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N-Hydroxyphthalimide and transition metal salts as catalysts of the liquid-phase oxidation of 1-methoxy-4-(1-methylethyl)benzene with oxygen

Research Article

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Abstract: The oxidation process of 1-methoxy-4-(1-methylethyl)benzene catalysed by N-hydroxyphthalimide (NHPI) or NHPI in combination with Cu(II), Co(II), Mn(II) and Fe(II) salts was studied. The effects of the amount of catalyst and the temperature were determined. 1-Methyl-1-(4-methoxyphenyl)ethyl hydroperoxide was obtained in a yield of 73 mol% when 1-methoxy-4-(1-methylethyl)benzene was oxidised for 3 h at 60°C in acetonitrile as a solvent in the presence of NHPI. 1-(4-Methoxyphenyl)ethanone with high selectivity up to 68 - 75 mol%, but low yield amounting to 11 mol% was obtained when 1-methoxy-4-(1-methylethyl)benzene was oxidised in the presence of the NHPI/Cu(II) system at 120°C.

Keywords: *Catalytic oxidations • NHPI • 1-Methyl-1-(4-methoxyphenyl)ethyl hydroperoxide • 1-(4-Methoxyphenyl)ethanone*

1. Introduction

Oxidation of 1-methoxy-4-(1-methylethyl)benzene (**1**) with oxygen proceeds according to a widely known free-radical chain mechanism [1,2]. The obtained product contains 1-methyl-1-(4-methoxyphenyl)ethyl hydroperoxide (**2**) as well as 1-(4-methoxyphenyl) ethanone (**3**) and 2-(4-methoxyphenyl)-2-propanol (**4**) (Scheme 1) [3,4].

The oxidation products of **1** have many applications. 4-Methoxyphenol widely used as a polymerisation inhibitor, *e.g.* in paints and as dental adhesives [5], antioxidants [6] or depigmenting agents in cosmetics [7] can be obtained by acidic decomposition of hydroperoxide **2** analogous to phenol synthesis from cumene. Ketone **3** is used in pharmaceutical and polymer synthesis and as fragrance components or repellents [8].

The processes of **1** oxidation to hydroperoxide have been described in only a few papers [3,4,9,10]. The oxidisability of **1** was determined in the temperature range of 60-110°C and compared with that of cumene [3,4,9,10]. The maximum concentration of hydroperoxide **2** amounting to 40% was obtained when **1** was oxidised without a catalyst at 60°C for 18 h [4].

Recently, it has been demonstrated that the use of N-hydroxyphthalimide (NHPI) as a catalyst in isopropylaromatic hydrocarbon oxidation processes significantly increases the hydrocarbon conversion and hydroperoxide yield [11-16]. The catalytic activity of NHPI results from phthalimido-N-oxyl radicals (PINO) formation in the propagation step of the oxidation process (Scheme 2). It has been observed experimentally that PINO radicals abstract hydrogen atoms from hydrocarbons much faster than peroxyl radicals. Moreover, PINO radicals terminate much more slowly than peroxyl radicals [17-21].

When cumene [22], 2,6-diisopropylnaphthalene [23] or 1,3,5-triisopropylbenzene [24] were oxidised in the presence of NHPI (10 mol%) at 75°C using acetonitrile as the solvent, the respective hydroperoxides were obtained in yields of approximately >70% with selectivities >90%. The reported data on alkoxy derivatives of cumene oxidation have been controversial. The oxidation of **1** in the presence of NHPI, mentioned in literature, gave hydroperoxide **2** in a low yield of 15 mol% [25]. On the other hand oxidation of an analogous compound 4-propyloxycumene gave respective hydroperoxide in a good yield of 80% [26].

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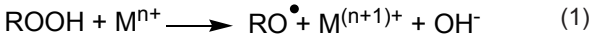
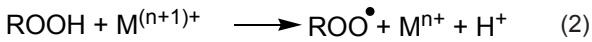
The composition of isopropylaromatic hydrocarbon oxidation products obtained in the presence of a catalyst composed of NHPI and transition metal salts is different: instead of hydroperoxides as main products, the respective alcohols and ketones are obtained [13,27,28]. This is a result of the catalytic effect of metal salts on hydroperoxide decomposition (Eqs. 1, 2).



