Chemistry of Heterocyclic Compounds, Vol. 38, No. 10, 2002

HETEROCYCLES BASED ON STERICALLY HINDERED PHENOLS AND THEIR DERIVATIVES. (REVIEW)

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Published data on the synthesis of heterocyclic compounds (derivatives of dibenzofuran, phenoxazine, benzodioxolane, etc.) based on sterically hindered 6-substituted 2,4-di-tert-butylphenols, 6-hydroxy-2,4- and 6-hydroxy-2,5-di-tert-butylphenols, and their redox-conjugated ortho-benzoquinones are reviewed.

Keywords: benzodioxolane, benzoxazine, di-*tert*-butyl-*ortho*-benzoquinones, dibenzofuran, 6-substituted 2,4- and 2,5-di-*tert*-butylphenols, phenoxazine, heterocyclization.

The introduction of *tert*-butyl groups into the phenol molecule has a significant effect on the reactivity as a result of the redox potential and steric effects. The selectivity of the processes is increased, and transformations uncharacteristic of the unsubstituted analogs become possible. Thus, a tendency toward heterocyclization with the formation of derivatives of dibenzofuran, phenoxazine, and benzodioxazines is observed in the reactions of 2,4- and 2,5-di-*tert*-butylphenols containing additional substituents (Br, NH_2, OH, CH_2NR_2) at position 6. Specific examples of the transformations leading to heterocyclic compounds are summarized in the present review. It must be emphasized that investigation of the heterocyclization of substituted phenols is of undoubted practical interest, since it opens up new synthetic possibilities. Moreover, it provides essential information for establishing the mechanism of the activity of the phenols used as bioantioxidants, medicinals, and agents for plant protection.

1. TRANSFORMATIONS OF 2,4-DI-*tert*-BUTYLPHENOL WITH THE FORMATION OF DIBENZOFURANS

It was found that the transformations of 2,4-di-*tert*-butylphenol (**1**) resulting from bromination and oxidation in various sequences provide a convenient method for the production of dibenzofuran, hydroxy- and dihydroxydibenzofurans, and related derivatives. The "bromination–oxidation" method involves the synthesis of 6-bromo-2,4-di-*tert*-butylphenol (**2**) and its oxidation by manganese triacetate in protic media (AcOH, ROH) [1], according to the following scheme:

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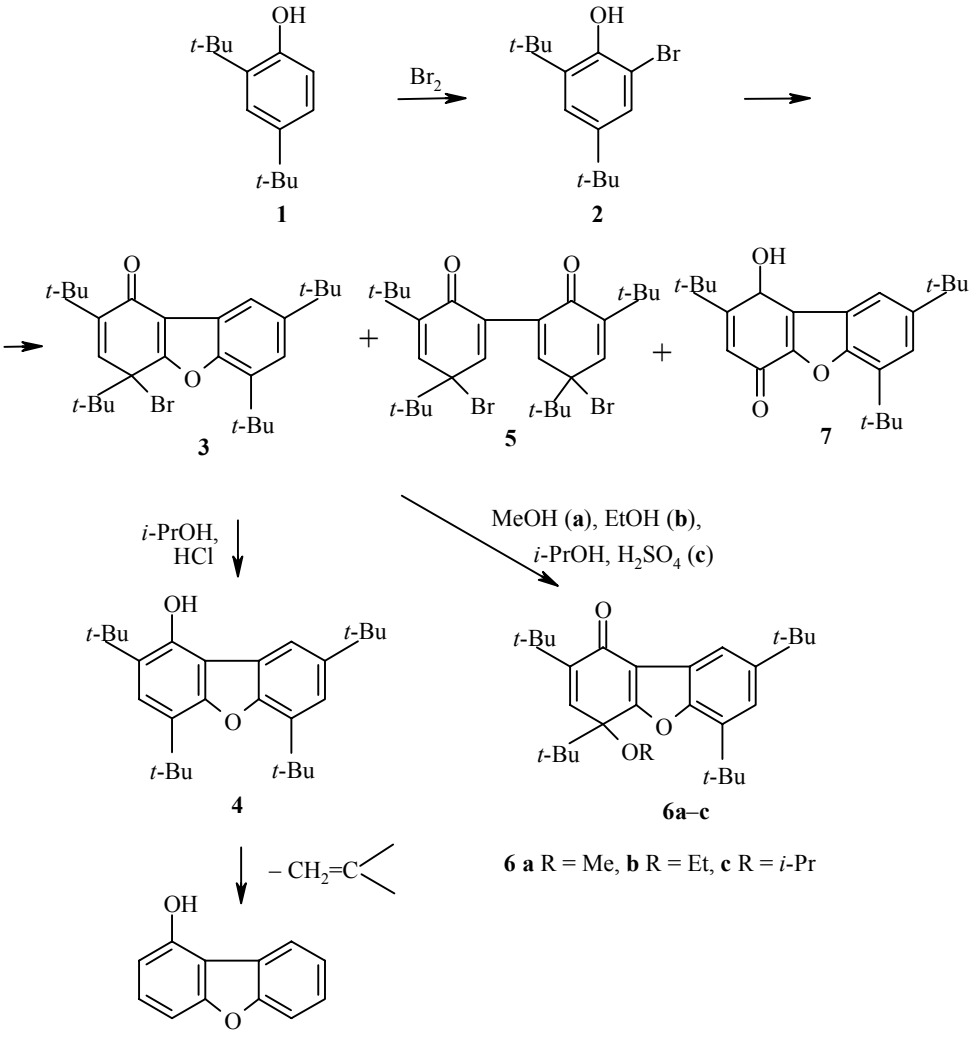
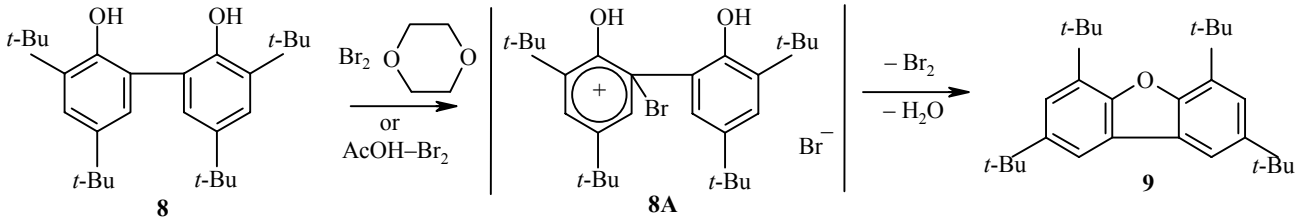


The composition of the oxidation products, the yield of the key quinobromide **3**, and the selectivity of the process depend on the nature of the solvent and the additives. In acetic acid the yield of the quinobromide **3** amounts to ~60%, and the parallel formation of the bisquinobromide **5** and quinone **7** is observed.

