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# Oxidation of Group II Metals with 3,5-Di-*tert*-butyl-1,2-benzoquinone

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**Abstract**—Kinetic regularities of zinc and cadmium oxidation in systems comprising 3,5-di-*tert*-butyl-1,2-benzoquinone and dimethylformamide or dimethyl sulfoxide are studied. Thermodynamic parameters of oxidant and ligand adsorption on metal surface are estimated. The oxidation of Group II metals (Be, Ca, Mg, Zn, Cd) with 3,5-di-*tert*-butyl-1,2-benzoquinone gives metal bis-*o*-semiquinolates. In the presence of excess metal in the medium of coordinating solvents, diradical products convert to the corresponding catecholate derivatives. It is shown that the dependence of the rates of metal reactions with 3,5-di-*tert*-butyl-1,2-benzoquinone on the donor number of solvent passes through a maximum. With less active metals, the maximum shifts to more basic ligands.

The aim of this work was to study physicochemical peculiarities of oxidation of Group II metals (Be, Mg, Ca, Zn, Cd, Ba, and Hg) with 3,5-di-*tert*-butyl-1,2-benzoquinone (**I**). The obtained results and published kinetic data [1–6] on oxidative solution of Group I–IV metals in the presence of 3,5-di-*tert*-butyl-1,2-benzoquinone in various solvents allowed some general regularities of this process to be established.

Oxidation of magnesium, zinc, and cadmium at temperatures below 100°C takes place only in solvents whose donor number [7] is no higher that 100 kJ/mol (DMF, DMAA, DMSO, and pyridine). Barium and calcium are more reactive, and they dissolve in the presence of 3,5-di-*tert*-butyl-1,2-benzoquinone in organic solvents of various nature. Berillium and mercury fail to react with 3,5-di-*tert*-butyl-1,2-benzoquinone in any of the above-mentioned solvents within 500 h at 90°C. This is probably connected with high ionization potentials of these metals [8].

As shown in [2–5], metal reactions with 3,5-di-*tert*-butyl-1,2-benzoquinone in various solvents may lead to formation of bis-*o*-semiquinolate complexes $M(SQ)_2$ (SQ is semiquinone formed by one-electron reduction of compound **I**) or catecholate complexes CatM (Cat is cathecol formed by two-electron reduction of compound **I**).

The reaction of Group II metals with 3,4-di-*tert*-butyl-1,2-benzoquinone is initially accompanied by changes in the color of the solution from red to green and then to blue. The products of magnesium, zinc, and cadmium reactions partially precipitate from the reaction mixtures as blue crystals. The weight loss of the reacting metal and the amount of the metal in the solution at the moment of disappearance of green coloration correspond to a 1:2 complex in the case of Mg, Zn, Cd, Ba, and Ca.

Monitoring of the reactions of metals (Mg, Zn, and Cd in DMF, Cd in pyridine, and Ba and Ca in THF and toluene) with 3,4-di-*tert*-butyl-1,2-benzoquinone by ESR spectroscopy showed that paramagnetic products are formed just after reagent mixing. In the case of DMF, pyridine and THF, the primarily observed isotropic spectra (295 K) are doublets of multiplets ($g$ 2.003) with splitting on the proton at $C^4$ of the *o*-quinone ring ($a_H$ 3.4 Oe) and on *tert*-butyl protons ($a_H$ 0.3 Oe). In the reaction of cadmium with compound **I**, satellite splitting on magnetic isotopes of the metal ($^{111}$Cd: $J$ 1/2, $\mu$ –0.5922, 12.86%; $^{113}$Cd: $J$ 1/2, $\mu$ –0.6195, 12.24%), $a_{Cd}$ = 6.4 Oe [9], is observed (Fig. 1a).