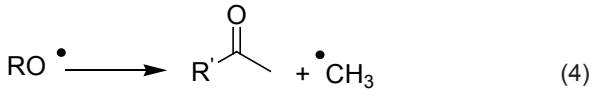
(1)



(2)

The alkoxyl radicals formed follow typical reactions: hydrogen abstraction or β-scission leading to alcohols or ketones (Eqs. 3, 4).

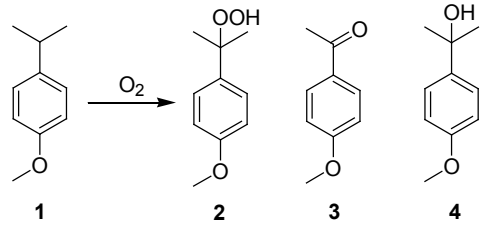
(3)



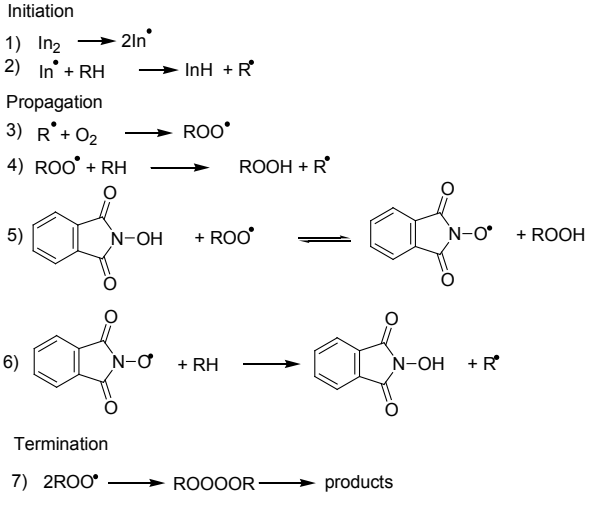
(4)

The catalyst composed of NHPI and Co(II) salts has been predominantly used. The product composition obtained depends on the process conditions. If 2,6-diisopropylnaphthalene and 4,4'-diisopropylbiphenyl were oxidised in the presence of NHPI and a Co(II) salt at 40°C using acetonitrile as the solvent, dialkohols 2,6-di(1-hydroxy-1-methylethyl) naphthalene and 4,4'-di(1-hydroxy-1-methylethyl) biphenyl, in yields of approximately 90%, were obtained [13,27]. When the same catalyst was used for the oxidation of 2,6-diisopropylnaphthalene at 75°C for 15 hours, 2-acetyl-6-isopropylonaphthalene was obtained in a yield of 31% [28].

Recently, it has been indicated that the type of metal salts used in combination with NHPI in the cumene oxidation process affects the hydrocarbon conversion and product composition [29]. The molar ratio of ketone to alcohol in the oxidation products was higher in the presence of Cu(II) salts than in the presence of Co(II) salts. The studies have showed that the catalytic system composed of NHPI and Cu(II) salts, rarely used previously, could be applied in the direct synthesis of ketones from isopropylaromatic hydrocarbons.



Scheme 1. Products of 1-methoxy-4-(1-methylethyl)benzene oxidation with oxygen



Scheme 2. Mechanism of hydrocarbon RH oxidation catalysed by NHPI

2. Experimental Procedure

2.1. Materials

1-Methoxy-4-(1-methylethyl)benzene (98+% Lancaster Synthesis) was purified by distillation over sodium. NHPI was purchased from Sigma-Aldrich. Metal salts and solvents were commercially available and used without further purification.