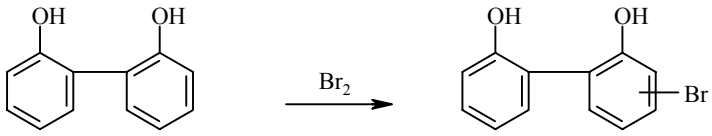
In methanol and ethanol, together with the quinobromide **3**, its solvolysis products the alkoxyquinolides **6a** and **6b** are formed. Oxidation is most selective in isopropyl alcohol, where a high yield of the quinobromide **3** is obtained and the process is not complicated by solvolysis. A further advantage of isopropyl alcohol is the possibility of its redox reaction with the quinobromide, leading to the hydroxydibenzofuran **4**. This makes it possible to convert the bromophenol **2** into hydroxydibenzofuran **4** without separating the stages: **2** → **3** → **4**. Reduction takes place more slowly in pure isopropyl alcohol but is greatly accelerated in the presence of HCl. The use of sulfuric acid as catalyst leads to a change in the reaction path, and the isopropoxyquinolide **6b** is formed instead of reduction to the hydroxydibenzofuran. The difference in the behavior of the quinobromide **3** in the *i*-PrOH–HCl and *i*-PrOH–H_2SO_4 systems is the appearance of dual reactivity within the limits of the two alternative types of nucleophilic substitution: Carbophilic and halogenophilic.

The reverse procedure, i.e., initial oxidation of the phenol **1** to the bisphenol **8** followed by bromination under various conditions, leads to the formation of tetra-*tert*-butyldibenzofuran **9** [2]. Heterocyclization under the influence of brominating agents takes place both in solutions (AcOH–Br_2) and in the solid phase (when the bisphenol **8** and dioxane dibromide are rubbed together) [3]. The process probably includes the formation and dehydrodehalogenation of an intermediate σ complex having the structure of the protonated *ortho*-quinobromide **8A**:



8 **8A** **9**

The mechanism of the participation of the halogen in this process can be called "phantom bromination" since the bromine is not introduced into the final reaction product (dibenzofuran). The adduct of the bisphenol with bromine acts as catalyst of cyclodehydration, the extreme ease of which is probably a specific characteristic of tetra-*tert*-butylbisphenol **8**. Under the same conditions the unsubstituted *ortho*-bisphenol **10** undergoes bromination, and the dibenzofuran is not detected:



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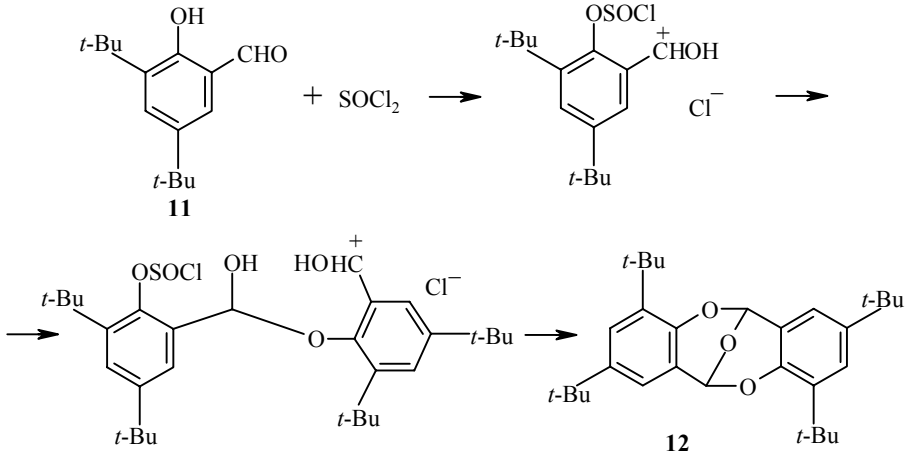
It is natural to suppose that the *tert*-butyl groups in tetra-*tert*-butylbisphenol provide positional protection of the rings against halogenation and also steric protection of the OH groups against participation in the formation of intermolecular hydrogen bonds concurrent with the intramolecular hydrogen bonds.

2. ANHYDRODIMERIZATION OF DI-*tert*-BUTYLSALICYLALDEHYDE WITH THE FORMATION OF DI(2,4-DI-*tert*-BUTYLBENZO)-2,6,9-TRIOXABICYCLO[3.3.1]NONA-3,7-DIENE

An interesting process of cyclodehydration presumably with the participation cationoid intermediates is observed in the reaction of di-*tert*-butylsalicylaldehyde **11** with thionyl chloride and phosphorus pentachloride [4]. Anhydridodimerization occurs both in a liquid weakly nucleophilic medium with a high concentration of the initial aldehyde (a solution in thionyl chloride, ratio of aldehyde and thionyl chloride 1:1.5) and in the solid phase with the aldehyde rubbed with a small excess of phosphorus pentachloride. Condensation takes place clearly as a stage process, beginning with the formation of the corresponding aryl ether. In the absence of an external competitor the displaced proton of the hydroxyl group is accepted by the neighboring carbonyl. The carbenium intermediate generated here reacts with a second molecule of the aldehyde, giving a semiacetal. The following stages, which duplicate preceding stages, complete the formation of the bicyclic system **12**:

