



SYNTHESIS OF SUBSTITUTED 2,2'- AND 4,4'-BIPHENYLDIOLS USING OXIDATIVE COUPLING REACTION

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Abstract. Based on the oxidative coupling reaction, methods for the synthesis of substituted 2,2'- and 4,4'-biphenyldiols have been developed. These biphenyldiols were not described in the literature. The article presents the potential applications and limitations of the method using potassium ferricyanide and iron (III) chloride hexahydrate.

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Introduction

Bisphenols are widely used as monomers for polyimides, polyamides, polyesters, polycarbonates, epoxy resins, and coatings [1, 2]. Therefore, the development of methods for the new bisphenols synthesis is an urgent task.

One of the economically feasible ways to produce bisphenols is the oxidative dimerisation reaction, which has been known to experts for a long time. Various variants of bisphenol synthesis using sodium hypochlorite [3], di-*tert*-butyl peroxide [4], hydrogen peroxide in the presence of peroxidase [5], oxygen in the presence of cobalt (II) tetraphenylporphyrin or its derivatives [6], copper complexes with amines [7], etc. have been described in the literature. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{K}_3[\text{Fe}(\text{CN})_6]$ are also known to be efficient, cheap, and widely used reagents for the oxidative dimerisation of phenols [8-10]. The use of these one-electron oxidants often provides high yields of target products, and may be suitable for large tonnage synthesis [11, 12].

Main body

In order to obtain known, but difficult to obtain and new bisphenols not described in the literature, we propose two variants of conducting the oxidative coupling reaction of phenols using potassium ferricyanide $\text{K}_3[\text{Fe}(\text{CN})_6]$ and iron (III) chloride hexahydrate (III) $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.



We used both variants phenols **1a-g** as substrates in containing substituents in positions 2 and 4 of the benzene ring. These substituents prevent the oxidative coupling reaction from proceeding, and the only available reaction centre was actually a carbon atom in position 6 of the benzene ring, which is in accordance with the results published in [14]. We conducted the reaction at room temperature according to the scheme shown in Fig. 1.

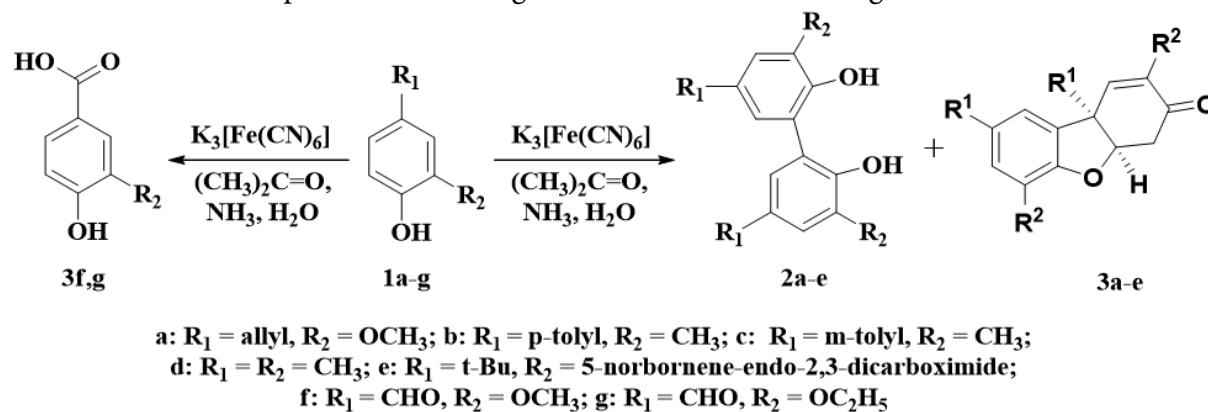


Fig. 1. The reaction scheme of 2,4-disubstituted phenols oxidative coupling under the action of potassium ferricyanide in alkaline medium

Due to the low water solubility of the initial phenols and target products, we conducted the reaction in an acetone-water mixture with the presence of ammonia. Potassium ferricyanide was still soluble under these conditions, and showed maximum activity. Moreover, the addition of acetone to the reaction mixture promoted better solubility of initial substrates, and formed target compounds, as well as the course of the reaction in homophase medium without heating.