2.2. Oxidation of 1-methoxy-4-(1-methylethyl) benzene in gasometric apparatus

The oxidations were carried out in a gasometric apparatus presented in paper [30]. 1-Methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), catalyst and initiator were placed in the thermostated 4 mL quartz flask connected to a thermostated burette. Oxygen was supplied into the flask from a burette, in which the pressure was kept constant (760 mm Hg). The reaction was monitored by measuring the oxygen uptake. The amount of oxygen consumed was used to calculate conversion of **1**. In the products the content of hydroperoxide **2** was determined iodometrically [31], and the content of ketone **3** was determined by HPLC [32].

2.3. Oxidation of 1-methoxy-4-(1-methylethyl) benzene in a bubbler reactor

The oxidation was carried out in a 20 mL glass reactor supplied with a bubbler, thermometer, reflux condenser and heating jacket. 1-Methoxy-4-(1-methylethyl) benzene (5 mL) and acetonitrile (15 mL) were placed in the reactor and heated to the reaction temperature. After

that, NHPI (3 mmol) and AIBN (1 mmol) were added, and the oxygen was passed through. During the process, samples were taken, and the content of hydroperoxide **2** was determined iodometrically [31].

2.4. Analytical method

A Waters Alliance 2690 HPLC equipped with an autosampler and UV detector (Waters photodiode array) was used. A column Nova-Pak Silica 60 Å 4 µm (150×3.9 mm; Waters) was applied with a mixture of hexane and 2-propanol 99:1 v/v as a mobile phase [32]. Ketone **3** was detected at 260 nm using an external standard.

3. Results and Discussion

In this study, the oxidation of 1-methoxy-4-(1-methylethyl) benzene **1** catalysed by NHPI or NHPI in combination with transition metal salts with oxygen was examined. The purpose of this investigation was to determine the influence of the aforementioned catalysts on the product composition to evaluate the feasibility of synthesising valuable products: hydroperoxide **2** or ketone **3**.

The effect of the catalyst and temperature was investigated using benzonitrile as a solvent. A polar solvent is needed because NHPI is insoluble in hydrocarbons, and benzonitrile was chosen because of its low vapour pressure at the process conditions required for use of the gasometric apparatus.

3.1. 1-Methoxy-4-(1-methylethyl)benzene oxidation catalysed by NHPI

3.1.1. Effect of NHPI

Based on the literature that demonstrated that hydroperoxide yield increases in oxidation processes catalysed by NHPI, the study of the catalytic effect of NHPI on **1** oxidation at 60°C was carried out. The obtained results showed that the addition of NHPI in amounts of 1 to 10 mol% significantly increased the yield of hydroperoxide **2** in comparison with the non-catalytic process (Table 1, Fig. 1).

The observed increase of reaction rate is a result of a known higher propagation rate and lower termination rate obtained in the presence of NHPI. The increase of selectivity can be an effect of the more selective hydrogen abstraction by PINO radicals than by peroxyl radicals and less by-product formation in the termination step [15,33].

The courses of reactions presented in Fig. 1 showed that the rate of the catalysed reactions was high only at the beginning of the process. Next, the rate steadily decreased, and then the process was completely stopped. This effect could be a result of the formation of products that retard chain oxidation. Such observations were in a good agreement with our previous studies [3] on long-term, non-catalytic oxidation of **1** that revealed no autocatalytic effects. This fact can be taken to prove that the decomposition of hydroperoxide **2** yields compounds that inhibit the free-radical oxidation process. It is known that phenols can be formed by acid rearrangement of hydroperoxides and that acids (*e.g.* formic acid) can be obtained by oxidation of methyl radicals formed during decomposition of hydroperoxides [2]. Even traces of phenolic compounds can retard the oxidation.

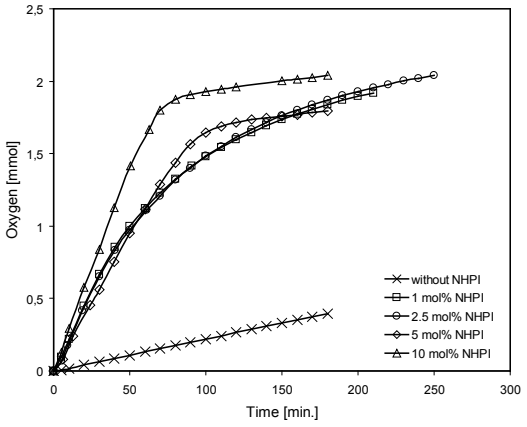


Figure 1. Effect of NHPI on 1-methoxy-4-(1-methylethyl)benzene oxidation at 60°C. Reaction conditions: 1-methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), 2,2'-azobis(2-methylpropionitrile) (0.62 mmol).