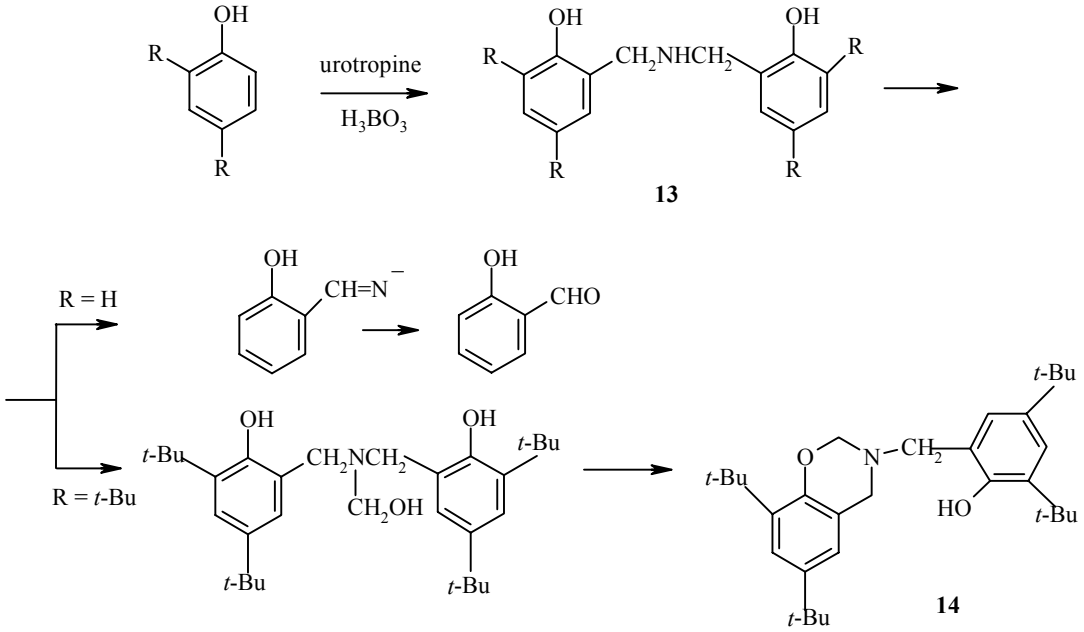


11 **12**

X-ray crystallographic analysis of the anhydrodimer supports unambiguously the structure of di(2,4-di-*tert*-butylbenzo)-2,6,9-trioxabicyclo[3.3.1]nona-3,7-diene (**12**).

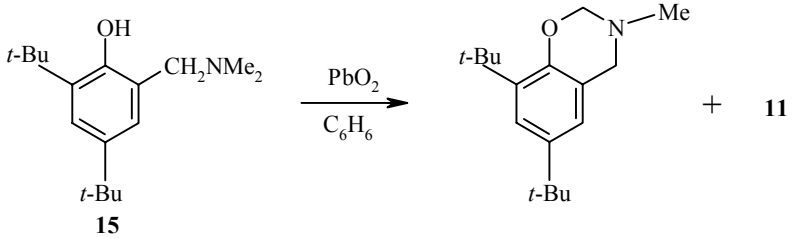
3. HETEROCYCLIZATION OF 2,4-DI-*tert*-BUTYL-6-DIALKYLAMINOMETHYLPHENOLS

A tendency for heterocyclization was detected during the development of methods for the synthesis of 2,4-di-*tert*-butyl-6-formylphenol through the 6-aminomethyl derivatives. One of the most suitable methods for the introduction of a formyl group at the *ortho* position of phenols is based on the Duff reaction – the reaction of phenol with urotropine in the presence of H_3BO_3 in ethylene glycol. The reaction includes the intermediate formation of dibenzylamine **13**, which is transformed into a Schiff base and then into the aldehyde (see the scheme). In the case of 2,4-di-*tert*-butylphenols, however, the reaction takes place differently and leads to the formation of a derivative of benzoxazine **14** [5]:

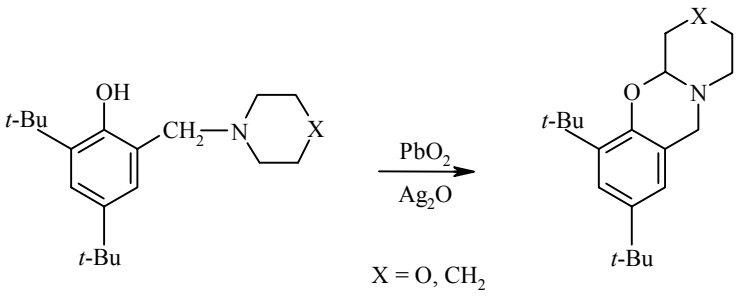


The direction of the transformation probably changes at the stage of the dibenzylamine **13**, which with R = CMe_3 is hydroxymethylated in stages at this nitrogen atom and condenses with the formation of the benzoxazine **14**.

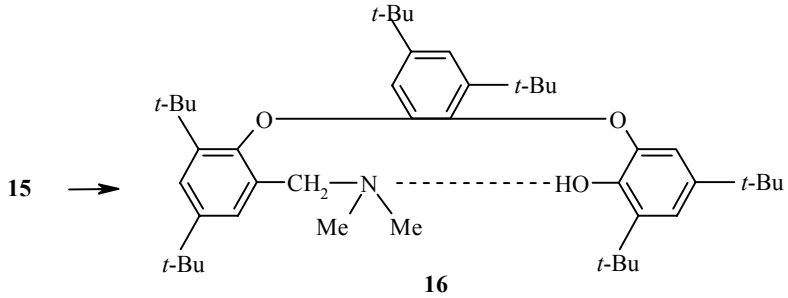
Related processes, leading to the formation of substituted benzoxazines, also proved typical of the redox transformations of 2,4-di-*tert*-butyl-6-dialkylaminomethylphenols [6]. Thus, dehydroheterocyclization prevails over the alternative benzyl oxidation of 2,4-di-*tert*-butyl-6-dimethylaminomethylphenol (**15**) with PbO_2:



The oxidative condensation of 2,4-di-*tert*-butyl-6-morpholylmethyl- and 6-piperidylmethylphenols takes place similarly (with the participation of the hydroxyl group and the α-carbon atom at the nitrogen atom):



The unusual quasiheterocyclic compound **16** is formed as a result of oxidative trimerization with partial fragmentation of compound **15** by the action of $Mn(OAc)_3$:



