acetone 15/1 (v/v), 70 cm^3 of silica gel) giving 0.11 g of still contaminated peroxide which was purified again by column chromatography (hexane/acetone 25/1 (v/v), 12 cm^3 of silica gel). 0.037g of pure 1P and about 0.005 g of bisperoxide was obtained (the procedure was repeated to obtain bisperoxide for analysis).

1P: Mp 123,5-126°C

^{1}H NMR (300MHz, $CDCl_3$) δ_H ppm: 7.73-7.78 (m, 6H, H_{Ar}), 7.55-7.62 (m, 4H, H_{Ar}), 7.37 (dd, 2H, H_{Ar}, *J*=8.7, 1.8), 3.06 (m, 2H, $C(CH_3)_2\underline{H}$), 1.62 (s, 12H, $C(C\underline{H}_3)_2OO$), 1.33 (d, 12H, $C(C\underline{H}_3)_2H$, *J*=7.2).

^{13}C NMR (75MHz, $CDCl_3$) δ_C ppm: 146.1, 143.0, 132.6, 131.7, 128.1, 127.0, 125.7, 124.7, 123.7, 123.7 (Ar), 81.6 ($\underline{C}CH_3)_2OO$), 34.2 ($\underline{C}CH_3)_2H$), 26.9 ($C\underline{C}H_3)_2OO$), 24.0 ($C\underline{C}H_3)_2H$).

2,6-Bis(1-methyl-1-(1-methyl-1-(6-isopropylnaphthyl-2-yl)ethyl)ethyldioxy) naphthalene

Mp 149-152°C

^{1}H NMR (300MHz, $CDCl_3$) δ_H ppm: 7.74-7.82 (m, 10H, H_{Ar}), 7.57-7.63 (m, 6H, H_{Ar}), 7.38 (dd, 2H, H_{Ar}, *J*=8.7, 1.8), 3.06 (m, 2H, $C(CH_3)_2\underline{H}$), 1.63 (s, 24H, $C(C\underline{H}_3)_2OO$), 1.33 (d, 12H, $C(C\underline{H}_3)_2H$, *J*=7.2).

^{13}C NMR (75MHz, $CDCl_3$) δ_C ppm: 146.1, 143.0, 132.6, 131.7, 128.1, 127.0, 125.7, 124.7, 123.7, 123.7 (Ar), 81.6 ($\underline{C}CH_3)_2OO$), 34.2 ($\underline{C}CH_3)_2H$), 26.9 ($C\underline{C}H_3)_2OO$), 24.0 ($C\underline{C}H_3)_2H$).

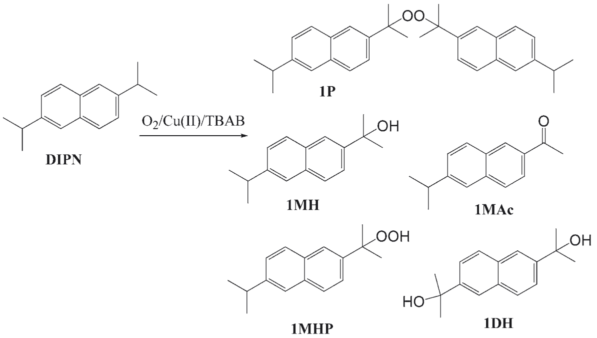
***Bis(1-methyl-1-(4-(4'-isopropylbiphenyl))ethyl) peroxide* 2P**

5 g of DIPB oxidation product (80°C, 10 h, Cu(II) 13.6 mmol mol^{-1} DIPB, TBAB 0.27 mmol mol^{-1} DIPB) containing 18.9% of 2P (0.95 g; 1.87 mmol) and 19 cm^3 (15 g) of EtOH were quickly boiled. The hot mixture was decanted from green precipitate of catalyst and cooled. Precipitated product was filtered off and dried. Crude 2P (1.44 g) with purity of 43% (0.62 g, 1.22 mmol) was obtained (crystallization yield 65%). Crude 2P (1.0 g) was recrystallized from EtOH (30 cm^3) giving product (0.33 g) with purity of 94% (yield 72%). Recrystallized 2P (0.28 g) was also purified by column chromatography (CH_2Cl_2/acetone 20/1 (v/v), 35 cm^3 of silica gel) giving 0.2 g of 2P.