These data suggest formation of monoradical species in the reaction mixture. The presence of satellite splitting does not permit us to assign the observed spectra to free *o*-semiquinolate radical anions. Analogous signals were observed in [2, 4] in oxidation of metallic tin and copper with *o*-quinone in THF. The appearance of the spectra of monoradical species may be caused by expulsion of one of the semiquinone ligands in $M(SQ)_2$ into the outer sphere of the coordination complex with the solvent [10]. An alternative are products of *o*-quinone reaction with the

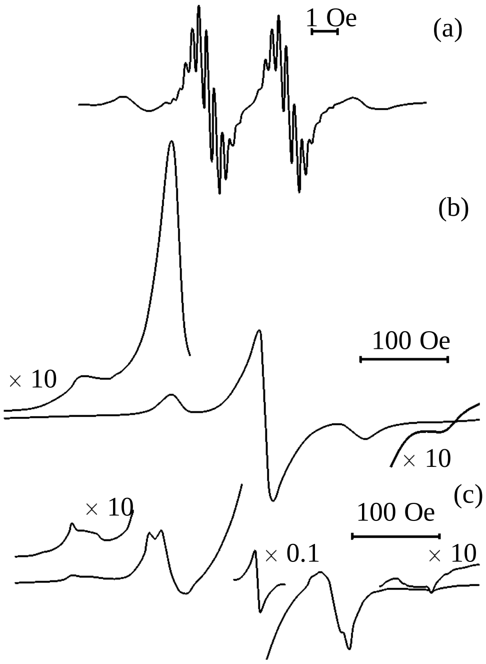


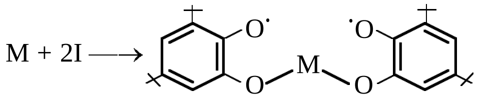
**Fig. 1.** ESR spectra of the reaction products in the system Cd–compound **I**–L. (a) Isotropic spectrum at 295 K, L = pyridine; (b) anisotropic spectrum at 130 K, L = pyridine; and (c) anisotropic spectrum at 130 K, L = DMF.

surface metal oxide film. But, in view of the fact that in the reactions of calcium and barium with 3,5-di-*tert*-butyl-1,2-benzoquinone in toluene which cannot effectively solvate metal ions, the ESR spectra contain a strong singlet with broad wings without splitting on protons of the *o*-quinone ligand even at initial degrees of transformation, the first first proposal seems to be more correct. Analogous alterations are observed in the ESR spectrum of the reaction products of 3,6-di-*tert*-butyl-1,2-benzoquinone with zinc and cadmium amalgams in toluene and THF [11].

The anisotropic ESR spectra of the reaction mixtures of 3,5-di-*tert*-butyl-1,2-benzoquione with Mg, Zn, and Cd in DMF, Cd in pyridine, and Ca in THF show a superposition of a singlet signal and signals typical of biradical species (Figs. 1b, 1c). The zero-field splitting constants of the resulting biradicals are as follows: $|D|$ 470 Oe and $|E|$ 0 for Mg in DMF; $|D|$ 436 Oe and $|E|$ 0 for zinc in DMFA; $|D|$ 448 Oe and $|E|$ 7 E for cadmium in DMF; $|D|$ 328 Oe and $|E|$ 0 for cadmium in pyridine; and $|D|$ 300 Oe and $|E|$ 0 for calcium in THF. The above-mentioned spectra can be assigned to bis-*o*-semiquinolate derivatives of bivalent metals $M(SQ)_2$ [11–13]. The distances between the radical centers, calculated from the parameter $D$ in the point-dipole approximation [14] are 4.9, 5.0, 5.0, 5.5, and 5.7 Å. The estimated zero-field splitting parameters for $Cd(SQ)_2$ and $Zn(SQ)_2$ [3] in various solvents tells that coordinating ligands may significantly affect the spatial arrangement of the resulting complexes. In pyridine instead of DMF with both the metals, the distance between the radical centers increases from 5.0 to 5.5 Å. In the half field in all the cases, too, a weak signal with $\Delta m_s$ 2 ($H$ 1698.5 Oe) is observed characteristic of biradical species.

The anisotropic ESR spectra of the reaction products of barium with 3,5-di-*tert*-butyl-1,2-benzoquinone in toluene and THF contain no signals assignable to triplet species. No biradicals were found also in the reactions of barium with 3,5-di-*tert*-butylcatecholate in THF or DMF. In all the above-mentioned cases we observed no other signals than a strong singlet indicative of the presence of radical species in the reaction mixtures. These results are evidently caused by oligomerization of $Ba(SQ)_2$ molecules via formation of bridging bonds through the *o*-semiquinone oxygen. This structure can be destroyed only under the action of strong coordinating agents, such as *N*,*N*,*N*',*N*'-tetraethylenediamine [12].

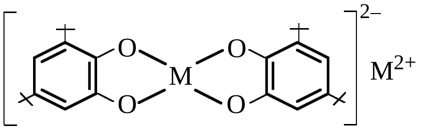
At a 1 : 2 metal : **I** ratio, the ESR spectra revealed formation of compounds like $M(SQ)_2$ in the first stage of reaction.