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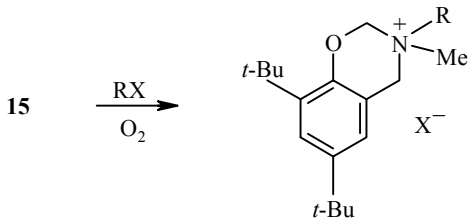
In this case, probably, a matrix effect is possible as a result of the coordination of Mn(III) and the initial aminomethylphenol as a bidentate ligand. As a result oxidative trimerization occurs with the loss of two dialkylaminomethyl substituents and the formation of the quasiheterocyclic compound **16**, the structure of which was established by X-ray crystallographic analysis. An essential element of the structure that makes it possible to regard it as macrocyclic is the hydrogen bond between the proton of the OH group and the nitrogen atom of the dialkylaminomethyl substituent. It was also found that during crystallization this compound gives stable solvates, in which a solvent molecule is included between two molecules of the macrocycle. The determining role of the Mn(III) as a coordinating oxidant, securing the participation of three molecules of the oxidized substrate in the observed oxidative heterocyclization, was confirmed by a special experiment in which an excess of a competing complexing agent [Mn(II)] blocks trimerization. In the **15**–Mn(III)–Mn(II), 1:1.5, system the formation of only the aldehyde **11** and its partial transformation into 3,5-di-*tert*-butyl-*ortho*-benzoquinone are observed.

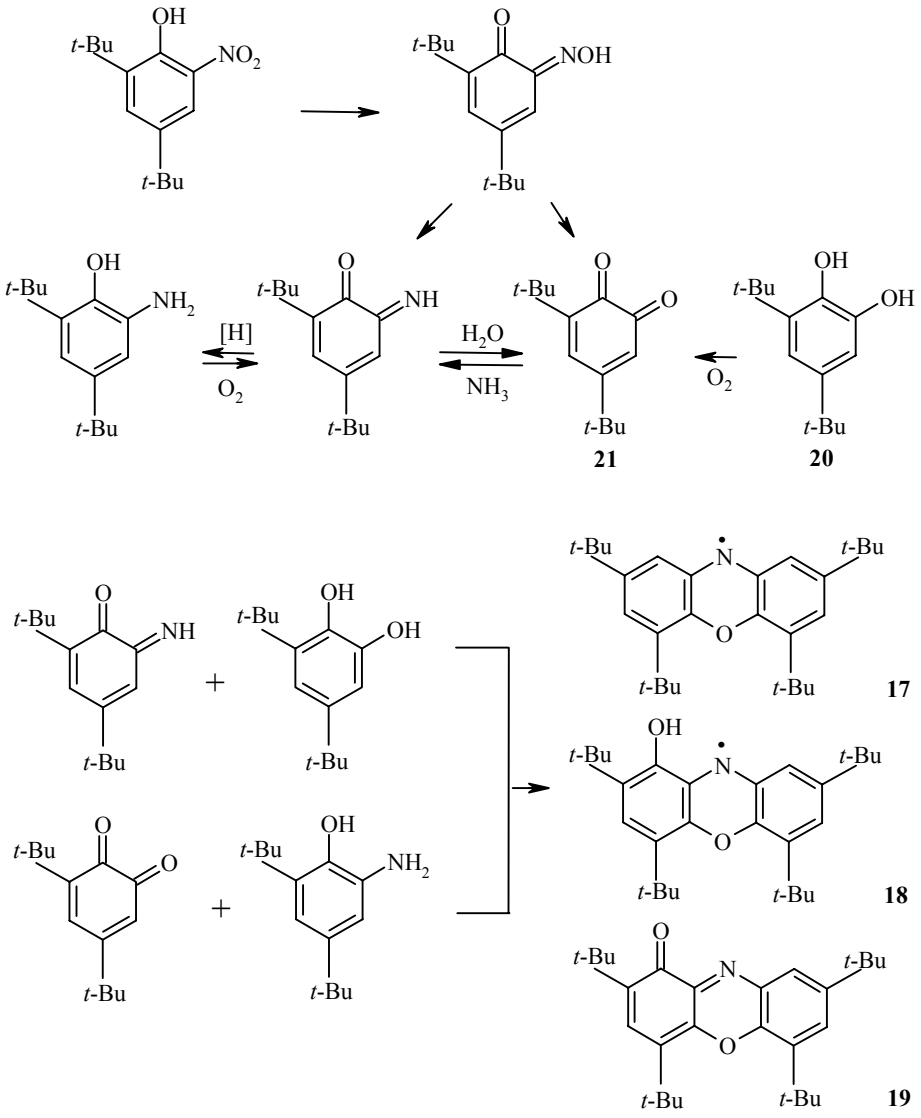
It is interesting to note that during investigation of the quaternization of dialkylaminomethylphenols by the action of RBr with the participation of atmospheric oxygen as oxidant spontaneous dehydroheterocyclization with the formation of quaternized derivatives of the respective benzoxazines was observed, for example:



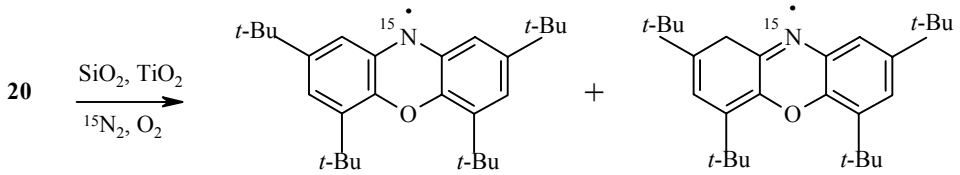
The processes occurring here were investigated by ^{1}H NMR.