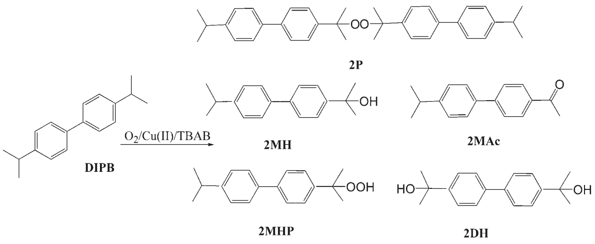
2P: Mp 124-131°C

^{1}H NMR (300MHz, $CDCl_3$) δ_H ppm: 7.20-7.47 (m, 16H, H_{Ar}), 2.80-2.97 (m, 2H, $C(CH_3)_2\underline{H}$), 1.54 (s, 12H, $C(C\underline{H}_3)_2OO$), 1.22 (d, 12H, $C(C\underline{H}_3)_2H$, *J*=6.9).

^{13}C NMR (75MHz, $CDCl_3$) δ_C ppm: 147.8, 144.8, 139.6, 138.5, 127.0, 126.8, 126.5, 126.0 (Ar), 81.6 ($\underline{C}CH_3)_2OO$), 33.8 ($\underline{C}CH_3)_2H$), 27.0 ($C\underline{C}H_3)_2OO$), 24.0 ($C\underline{C}H_3)_2H$).



Scheme 3. Products of DIPN oxidation in the presence of $CuCl_2$•$2H_2O$ and TBAB.



Scheme 4. Products of DIPB oxidation in the presence of $CuCl_2$•$2H_2O$ and TBAB.

3. Results and Discussion

The present paper reports the oxidation processes of diisopropylaromatic coumpounds such as DIPN and DIPB with oxygen in the presence of $CuCl_2$•$2H_2O$ and TBAB as catalyst at temperature range of 90 to 110°C.

The studied oxidation processes proceed according to a widely known free-radical chain mechanism, and hydroperoxides are the primary products. In our previously described non-catalytic oxidation processes of DIPN and DIPB, hydroperoxides were the main products; at 110°C, they were obtained with yields ranging from 40 to 60% and a selectivity of about 80% [8,10]. Application of catalyst, $CuCl_2$•$2H_2O$ or $CuCl_2$•$2H_2O$ with TBAB, yielded significantly different products. A decrease of hydroperoxide content and increasing amounts of other products were observed. The composition of obtained products is presented in Schemes 3 and 4, as well as in Table 1. Courses of typical catalytic oxidation processes of DIPN and DIPB are shown in Figs. 1 and 2.

It has been found that, in essence, only one isopropyl group in DIPN and DIPB undergoes catalytic oxidation. Alcohols 1DH and 2DH were the only products of oxidation of two groups detected, but their amount did not exceed 10% mol.

The composition of the obtained oxidation products of DIPN and DIPB, consisting mainly of peroxide, alcohol and ketone, is a result of, among others, the increase

Table 1. Oxidation of DIPN and DIPB in the presence of catalytic system $CuCl_2•2H_2O$/TBAB.

			DIPN [a]				
Temp. [°C]	**Cu(II) [mmol]**	**TBAB x10^2 [mmol]**	**1P**	**1MH**	**1MAc**	**1DH**	**1MHP**
					Yield [% mol]		
80	1.28	5.1	25.0	25.9	9.0	4.2	2.2
80	5.14	5.1	23.2	25.5	11.7	6.4	1.7
80	2.57	5.1	23.2	26.8	10.0	4.9	6.2
90 [c]	2.57	-	13.0	30.5	9.0	2.3	4.7
90	2.57	5.1	14.8	24.0	14.5	4.3	5.2
110	2.57	5.1	6.7	22.2	24.0	3.5	2.7

			DIPB [b]				
Temp. [°C]	**Cu(II) [mmol]**	**TBAB x10^2 [mmol]**	**2P**	**2MH**	**2MAc**	**2DH**	**2MHP**
					Yield [% mol]		
80	1.00	4.0	12.0	12.4	5.3	0.7	1.3
80	4.00	4.0	26.0	18.1	13.8	5.7	1.1
80	2.00	4.0	18.9	19.8	7.8	0.4	2.8
90 [c]	2.00	-	0.0	1.3	0.9	0.0	9.7
90	2.00	4.0	26.8	21.7	12.0	3.3	2.5
110	2.00	4.0	10.6	14.0	22.7	5.4	1.8