(1)

As radical species are accumulated, the intensity of the initial signal in the isotropic EPR spectrum increases. Simultaneously, spectral lines broaden and the hyperfine splitting from *o*-semiquinone protons disappear.

On further keeping of reaction mixtures containing $M(SQ)_2$ over excess metals in all the solvents used, except for hydrocarbons (with calcium and barium), the ESR signal gradually disappears, and the blue solution gets yellowish brown. The reagent ratio in the liquid phase becomes 1 : 1. Upon completion of reaction, the ESR signal disappear completely. In view of the aforesaid, as well as data in [3], we can state that the final products of the reactions of magnesium, zinc, cadmium, calcium, and barium with 3,5-di-*tert*-butyl-1,2-benzoquinone in coordinating solvents in the presence of excess metals are the corresponding catecholate derivatives. By analogy with [11, 15], we propose that the catecholate complex comprises a dianion with a four-coordinate metal atom.



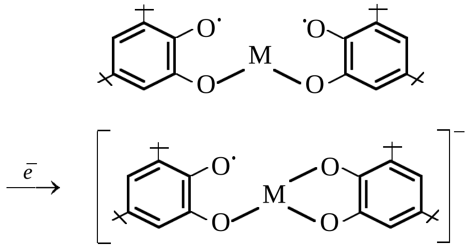
Note that our present results for the reactions with compound **I** of magnesium in DMF and of zinc in pyridine disagree with data in [3, 5], according to which the same systems after 50 h contain bis-*o*-semiquinolate metal derivatives. As we found in the present work, such reactions require more time to be complete.

Metal catecholates are formed by reactions of bis-*o*-semiquinolates with excess metals. Therewith, reaction (1) is faster than reaction (2).

$$M(SQ)_2 + M \longrightarrow CatM. \qquad (2)$$

Hence, the rates of the reactions of zinc with 3,5-di-*tert*-butyl-1,2-benzoquinone and $Zn(SQ)_2$ in DMF at reagent concentrations of 0.05 M (343 K) are $2 \times 10^{-4}$ and $1.4 \times 10^{-4}$ g $cm^{-2}$ $min^{-1}$, respectively.

It should specially be noted that the isotropic ESR spectra measured at final steps of the reactions of Group II metals with compound **I** contain a signal characteristic of mono-SQ metal complexes, similar to that presented in Fig. 1a. This signal can be assigned an intermediate reduced form $M(SQ)_2$ containing one o-semiquinolate and one catecholate ligands.



(3)

Analogous ESR signal, together with the signal of $M(SQ)_2$, appears when *o*-quinone is added to a solution of CatM in an amount smaller than required by the stoichiometry of reaction (4).

$$CatM + \mathbf{I} \rightleftarrows 2M(SQ)_2. \qquad (4)$$

It occurred that equilibrium (4) for Group II metals is strongly shifted to the right. In the reactions of stoichiometric amounts of metal catecholates with 3,5-di-*tert*-butyl-1,2-benzoquinone in THF, DMF, DMSO, and pyridine, metal bis-*o*-semiquinolates are formed just on reagent mixing, as judged from the blue coloration of the solution and the appearance of the corresponding ESR spectrum. Analogous results were obtained by Razuvaev and co-workers [16, 17], who showed that reactions of sodium and thallium catecholates with compound **I** in THF yield *o*-semiquinolates.

On the other side, according to [2–4], the position of equilibrium (4) may strongly depend on the nature of the solvent used. It was found that CatM (M = Zn) converts to a biradical product when DMAA is substituted by pyridine [3]. We relate these results to uncontrolled penetration of traces of atmospheric oxygen on changing the solvent. The fact is that in the absence of the free quinone the trasformation of metal catecholate to bis-*o*-semiquinolate must be accompanied by metal separation, but this is not the case. In [2, 4], the transformation of copper and tin bis-*o*-semiquinolates to the corresponding catecholates on substitution of THF by the more basic pyridine has also been reported. Such transformations are most likely connected with the ability of *o*-quinone complexes of these metals to intramolecular redox reactions [18]. Coordination of the ligand possessing a high donor capacity to a metal facilitates transfer of the second electron from the metal to *o*-semiquinolate radical anion, which leads to formation of a catecholate structure.