4. THE FORMATION OF HETEROCYCLIC COMPOUNDS IN THE REACTIONS OF 2,4- AND 2,5-DI-*tert*-BUTYL-6-NITRO(AMINO-,HYDROXY-)PHENOLS

During the development of methods for the introduction of an amino group at position 6 of 2,4-di-*tert*-butylphenol and also during investigation of the possibility of mutual transformations in 6-amino- and 6-hydroxy-substituted phenols it was found that under certain conditions these transformations are accompanied by the formation of phenoxazine derivatives. These are the hydroxytetrabutylphenoxazinyl radicals **17** and **18** respectively, which were detected by ESR, and tetra-*tert*-butylphenoxazinone **19**, which was isolated preparatively. These compounds are formed during the reduction of 6-nitro-2,4-di-*tert*-butylphenol [7], the oxidation of 6-amino-2,4-di-*tert*-butylphenol, the autooxidation of 3,5-di-*tert*-butylpyrocatechol (**20**) in the presence of ammonia, and the reduction of 3,5-di-*tert*-butyl-*o*-benzoquinone (**21**) by sodium borohydride also in the presence of ammonia. Possible intermediate products common to the above-mentioned reactions are shown in the following scheme:



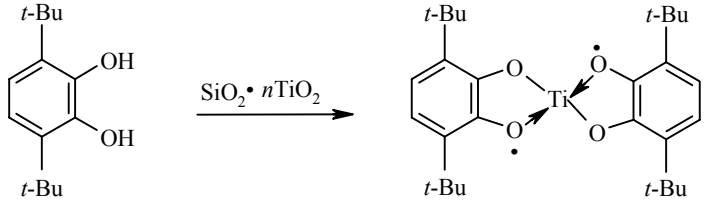
The formation of easily identified colored derivatives of phenoxazine **17** and **18** possessing paramagnetic characteristics proved useful as an indicator reaction that made it possible to detect the participation of atmospheric nitrogen in the transformation of compound **20** on SiO_2 containing Ti and Mn ions [8, 9]. The reaction was also reproduced using $^{15}N_2$. The detection of ^{15}N radicals in the tetra-*tert*-butylphenoxazinyls (ESR spectrum) and tetra-*tert*-butylphenoxazinone (mass spectrum) confirmed the possibility of the reductive fixation of nitrogen in this heterophase system:



17-^{15}N ESR spectrum

18-^{15}N $M^+ = 422$

Analysis of published data on the reductive fixation of nitrogen with the formation of N^{3-} derivatives makes it possible to suppose that the most likely mechanism for the heterophase system based on pyrocatechol **20**–$SiO_2 \cdot n TiO_2$ is a multielectron coordination–catalytic mechanism, in which the pyrocatechol **20** functions as a reducing agent and a redox-labile coordinating ligand. Direct evidence for the formation of metal complexes on the surface of the adsorbent was obtained with the use of 3,6-di-*tert*-butylpyrocatechol (**22**), isomeric with the pyrocatechol **20**, as complexing agent. In particular, it was possible to detect titanium compounds as a natural impurity in nitrogen-fixing samples of SiO_2 [10]:



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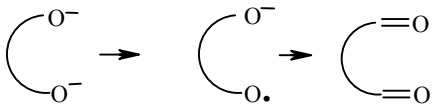
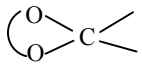
The powerful chelating effect of the two adjacent hydroxyl groups, screened by the *tert*-butyl substituents in the pyrocatechols **20** and **22**, shows up in the ease of formation of various metal complexes with practically all metals of the periodic system [11]. The electronic state of the ligand in these complexes depends on the nature of the metal and its degree of oxidation and varies in the order pyrocatechate–semiquinolate–quinone:



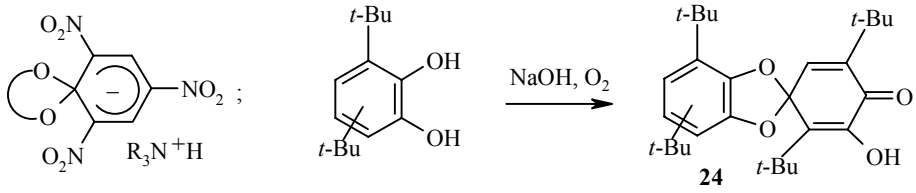
Zerovalent metals, their oxides, and their salts can participate in the formation of chelate complexes [12]. The complexation processes take place both in the solid phase and in solutions. Apart from metals the nodal unit in the cyclic group

can also be carbon, sulfur, or phosphorus atoms. Heterocyclic compounds containing such a structural fragment are formed in various reactions of the pyrocatechols **20** and **22** and also the redox-conjugated quinones **21** and **23**.

Thus, the formation of the ring



is observed in the reactions of pyrocatechols with carbonyl compounds [13], dialkyl and diaryl carbonates [14], dihalogenomethanes [15], tri- and tetrahalogenomethanes, and dihalogenocarbenes [16], and also in the reactions of quinones **21** and **23** with diazomethane [17] and ylides [18, 19]. The cyclic ethers have high stability and do not undergo ring opening during the action of acids, alkalis, and oxidizing agents. One of the clearest signs of this feature in the pyrocatechol derivatives **22** is the extremely high stability of the trinitrocyclohexadienate Meisenheimer complexes with the 1,3-benzodioxolane ring [20]. The structurally similar spirocyclic compounds **24** are formed during the oxidative autocondensation of the pyrocatechols **20** and **22** in the presence of bases:



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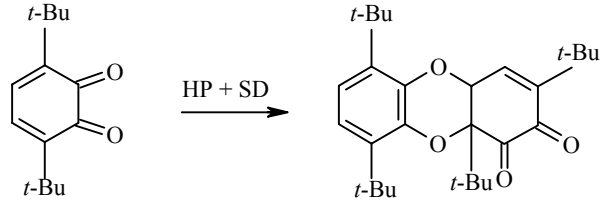
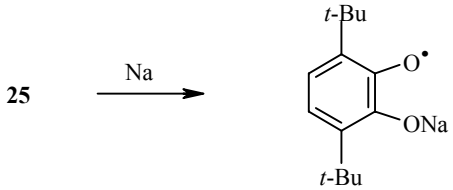
The dimerization of the quinone **23** by the action of high pressure (HP) and shearing deformations (SD) leads to the heterocyclic compound **25** [21].