Table 1. Effect of NHPI on 1-methoxy-4-(1-methylethyl)benzene oxidation at 60°C

NHPI [mol%]	Time [min.]	Conversion [mol%]	Hydroperoxide Selectivity [mol%]
0	180	11	87
1.0	180	54	96
2.5	180	55	93
5.0	180	54	100
10.0	180	60	97

1-methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), 2,2'-azobis(2-methylpropionitrile) (0.62 mmol)

3.1.2. Effect of temperature

Next, the influence of temperature on **1** oxidation in the presence of NHPI (5 mol%) was studied in the range of 50 to 100°C (Table 2, Fig. 2).

The highest conversion of **1** was achieved at 60°C. Further increase of the temperature led to faster process inhibition, which can be an effect of NHPI decomposition [14] as well as an increase of rate of inhibitor formation reactions. The negative influence of the temperature on hydrocarbon conversion was also observed in cumene oxidation in the presence of NHPI [29].

Table 2. Effect of temperature on 1-methoxy-4-(1-methylethyl)benzene oxidation catalysed by NHPI

Temperature [°C]	Initiator[a] [mmol]	Time [min.]	Conversion [mol%]	Hydroperoxide Selectivity [mol%]
50	2.59	90	44	100
60	0.62	90	47	100
60	0.62	180	54	100
70	0.15	72	36	99
80	0.04	90	19	100
90	0.23	74	24	97
100	0.06	35	20	94

1-methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), NHPI (5 mol%, 0.17 mmol);
[a]Different amounts and types of initiator were added in order to obtain the same rate of initiation at different temperatures, amounting to 3.5×10^{-6} mol dm^{-3} s^{-1} (calculated based on data presented in [34,35]); 2,2'-azobis(2-methylpropionitrile) was used at 50 to 80°C and 1,1'-azobis(cyclohexanecarbonitrile) at 90 and 100°C

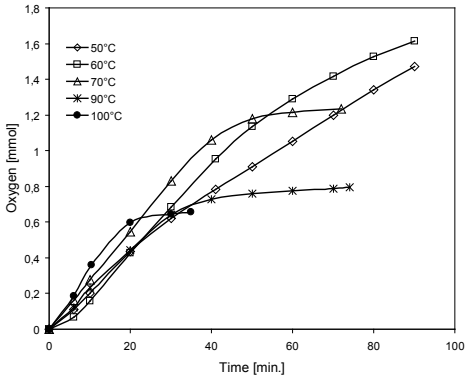


Figure 2. Effect of temperature on 1-methoxy-4-(1-methylethyl) benzene oxidation catalysed by NHPI. Reaction conditions: 1-methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), NHPI (5 mol%, 0.17 mmol), 2,2'-azobis(2-methylpropionitrile) was used at 50 to 80°C and 1,1'-azobis(cyclohexanecarbonitrile) at 90 to 100°C; initiation rate 3.5×10^{-6} mol dm^{-3} s^{-1}.

3.1.3. Oxidation in a bubbler reactor

The process of **1** oxidation was also carried out in a 20 mL bubbler reactor. At 60°C, using 10 mol% of NHPI and acetonitrile as a solvent, after 3 h hydroperoxide **2** was obtained in a yield of 73 mol%. The yield of hydroperoxide **2** obtained was significantly higher in comparison to the processes described in the literature [4,25].

3.2. 1-Methoxy-4-(1-methylethyl)benzene oxidation catalysed by NHPI in combination with Cu(II), Co(II), Mn(II) or Fe(II) salts

3.2.1. Effect of metal salt

In order to determine the possibility of the direct synthesis of ketone **3** from isopropylaromatic **1**, studies of the influence of catalysts composed of NHPI and Cu(II), Co(II), Mn(II), Fe(II) acetylacetonate or Cu(II) chloride on the oxidation process of **1** were examined (Table 3).