The rate of accumulation of catecholate derivatives in the system metal–3,5-di-*tert*-butyl-1,2-benzoquinone–solvent may considerably differ from the rate of metal oxidation. Magnesium reacts with compound **I** in pyridine several times slower than in DMF [5]. But the highest rate of CatM (M = Mg, Zn, Cd) formation is observed in pyridine, where at a 8 : 1 metal : *o*-quinone molar ratio the process is complete in 50 h at 70°C with zinc and cadmium and in 2 weeks with magnesium. When DMF or DMAA is used instead of pyridine, from 2 weeks to 2 months are required for complete reaction. In dimethyl sulfoxide, CatZn is formed relatively quickly (~50 h) compared to CatMg and CatCd (10 weeks).

Magnesium, zinc, and cadmium react with 3,5-di-*tert*-butyl-1,2-benzoquinone at 70°C considerably slower than barium and calcium. For complete oxidation of the latter, from 5 to 48 h are needed, depending on the nature of the solvent. The reactions of Ba and Ca with compound **I** in hydrocarbons stop on the stage of formation of bis-*o*-semiquinolate complexes even with excess metal. In other solvents, the ratio of converted metal and o-quinone and the absence of an ESR signal point to formation of CatM as the final product. The highest reaction rate in the case of barium and calcium is observed in THF and ethyl acetate, while DMF and DMSO proved to be much less active solvents.

Probably, the relative facility of formation of catecholate complexes in the oxidation of Mg, Zn, and Cd with 3,5-di-*tert*-butyl-1,2-benzoquinone in pyridine, as compared to the same process in DMF, is

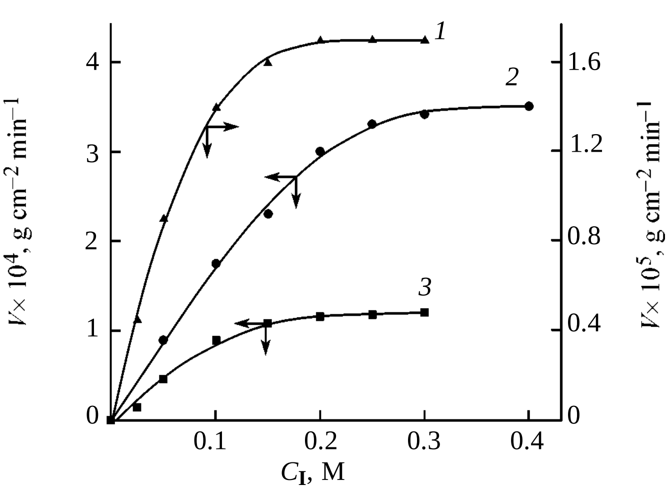


**Fig. 2.** Rate of metal oxidaton in the system M–compound **I**–L vs. *o*-quinone concentration. (*1*) M = Cd, L = DMF, $c_L$ 13 M; (*2*) M = Zn, L = DMF, $c_L$ 13 M; and (*3*) M = Cd, L = DMSO, $c_L$ 14 M. Temperature, K: (*1, 3*) 353 and (*2*) 323.

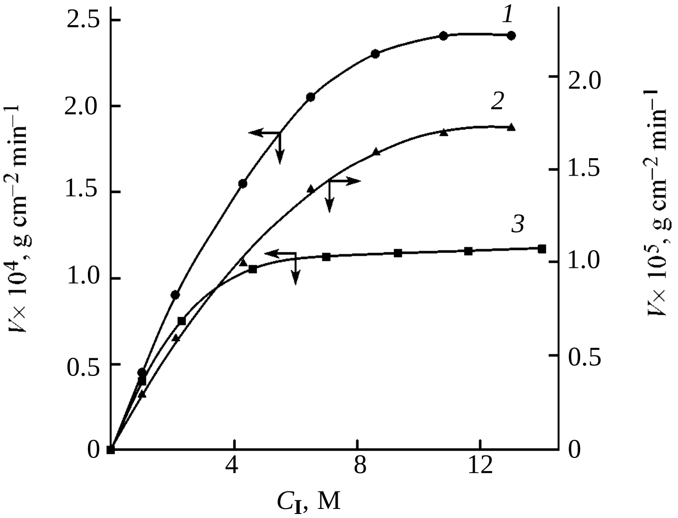


**Fig. 3.** Rate of metal oxidation in the system M–compound **I**–L vs. ligand concentration. $c_I$ 0.15 M. (*1*) M = Zn, L = DMF; (*2*) M = Cd, L = DMF; and (*3*) M = Cd, L = DMSO. Temparature, K: (*1*) 323 and (*2, 3*) 353.

connected with the higher donor ability of the former solvent [7]. Contrary to that, in hydrocarbons which have low $D_N(\mathrm{SbCl_5})$ values no reduction of $\mathrm{M(SQ)_2}$ to CatM (M = Ca, Ba) over excess metal occurs.