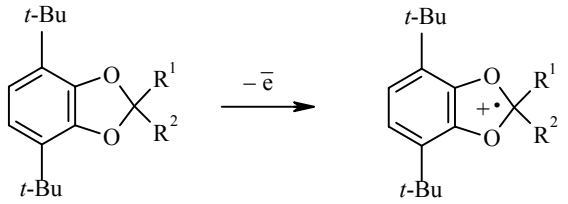
23 **25**

The retrocyclization of such a dimer can be achieved by the action of one-electron reducing agents:



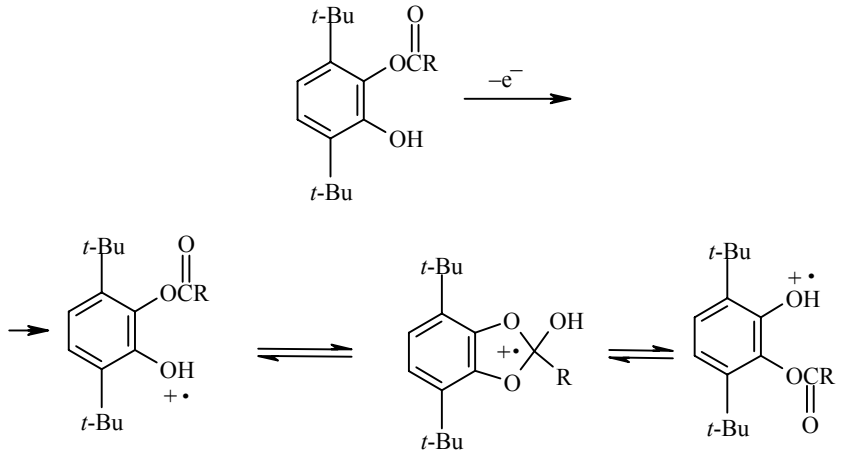
An interesting feature of the heterocyclic compounds **24** and **25** is their ability to undergo single-electron oxidation without destroying the structure. The radical-cations that formed were identified by ESR [22].

It was later found that fairly stable radical-cations are also formed during the oxidation of benzodioxolanes:



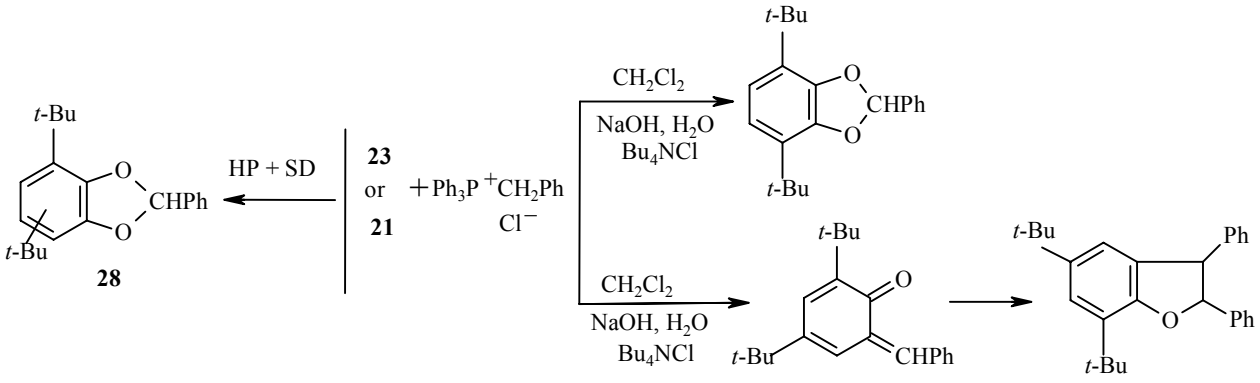
26 **27**

The spectral parameters of a large range of radical-cations based on benzodioxolanes with various substituents R^1 and R^2 have been investigated [23]. It was established that heterocyclic radical-cations of type **27** (with R^1 = OH) are formed as intermediate compounds during the oxidation of monocarboxylic esters of pyrocatechol **22**, which undergo degenerate isomerization with migration of the acyl group [24]:



The capacity for electron donation in conjunction with the proton-accepting characteristics of compounds **26** provided the prerequisite for their use as inhibitors of the thermooxidative destruction of polymers with the release of hydrogen halides (PVC and similar halogenated olefins) [25, 26]. The existence of useful properties in the benzodioxolanes **26** has prompted detailed development of methods for their synthesis. Versions of the realized syntheses are presented in the Scheme 1:

It should be noted that the structurally related isomeric pyrocatechols **20** and **22** and the redox-conjugated quinones **21** and **23** are analogs close in reactivity but nevertheless have a number of significant differences. Thus, the reaction of the quinones **21** and **23** with benzylidenetriphenylphosphorane, generated in a two-phase catalytic system, leads to the formation of structurally different heterocyclic compounds, depending on the steric situation at the carbonyl groups [18]:



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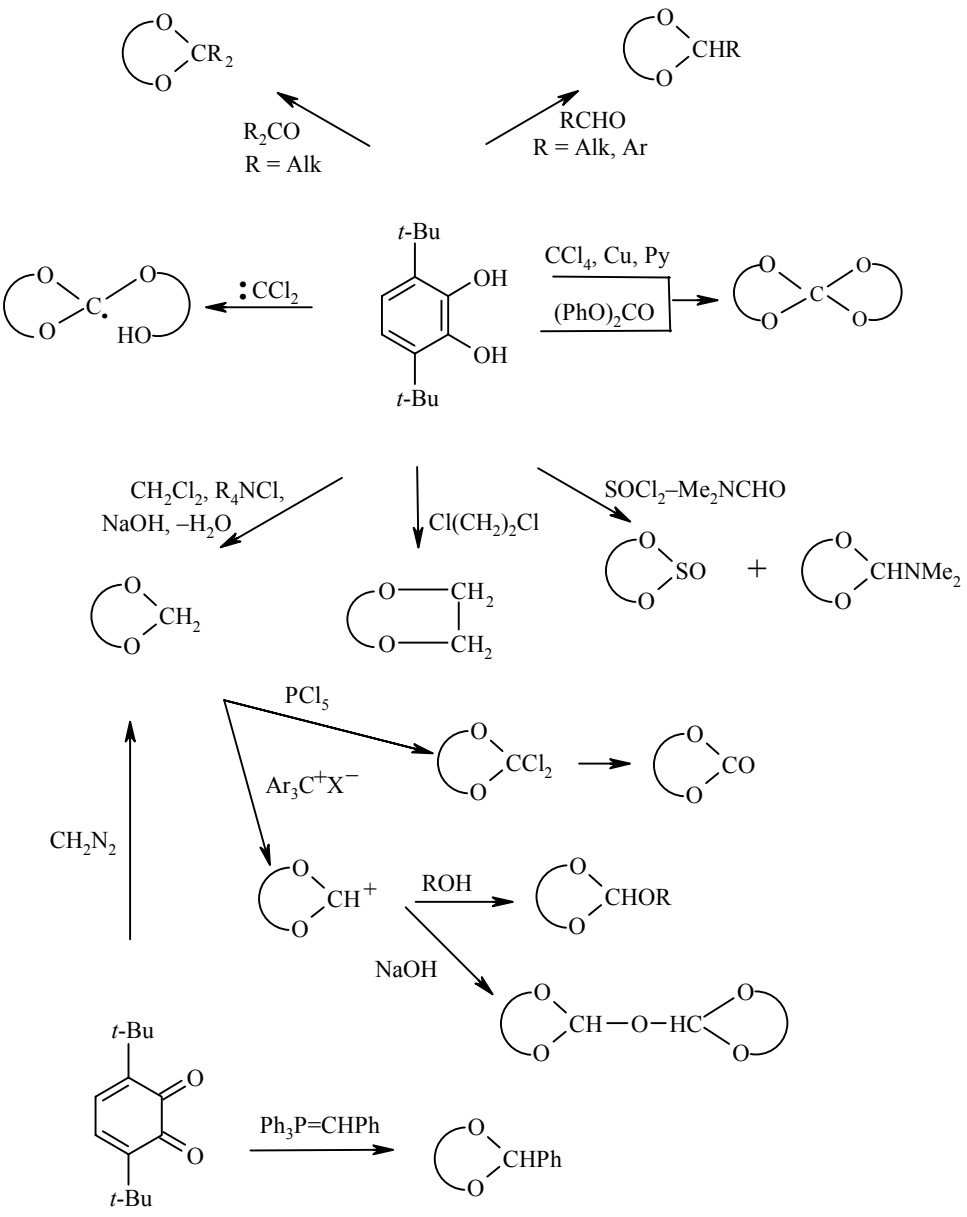
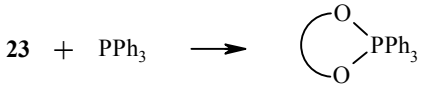
Scheme 1