Oxygen - 10 $dm^3 h^{-1}$; stirrer – 1000 rpm; reaction time – 10h

[a] *DIPN – 0.190 mol (40g)*

[b] *DIPB – 0.147 mol (35g)*

[c] *4 mmol of 1,1'-azobis(cyclohexanecarbonitrile) was added*

Conversion of DIPN and DIPB can be estimated on sum of the products presented in Table 1, because any other possible products like 2,6-bis(1-hydroperoxy-1-methylethyl)naphthalene, 4,4'-bis(1-hydroperoxy-1-methylethyl)biphenyl), 2-(1-hydroperoxy-1-methylethyl)-6-(1-hydroxy-1-methylethyl)naphthalene or 4-(1-hydroperoxy-1-methylethyl)-4'-(1-hydroxy-1-methylethyl)biphenyl as well as carboxylic acids were not observed in amounts of exceeding 1%.

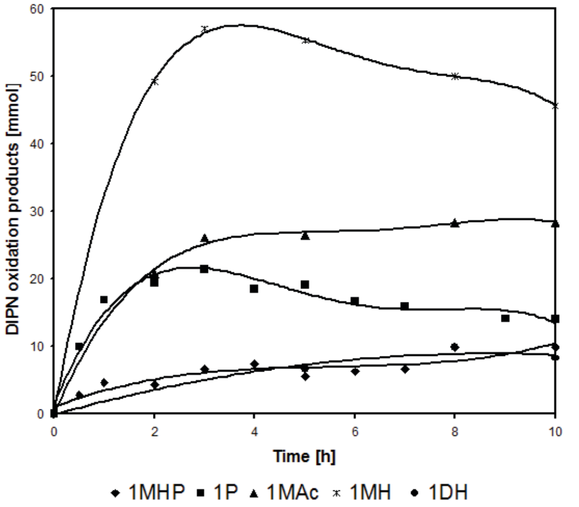


Figure 1. Course of oxidation process of DIPN in the presence of $CuCl_2•2H_2O$ and TBAB. (0.190 mol DIPN, 90°C, O_2 - 10 $dm^3 h^{-1}$; 1000 rpm.

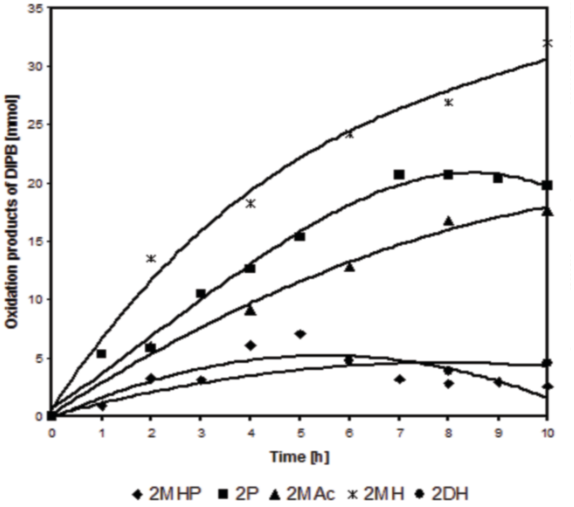
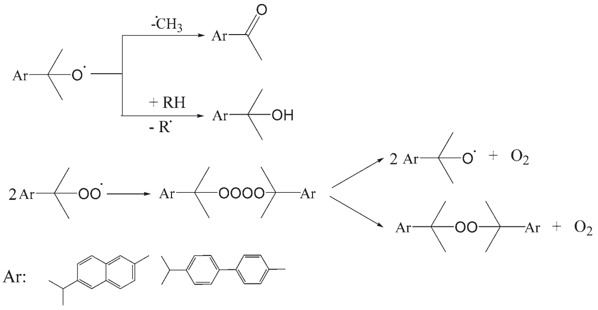


Figure 2. Course of oxidation process of DIPB in the presence of $CuCl_2•2H_2O$ and TBAB. (0.147 mol - DIPB, 90°C, O_2 - 10 $dm^3 h^{-1}$; 1000 rpm).

of hydroperoxide decomposition rate in the presence of both Cu(II) and TBAB. It is well known that copper(II) ions, similar to other transition metal ions, lower the activation energy of the hydroperoxide decomposition reaction [22]. The catalytic effect of quaternary ammonium salts on the hydroperoxide decomposition has also been described [23]. Free alkylperoxyl or alkoxyl radicals generated during the decomposition of hydroperoxides can initialize further reaction chains. Alkoxyl radicals undergo typical hydrogen abstraction or β-scission that generate alcohols and ketones. Peroxides can be formed by recombination of alkoxyl as well as peroxyl radicals generated during the oxidation process. Peroxyl radicals can recombine to tetraperoxides, which decompose with generation of two alkoxy radicals. Some of those radicals recombine to peroxide and some undergo aforementioned reactions, such as H-abstraction and β-scission (Scheme 5) [24].