Therefore, taking into account all the above-mentioned limitations, we add the oxidant to the reaction mixture in small portions, waiting until the ferrocyanide $\text{K}_4[\text{Fe}(\text{CN})_6]$ was formed during the reaction. Indeed, on account of its low solubility in aqueous acetone, it did not precipitate out of the reaction mixture.

We monitored the progress of the reaction by TLC. At the end of the reaction the precipitate of potassium ferrocyanide and by-products was filtered off, the filtrate was acidified with hydrochloric acid to pH=1.

Unfortunately, a waxy mass of yellow colour was formed as a result of side reactions, which could not be fully identified. According to the results of our studies, in agreement with literature data, this waxy mass includes oxidation by-products, mainly dihydrodibenzofuranones **3a-e**, as well as insignificant amounts of the initial phenols **1a-e** [15, 16].

It was not possible to conduct the oxidative coupling of vanillin **1f** and ethylvanillin **1g** using potassium ferricyanide under the conditions above. The reason is that potassium ferricyanide in alkaline medium is able to oxidise aromatic aldehydes to the corresponding carboxylic acids **3f-g**, which makes it impossible to use it for the oxidative coupling of vanillin **1f** and ethylvanillin **1g** containing a formyl group [13].

The oxidative coupling of vanillin **1f** and ethylvanillin **1g** to the corresponding bisphenols **2f,g** was successfully performed by using iron (III) chloride hexahydrate $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ as oxidant (Fig. 2).

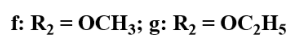
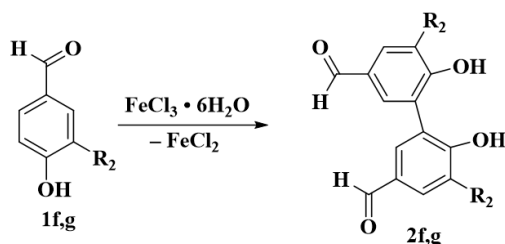


Fig. 2. Scheme of the oxidative coupling reaction of 2,4-disubstituted phenols under the action of iron (III) chloride in aqueous solution

The best results were obtained by heating the starting phenols **1f,g** in aqueous iron (III) chloride solution at 60–70 °C for several hours. When the reaction was completed, the product precipitated from the reaction medium was filtered off, washed with hot water to remove unreacted phenol, and then dissolved in an aqueous solution of alkali. Precipitated iron (II) and (III) hydroxides were filtered off and the filtrate was acidified with HCl to pH=1.

We also failed to conduct oxidative dimerisation of 2,6-disubstituted phenols **5a,b** in the presence of potassium ferricyanide. The formation of quinol esters **I** apparently occurred under the chosen conditions, which were further rapidly rearranged into the corresponding 4-hydroxydiphenyloxides **II** and further oxidation yielded the corresponding polyphenylene esters **III** [12] (Fig. 3).