It was found that the presence of metal salts decreased the selectivity of hydroperoxide as a result of its decomposition to ketone **3** and alcohol **4**. The oxidation product of **1** also contained 1-methoxy-4-(1-methylethenyl)benzene as a dehydrating product of alcohol **4**.

Among the various metal salts, Fe(II) salts exhibited the lowest activity in catalysing the hydroperoxide decomposition reaction. The highest expected selectivity of ketone **3** was achieved in the presence of NHPI in combination with Cu(II) salts. The different behaviours of the metal salts may be a result of their different redox potentials and, in association with this, their different behaviours in catalysing hydroperoxide decomposition [2,36].

Based on these results, the catalytic system of NHPI/Cu(II) was chosen for further studies of the influence of the temperature increase and catalyst amount on ketone **3** selectivity.

3.2.2. Effect of temperature

According to literature data, temperature favours the β–scission reaction of alkoxyl radicals leading to ketones more than the hydrogen abstraction reaction leading to alcohols [13]. Hence, we studied the influence of increasing the temperature from 70 to 120°C on the oxidation process of **1** catalysed by NHPI/Cu(II) (Table 3). It can be seen that the temperature increase led to the expected increase of ketone **3** selectivity up to 68 - 75 mol%, but the conversion of **1** was simultaneously decreased. Therefore, the obtained yield of ketone **3** was only about 11 mol% when **1** was oxidized at 120°C in the presence of NHPI/$Cu(acac)_2$ system. The observed effect of decrease of conversion at higher temperature was probably a result of NHPI decomposition to non-active products and faster inhibitor formation at higher temperature. When ketone is obtained by β-scission of alkoxy radicals, methyl radicals are formed and can be oxidized to acids. Therefore, ketone formation can be connected to acid rearrangement of hydroperoxide **2** to inhibitor - 4-methoxyphenol.

Table 3. Effect of NHPI/metal salt systems on 1-methoxy-4-(1-methylethyl)benzene oxidation

Catalyst	Initiator[a] [mmol]	Time [min.]	Temperature [°C]	Conversion [mol%]	Selectivity [mol%]	
					Hydroperoxide	Ketone
NHPI/$Co(acac)_2$	0.61	50	70	28	31	19
NHPI/$Mn(acac)_2$	0.61	75	70	27	61	30
NHPI/$Fe(acac)_3$	0.61	115	70	38	77	3
NHPI/$Cu(acac)_2$	0.61	64	70	35	57	10
NHPI/$CuCl_2 \cdot 2H_2O$	0.61	64	70	23	13	36
NHPI/$Co(acac)_2$	0.24	60	100	21	22	23
NHPI/$Mn(acac)_2$	0.24	45	100	34	21	32
NHPI/$Fe(acac)_3$	0.24	45	100	16	53	32
NHPI/$Cu(acac)_2$	0.24	58	100	29	8	45
NHPI/$CuCl_2 \cdot 2H_2O$	0.24	45	100	18	10	57
NHPI/$Cu(acac)_2$	0.069	45	110	20	16	49
NHPI/$Cu(acac)_2$	0.022	45	120	16	22	68
NHPI/$CuCl_2 \cdot 2H_2O$	0.022	45	120	10	1	65

1-methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), NHPI (5 mol%, 0.17 mmol), metal salt (1 mol%, 0.034 mmol); [a]Different amounts and types of initiator were added in order to obtain the same rate of initiation at different temperatures, amounting to 13.6×10^{-6} mol dm^{-3} s^{-1} (calculated based on data presented in [34,35]); 2,2'-azobis(2-methylpropionitrile) was used at 70°C and 1,1'-azobis(cyclohexanecarbonitrile) at 100 to 120°C

Table 4. Effect of $Cu(acac)_2$ amounts on 1-methoxy-4-(1-methylethyl)benzene oxidation catalysed by NHPI