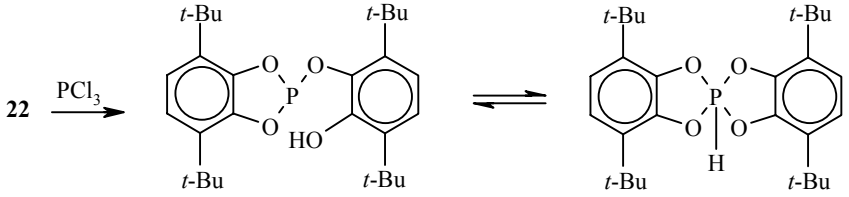


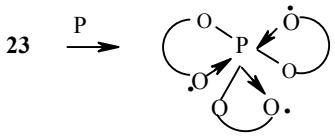
In the solid-phase version of the process in the three-component quinone–benzyltriphenylphosphonium chloride–sodium hydroxide system under the influence of high pressure and shearing deformations the isomeric benzodioxolanes (derivatives of pyrocatechols **20** and **22**) are formed in each case. This indicates a change in the mechanism of the reaction under these conditions as a result probably of change in the electronic structure of the ylide and the realization of a common reaction path in both cases [19].

It is interesting to note that together with the ethers **28** the formation of a certain amount of phosphoranes is observed as a result of reaction of the quinones with the triphenylphosphine released at the first stage:

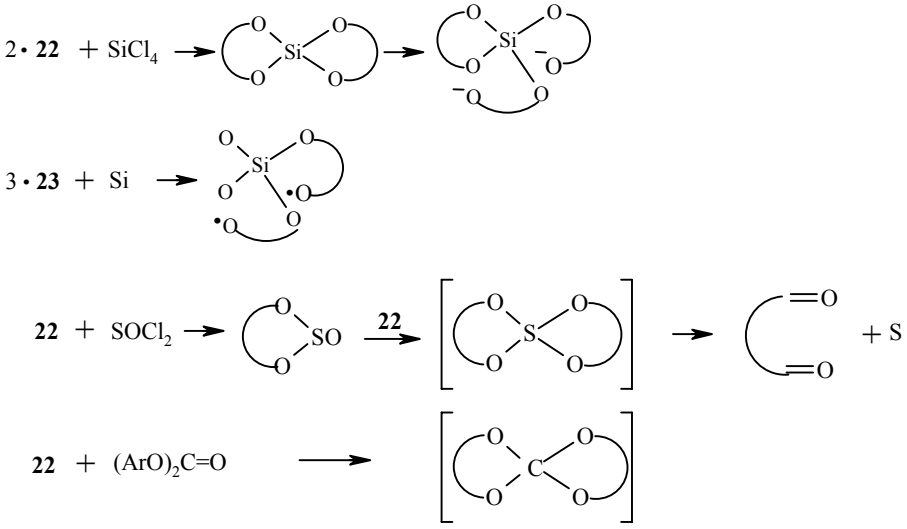


Heterocyclic ethers of pyrocatechols with a nodal phosphorus atom are formed in the reaction of compound **22** with phosphorus trichloride [27] and other phosphorus compounds and also in the reaction of quinones with white phosphorus [28]. A characteristic feature of these reactions is substitution of the maximum number of coordination vacancies at the phosphorus atom by pyrocatechol (or semiquinolate) ligand with the formation of phosphorane structures:

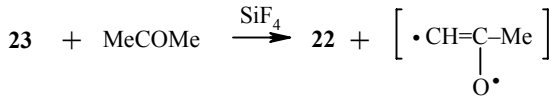




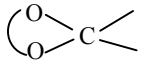
The same tendency for coordination saturation appears in the reaction of the pyrocatechol **22** and quinone **23** with silica and its derivatives [29], with thionyl chloride [14], and with diaryl carbonates [14]:



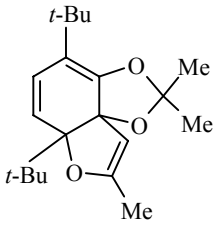
The overwhelming majority of heterocyclic compounds based on the isomeric pyrocatechols **20** and **22** (and the corresponding quinones) are formed with the participation of both hydroxyl (or carbonyl) groups. However, a series of reactions with heterocyclization in the ring was discovered for derivatives of the 3,6 isomer. Thus, the complex of the quinone **23** with SiF_4, being a strong oxidizing agent [30], is capable of dehydrogenating acetone:



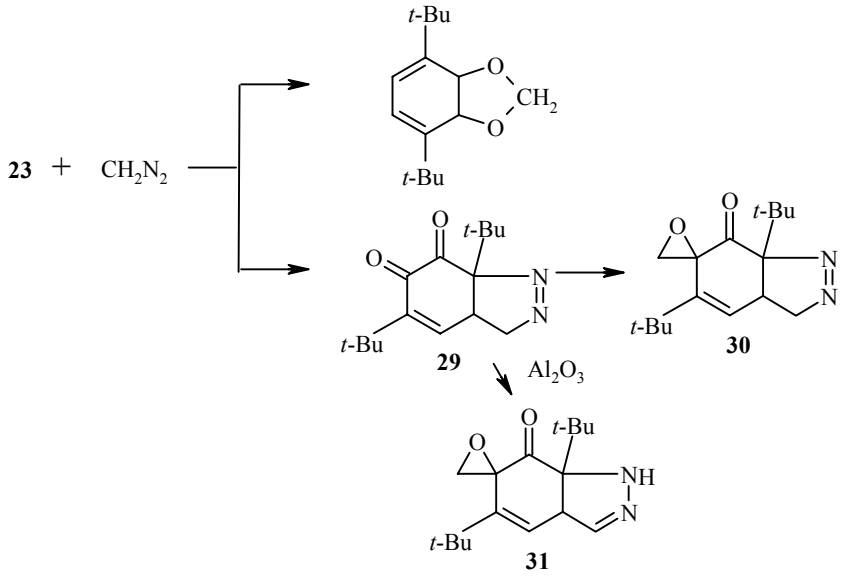
The condensation of the obtained pyrocatechol **22** with acetone leads to benzodioxolane:



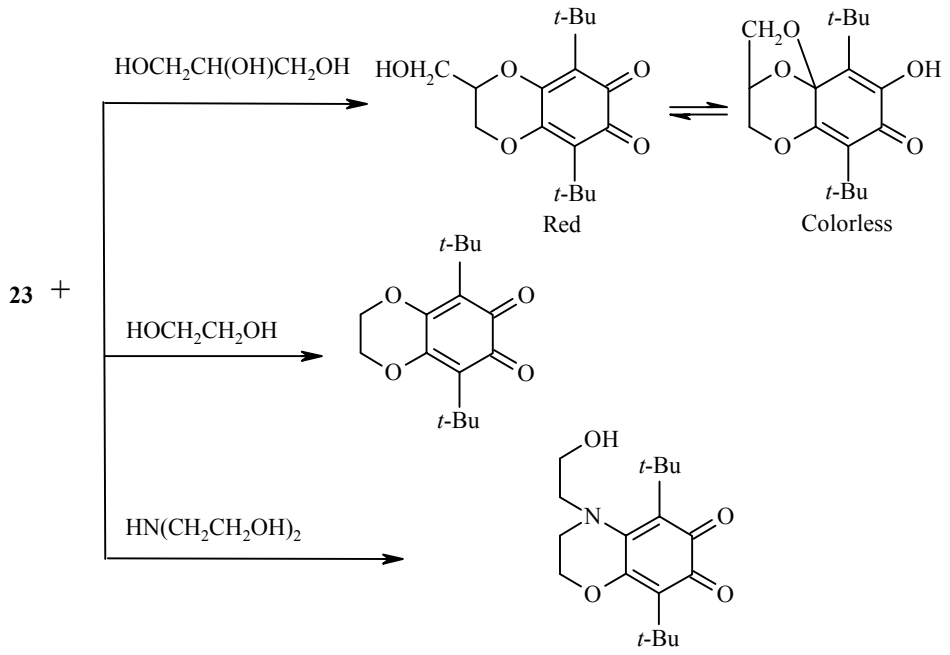
The reaction of the latter with dehydroacetone leads to the formation of a heterocyclic adduct involving the C=C bond of the ring:



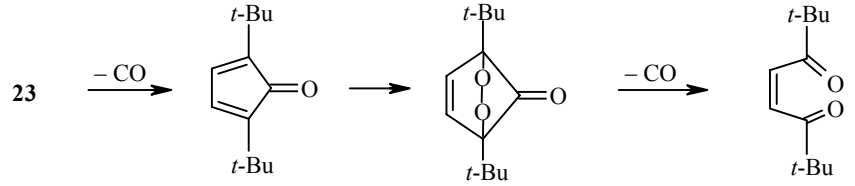
In reaction with diazomethane both isomeric di-*tert*-butyl-*o*-benzoquinones form cyclic ethers with the elimination of N_2. In the case of the isomer **23**, however, an adduct with diazomethane at the C=C bond of the ring, i.e., the pyrazoline derivative **29**, and also the product from its reaction with diazomethane containing an oxirane ring **30** are formed in parallel [17]; the pyrazoline **29** is stable under the conditions of thermolysis but undergoes prototropic isomerization at Al_2O_3 with the formation of the isomeric pyrazoline **31**:



Dehydrocondensation of the quinone **23** with ethylene glycol, glycerol, and diethanolamine and leads to the formation of heterocyclic derivatives of quinone [31]. In the case of glycerol the dehydro adduct exhibits indicator and thermochromic characteristics, due to the mutual transformation of the two isomeric forms:



Most recently a new type of transformation of the quinone **23** under the conditions of photolysis with access to oxygen was discovered. The formation of 1,2-dipivalylethylene as product of the phototransformation forces one to suppose that the reaction takes place in stages through the formation of di-*tert*-butylcyclopentadienone and its cyclic adduct with a dioxygen bridge, loss of the second carbonyl group, and the isomerization characteristic of cyclic peroxides leading to the formation of a dicarbonyl compound:



The scheme proposed for its formation is supported by the phototransformation of the authentic di-*tert*-butylcyclopentadienone obtained as a result of photolysis of the quinone **23**, which also leads to dipivalylethylene. It seems possible that similar transformations occur in natural substituted pyrocatechols and quinones with the participation of atmospheric oxygen and sunlight.

The presented examples of heterocyclization in the transformations of mono- and diatomic di-*tert*-butylphenols give an idea of the possibilities of using phenols in the chemistry of heterocyclic compounds.

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