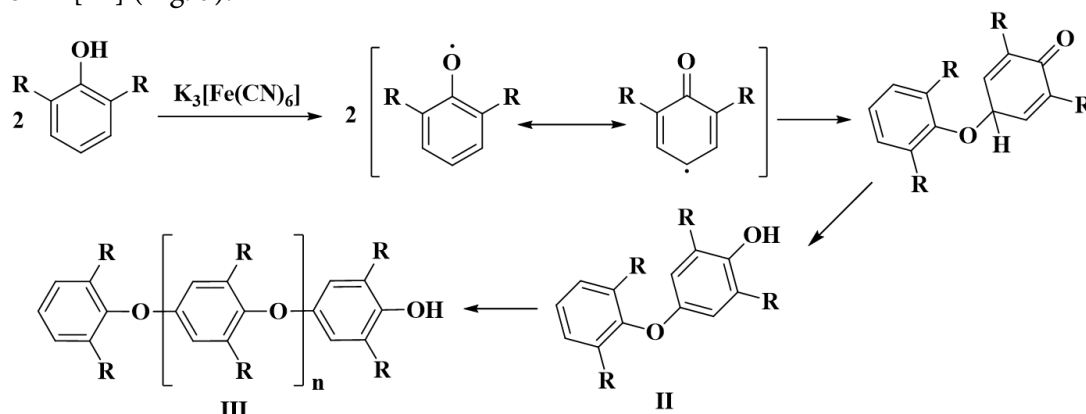


Fig. 3. Scheme of side reactions occurring during oxidative coupling of 2,6-disubstituted phenols in the presence of potassium ferricyanide

Therefore, iron (III) chloride hexahydrate $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was used as an oxidising agent for the synthesis of biphenyl-4,4'-diols **5a,b** (Fig. 4).

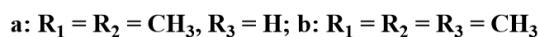
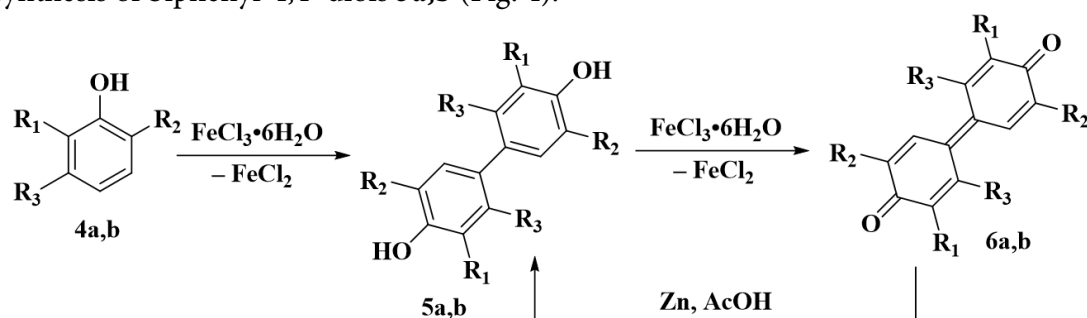


Fig. 4. Schematic of the oxidative coupling reaction of 2,6-disubstituted phenols



We found that conducting the oxidative coupling of 2,6-disubstituted phenols **4a,b** in the presence of a 3-fold excess of iron (III) chloride allows us to obtain significantly higher yields of side (in our case, intermediate) diphenoquinones **6a,b** than when using 0.5 mol $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ per 1 mol phenol, as proposed in [14]. The reason for this is that with excess oxidant, the bisphenols **5a,b** formed under the selected conditions were further oxidised and converted to the thermodynamically stable quinoid form **6a,b**. Subsequent reduction of the carbonyl function by treatment of the isolated diphenoquinones **6a,b** with zinc in acetic acid with high yield allows us to obtain the target substituted biphenyl-4,4'-diols **5a,b**.

The substituted 2,2'-biphenyldiols obtained are promising for the preparation of new heterocyclic systems. They were not described in the literature, and on the basis of substituted 4,4'-biphenyldiols it is possible to obtain polyetherimides with new properties.

Experimental part

IR spectra were recorded on a PerkinElmerRX-1 FTIR spectrometer at 700–4000 cm^{-1} (KBr).

^1H NMR spectra were recorded on a BrukerDRX-400 instrument for sample solutions in DMSO-d_6 at 30 °C. The signals of residual solvent protons in proton spectra ($\delta_{\text{H}}2.50$) were used as a reference for chemical shifts.

Phenols **1a-d,f,g** and oxidants are commercially available. Phenol **1e** was prepared by condensation of endic anhydride with commercially available 2-amino-4-*tert*-butylphenol according to the known procedure [17].