$Cu(acac)_2$ [%mol]	Time [min.]	Conversion [mol%]	Selectivity [mol%]	
			Hydroperoxide	Ketone
0.1	55	27	12	46
1.0	58	29	8	45
10.0	40	16	19	19

1-methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), NHPI (5 mol%, 0.17 mmol), 1,1'-azobis (cyclohexane carbonitrile) (0.24 mmol); 100°C

Table 5. Effect of NHPI amounts on 1-methoxy-4-(1-methylethyl)benzene oxidation catalysed by NHPI/Cu(II) system

NHPI [mol%]	Temp. [°C]	Time [min.]	Conversion [mol%]	Selectivity [mol%]	
				Hydroperoxide	Ketone
			$Cu(acac)_2$		
0	70	140	31	16	34
5	70	64	35	57	10
10	70	90	55	59	7
15	70	90	57	46	9
0	120	45	5	1	40
5	120	45	16	22	68
10	120	45	18	5	53
			$CuCl_2 \cdot 2H_2O$		
0	70	58	10	20	39
5	70	64	23	13	36
10	70	90	33	10	33
0	120	45	14	1	59
5	120	45	10	1	65
10	120	45	12	1	75

1-methoxy-4-(1-methylethyl)benzene (3.4 mmol, 0.56 mL), benzonitrile (1.44 mL), 0.034 mmol (1 mol%) metal salt. Different amounts and types of initiator were added in order to obtain the same rate of initiation at different temperatures, amounting to $13.6 \cdot 10^{-6}$ mol dm^{-3} s^{-1} (calculated based on data presented in [34,35]); 2,2'-azobis(2-methylpropionitrile) (0.61 mmol) was used at 70°C and 1,1'-azobis(cyclohexanecarbonitrile) (0.022 mmol) at 120°C

3.2.3. Effect of amount of NHPI and $Cu(acac)_2$

The conversion of **1** was decreased when the amount of $Cu(acac)_2$ was increased to 10 mol% (Table 4).

This phenomenon most likely resulted from the reaction of copper ions with peroxyl radicals that could lead to chain termination (Eq. 5) [2].

$$ROO^{\bullet} + Cu^{I} \longrightarrow ROOCu^{II} \quad (5)$$

The influence of the amount of NHPI was determined for NHPI/$Cu(acac)_2$ and NHPI/$CuCl_2$ at temperatures of 70 and 120°C (Table 5).

It was found that at 70°C, an increase in NHPI led to an increase in conversion with a simultaneous decrease in ketone selectivity. It is probably an effect of the increase of alcohol formation in the reaction of NHPI with alkoxyl radicals [14]. At 120°C, the increase of the NHPI concentration enhanced the ketone **3** yield. It suggests that the participation of a mentioned reaction of NHPI with alkoxyl radicals is lower at higher temperature probably as a result of NHPI decomposition.

4. Conclusions

It has been demonstrated that NHPI was an effective catalyst in the oxidation process of 1-methoxy-4-(1-methylethyl)benzene to hydroperoxide. Hydroperoxide **2** can be obtained with yield of 73 mol% when **1** is oxidised for 3 h at 60ºC in acetonitrile as a solvent in the presence of NHPI (10 mol%). Higher yield and reaction time reduction were achieved in comparison to a process described in literature [4,25]. The oxidation process of **1** to hydroperoxide can have an application as a step in 4-methoxyphenol synthesis by a method analogous to phenol synthesis from cumene.

It has been established that the highest selectivity of ketone **3** amounted to about 65-75 mol% was obtained by oxidation of 1-methoxy-4-(1-methylethyl)benzene in the presence of a catalyst composed of NHPI and Cu(II). However direct synthesis of ketone **3** by 1-methoxy-4-(1-methylethyl)benzene oxidation catalysed by the NHPI/Cu(II) system can be limited by the low conversion of **1**. On the other hand both unreacted hydrocarbon **1** as well as by-product 4-isopropenylanisole (product of alcohol **4** dehydration) can be recycled in the oxidation step after hydrogenation.

Acknowledgements

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