It should also be noted that copper(II) chloride is slightly soluble in hydrocarbons and remains insoluble in the reaction mixture. Thus, TBAB probably acts also as a phase transfer catalyst of copper



Scheme 5. Ketone, alcohol and peroxide formation reactions from alkoxyl and peroxyl radicals.

ions, transmitting them in the form of complexes - $[Bu_4N]^+[CuCl_3]^-$ or $[Bu_4N]^+[CuCl_2]^-$ - to the organic phase [25]. Similar catalytic system consisting of $CoCl_2$ and didecyldimethylammonium bromide was recently described for methylbenzenes oxidation [26].

The results presented in Table 1 have shown that higher concentrations of mono- peroxides, alcohols and ketones are obtained in case of using $CuCl_2$•$2H_2O$ with TBAB than only $CuCl_2$•$2H_2O$. Under the same reaction conditions, higher concentrations of oxidation products are obtained in cases of DIPN than DIPB. The kinetic profiles of products formation are similar for DIPN and DIPB (Figs. 1 and 2). Monoalcohols and peroxides concentrations pass through the maximum (1MH and 1P after 3 h, 2P after 7 h and 2MH after 10 h). This suggests that subsequence reactions occurred, *e.g.* oxidation of 1MH leading to 1DH or decomposition of 1P to 1MAc or 1MH.

Depending on the applied process conditions, it was possible to obtain expected peroxides as well as mono-alcohols and ketones with yields of over 20% (Table 1) The temperature increase influenced the alcohol to ketone ratio. This is in agreement with literature data, which has demonstrated that higher temperatures favoured the β-scission reaction to ketones [27]. Thus, for higher yields of ketones, the process must be carried out at 110°C. On the other hand, higher yields of alcohols are obtained at 80°C. Due to lower stability of peroxides at higher temperature, smaller amounts were obtained at 110°C.

Obtained by DIPN and DIPB oxidation symmetrical peroxides 1P and 2P were separated from post-oxidation mixtures by crystallization with EtOH (hexane gave poorer results). Purities of isolated peroxides and yields of crystallization are given in Table 2.

Peroxide 1P separation from oxidation products of DIPN, with EtOH used in ratios of 1:1 and 1:2, did not produce positive results, as the whole mixture solidified. When higher amounts of EtOH were used, 1P was obtained with high purity, but with low yields. Purity of isolated 2P was rather low. However, the recrystallization produced peroxide with a purity of 94% and a yield of 72%.

The results have demonstrated that the oxidation of DIPN and DIPB in the presence of the $CuCl_2$•$2H_2O$ and TBAB catalyst can be potentially useful as a simple method for symmetrical peroxide synthesis directly from hydrocarbons.

4. Conclusions

It was found that oxidation of hydrocarbons containing two isopropyl groups, such as DIPN and DIPB, in the presence of copper(II) chloride and tetrabutylammonium bromide catalyst gave products containing mainly mono-peroxide, alcohol, ketone and only small amounts of hydroperoxide. It is a result of the high activity of catalyst in increasing the decomposition rate of hydroperoxides created in this process. The catalytic mechanism is complicated and includes catalytic influence of copper ions and TBAB on hydroperoxide decomposition; TBAB likely also transfers copper ions in the form of complexes to the organic phase.

The obtained products have different applications, e.g. peroxides as initiators or cross-linking agents, 1MAc as raw material for 6-hydroxy-2-naphthoic acid [28].

The process of isopropylaromatic oxidation catalyzed by Cu(II)/TBAB can be used as a simple method of symmetrical peroxides synthesis.

Table 2. Isolation of peroxides from oxidation products by crystallization with EtOH.

Product : EtOH w/w	**DIPN** [a]	**DIPB** [b]		
	1P [c] **[%]**	**Yield** [d] **[%]**	**2P** [c] **[%]**	**Yield** [d] **[%]**
1 : 1	-	-	38	52
1 : 3	80	37	43	65
1 : 6	97	12	45	63

[a] *5 g of DIPN oxidation products containing 25% of 1P*
[b] *5 g of DIPB oxidation products containing 18% of 2P*
[c] *purity of peroxide after crystallization*
[d] *yield of crystallization*

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