Oxidative dimerisation of 2,4-disubstituted phenols in the presence of $(\text{K}_3[\text{Fe}(\text{CN})_6]$ (general procedure). 0.012 mol of phenol, 15 ml of water, 35 ml of acetone and 45 ml of ammonia were added to a flask equipped with a stirrer. Equimolar amounts of potassium ferricyanide were added to the reaction mixture in portions over 40 minutes after dissolution under vigorous stirring. The reaction was conducted at room temperature for 15 hours. We monitored the progress of the reaction by TLC. The flask contents (potassium ferrocyanide and alkaline insoluble precipitates) were filtered off at the end of the reaction, and the filtrate was acidified to $\text{pH}=1$. The precipitate was filtered off, washed with excess water, and dried in air.

3,3'-dimethoxy-5,5'-di(prop-2-en-1-yl)diphenyl-2,2'-diol (2a): Light beige powder. Yield 50%, $T_{\text{melt}} = 145\text{--}147$ °C. IR (ν_{max} , KBr): 3273 (OH), 1637 (C=C), 1246 (OCH_3). Found, %: C, 73.63; H, 6.83. $\text{C}_{20}\text{H}_{22}\text{O}_4$. Calculated, %: C, 73.60; H, 6.79. ^1H NMR (400 MHz, DMSO-d_6) δ , ppm.: 3.26 (d, $J=6.59$ Hz, 4 H, C(5, 5') CH_2), 3.67 (s, 6 H, C(3, 3') OCH_3), 4.96-5.11 (m, 4 H, C(5, 5')= CH_2), 5.94 (ddt, $J=16.98, 10.01, 6.78, 6.78$ Hz, 2 H, C(5, 5')= CH), 6.57 (s, 2H, C(4, 4')H), 6.61-6.63 (m, 2 H, C(6, 6')H).

4,4''',5',5''-tetramethyl-1,1':3',1''':3'',1''''-quaterphenyl-4',6''-diol (2b): A beige powder with a greenish tinge. Yield is 74%. IR (ν_{max} , KBr): 3519 (OH), 1177 ($\text{C}_{\text{Ar}}\text{-OH}$). Found, %: C, 86.04; H, 6.83. $\text{C}_{28}\text{H}_{26}\text{O}_2$. Calculated, %: C, 85.25; H, 6.64. ^1H NMR (400 MHz, DMSO-d_6) δ , ppm.: 2.33 (s, 12 H, C(4, 5', 4''', 5''') CH_3), 7.37 (d, 4 H, $J=7.03$ Hz, C(3', 5', 3''', 5''')H), 7.49 – 7.62 (m, 8 H, C(2, 6, 2', 6', 2'', 4'', 2''', 6''')H), 8.02 (s, 2 H, OH).

3,3''',5'',5''''-tetramethyl-1,1':3'',1''':3''',1''''-quaterphenyl-4',6''-diol (2c): Green coloured oil. Yield is 20%. IR (ν_{max} , KBr): 3515 (OH), 1174 ($\text{C}_{\text{Ar}}\text{-OH}$). ^1H NMR (400 MHz, DMSO-d_6) δ , ppm.: 2.34 (s, 12 H, C(3, 5', 3''', 5''') CH_3), 7.15 (m, 2H, C(4, 4''')H), 7.31 (t, $J = 7.65$ Hz, 2 H,



C(5, 5'')H), 7.54 (m, 2 H, C(6, 6'')H), 7.59 (s, 2 H, C(2, 2'')H), 7.68 (s, 2 H, C(6', 4'')H), 7.72 (s, 2 H, C(2', 2'')H).

3,3',5,5'-tetramethylbiphenyl-2,2'-diol (2d): Beige powder. Yield is 83%. IR (ν_{\max} , KBr): 3280 (OH), 1175 (C_{Ar} -OH). Found, %: C, 79.39; H, 7.33. $C_{16}H_{18}O_2$. Calculated, %: C, 79.31; H, 7.49. 1H NMR (400 MHz, DMSO- d_6) δ , ppm.: 2.19 (s, 6 H, C(3, 3')CH₃), 2.21 (s, 6 H, C(5, 5')CH₃), 6.79 (s, 2 H, C(4, 4')H), 6.90 (s, 2 H, C(6, 6')H), 8.13 (s, 2 H, C(2, 2')OH).

2,2'-(5,5'-di-*tert*-butyl-2,2'-dihydroxybiphenyl-3,3'-diyl)bis(3a,4,7,7a-tetrahydro-1H-4,7-methanoisoindole-1,3-dione) (2e): A light beige powder. Yield is 28%. IR (ν_{\max} , KBr): 3472 (OH), 1709 (C=O имид), 1181 (C_{Ar} -OH). Found, %: C, 74.23; H, 5.53; N, 4.65. $C_{38}H_{40}N_2O_6$. Calculated, %: C, 73.53; H, 6.50; N, 4.51. NMR 1H (400 MHz, DMSO- d_6) δ , ppm.: 1.38 (br.s., 18 H, C(5,5')*t*-Bu), 1.60 (m, 4 H, C(4*, 7*)CH₂), 3.42 (m, 8 H, C(3*a, 4*)H), 6.20-6.30 (m, 4 H, C(5*, 6*)H), 6.95 (br.s., 2 H, C(1, 1')H), 7.73 (br.s., 2 H, C(6, 6')H), 8.24 (br.s., 2 H, C(6,9)H).

Oxidative dimerisation of 2,4-disubstituted phenols in the presence of iron (III) chloride hexahydrate $FeCl_3 \cdot 6H_2O$ (general procedure). 0.024 mol of phenol and 40 ml of water were added to a flask equipped with a stirrer and a reflux condenser; the reaction mixture was heated to 60–70 °C, and kept for 15–20 minutes until phenol was completely dissolved. 0.028 mol $FeCl_3 \cdot 6H_2O$ in solid form or as an aqueous solution was added to the reaction mixture after dissolution under vigorous stirring. The reaction mixture was intensively stirred at 90–100 °C for 2–4.5 hours depending on the nature of the initial phenol. We monitored the progress of the reaction by TLC. We cooled the contents of the flask to room temperature at the end of the reaction, and filtered the precipitate. The precipitate was washed with excess hot water to dissolve unreacted phenol, after which the product was dissolved in concentrated NaOH solution, filtered from iron hydroxides. The filtrate was carefully acidified with concentrated hydrochloric acid. We filtered the precipitate and washed with excess water (3×50 cm³). We air-dried the isolated target products **2f,g**.

6,6'-dihydroxy-5,5'-diethoxybiphenyl-3,3'-dicarbaldehyde(2f): Brown powder. Yield is 52%. $T_{\text{melt}} = >300$ °C. IR (ν_{\max} , KBr): 3349 (OH), 1680 (C=O), 1276, 1250, 1152 (C_{Ar} -OC₂H₅). Found, %: C, 66.03; H, 5.83. $C_{18}H_{18}O_6$. Calculated, %: C, 65.45; H, 5.49. NMR 1H (400 MHz, DMSO- d_6 , J/Hz) δ , ppm.: 1.39 (t, J = 7.10 Hz, 6 H, C(5, 5')CH₃), 4.10-4.33 (m, 4 H, C(5, 5')CH₂), 7.79 (s, 2 H, C(4, 4')H), 7.85 (s, 2 H, C(2, 2')H), 10.00 (s, 2 H, C(3, 3')CHO).

6,6'-dihydroxy-5,5'-dimethoxybiphenyl-3,3'-dicarbaldehyde(2g): Brown powder. Yield is 54%. $T_{\text{melt}} = >300$ °C. IR (ν_{\max} , KBr): 3236 (OH), 1670 (C=O), 1256, 1145 (C_{Ar} -OCH₃). Found, %: C, 63.89; H, 4.83. $C_{16}H_{14}O_6$. Calculated, %: C, 63.57; H, 4.67. 1H NMR (400 MHz, DMSO- d_6) δ , ppm.: 3.88 (s, 6 H, C(5, 5')OCH₃), 7.61 (s, 2 H, C(4, 4')H), 7.79 (s, 2 H, C(4, 4')H), 7.91 (s, 2 H, C(2, 2')H), 10.02 (s, 2 H, C(3, 3')CHO).

Oxidative dimerisation of 2,6-disubstituted phenols in the presence of iron (III) chloride hexahydrate $FeCl_3 \cdot 6H_2O$ (general procedure). We added 0.024 mol of finely ground phenol, 40 ml of water and heated the contents of the flask to 40–80 °C depending on the nature of the phenol in a flask equipped with a stirrer and a reflux condenser. We added 0.065 mol $FeCl_3 \cdot 6H_2O$ ($\omega = 16\%$) to the reaction mixture 10–15 min after partial dissolution of phenol, and continued stirring at 40–80 °C for 2.5 hours. We monitored the progress of the reaction by TLC.



We cooled the contents of the flask to room temperature at the end of the reaction, and filtered the precipitate. We air dried the diphenoquinones **6a,b** isolated in 77-84% yields.

To obtain the target products **5a,b**, we added a threefold excess of zinc dust to diphenoquinones suspended in hot acetic acid. We filtrated the reaction mass from zinc dust after one hour, poured the filtrate into 50 cm³ of water, and left it for the crystallisation. We filtered the precipitate and washed it with excess water (2×50 cm³). The target products obtained in yields of 26–39 % of theory were air dried.

3,3',5,5'-tetramethyl-[1,1'-biphenyl]-4,4'-diol (5a): Yield: 39%. IR (ν_{\max}) cm⁻¹: 3368 (OH), 1192 (Ar-OH). Found, %: C, 80.10; H, 8.33. C₁₆H₁₈O₂. Calculated, %: C, 79.31; H, 7.49. ¹H NMR (400 MHz, DMSO-d₆) δ , ppm.: 2.18 (s, 12 H, C(3, 5, 3', 5')CH₃), 7.39 (s, 4 H, C(2, 6, 2', 6')H).

2,2',3,3',5,5'-hexamethyl-[1,1'-biphenyl]-4,4'-diol (5b): Yield 26%. IR (ν_{\max}) cm⁻¹: 3516 (OH). Found, %: C, 78.90; H, 9.44. C₁₈H₂₂O₂. Calculated, %: C, 80.56; H, 7.51. ¹H NMR (400 MHz, DMSO-d₆) δ , ppm.: 1.94 (s, 6 H, C(2, 2')CH₃), 2.02-2.10 (m, 12 H, C(3, 5, 3', 5')CH₃), 7.12 (s, 2 H, C(6, 6')H).

3,3',5,5'-tetramethyl-1,1'-bi(cyclohexa-2,5-dien-1-ylidene)-4,4'-dione (6a): Yield 84%. IR (ν_{\max}) cm⁻¹: 1636 (C=O). Found, %: C, 78.98; H, 6.84. C₁₆H₁₆O₂. Calculated, %: C, 79.97; H, 6.71. ¹H NMR (400 MHz, DMSO-d₆) δ , ppm.: 1.90 (s, 12 H, C(3, 5, 3', 5')CH₃), 6.88 (s, 4 H, C(2, 2', 6, 6')H).

2,2',3,3',5,5'-hexamethyl-1,1'-bi(cyclohexa-2,5-dien-1-ylidene)-4,4'-dione (6b): Yield 77%. IR (ν_{\max}) cm⁻¹: 1633 (C=O). Found, %: C, 78.91; H, 6.98. C₁₈H₂₀O₂. Calculated, %: C, 80.56; H, 7.51. ¹H NMR (400 MHz, DMSO-d₆) δ , ppm.: 1.91 (s, 6 H, C(2,2')CH₃), 2.05-2.10 (m, 12 H, C(3, 5, 3', 5')CH₃), 6.78 (s, 2 H, C(6, 6')H).

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