Dependences of the rate of developed oxidation of zinc and cadmium on the concentrations of oxidant and look like saturation curves (Figs. 2, 3). Such kinetic curves relate to Langmuir–Hinshelwood reactions [18]. The $V/V_{\max} = f(c_{\mathrm{ox}})$ curves at different ligand concentrations coincide with each other. That means that the reagents are adsorbed on reaction centers of different nature. Analogous schemes were earlier proposed for the oxidations with *o*-quinone of magnesium in DMF [5] and of zinc, tin, and indium in DMSO [3, 4, 6]. The existence of two types of reaction centers on the surface of zinc, cadmium, and tin has been reported in [20]. Hence, independently of the nature of the solvent, Group II–IV metals are oxidized with 3,5-di-*tert*-butyl-1,2-benzoquinone by a common mechanism. The total scheme of the process can be described by the following equations:

$$\mathrm{Ox} + \mathrm{S_1} \xrightleftharpoons{K_{\mathrm{Ox}}} \mathrm{OxS}, \tag{5}$$

$$\mathrm{L} + \mathrm{S_2} \xrightleftharpoons{K_{\mathrm{L}}} \mathrm{LS}, \tag{6}$$

$$\mathrm{OxS} + \mathrm{LS} \xrightarrow{k} \text{Reaction products.} \tag{7}$$

Here $K_{\mathrm{ox}}$ and $K_{\mathrm{L}}$ are the equilibrium constants of oxidant and ligand adsorption, respectively, and $k$ is the rate constant of the surface reaction.

The rate of the process has the equation:

$$V = \frac{kS_{01}S_{02}K_{\mathrm{L}}K_{\mathrm{Ox}}C_{\mathrm{L}}C_{\mathrm{Ox}}}{(1 + K_{\mathrm{L}}C_{\mathrm{L}})(1 + K_{\mathrm{Ox}}C_{\mathrm{Ox}})}, \tag{8}$$

where $S_{01}$ and $S_{02}$ are the numbers of active centers per unit metal surface.

Linearization of the kinetic curves in $1/V$–$1/C$ coordinates allows the equilibrium constants of oxidant and ligand adsorption and the reaction rate constants to be estimated. The temperature dependences of the resulting values give the apparent activation energies, enthalpies and entropies of adsorption of the components of the oxidizing mixture on metal surface. Table 1 lists experimental data, as well as published data for oxidation of magnesium with 3,5-di-*tert*-butyl-1,2-benzoquinone in DMF [5].

Noteworthy is an unusual reactivity order of metals for their oxidation with 3,5-di-*tert*-butyl-1,2-benzoquinone in DMF (Mg < Zn > Cd). The redox potentials of these metals in water [21] expect another activity order: Mg > Zn > Cd. On the other side, the equilibrium constants of oxidant adsorption on metal surface increase in the following order: Mg < Zn < Cd (Table 1). Evidently, a joint effect of these two factors acting in opposite directions is responsible for the observed trends in reaction rate constants for these metals.

As shown in [2–6], the dependence of the rate of copper, zinc, tin, magnesium, and indium oxidation with 3,5-di-*tert*-butyl-1,2-benzoquinone on the donor number of the solvent passes through a maximum.

**Table 1.** Apparent rate constants and activation energies, equilibrium constants, enthalpies and entropies of ligand and oxidant absorption for reactions with 3,5-di-*tert*-butyl-1,2-benzoquinone of magnesium [5], zinc, and cadmium in DMF and of cadmium in DMSO

| $T$, K | $K_{\mathrm{Ox}}$ | $K_{\mathrm{L}}$ | $-\Delta H_{\mathrm{ads}}^{\mathrm{Ox}}$, kJ/mol | $-\Delta S_{\mathrm{ads}}^{\mathrm{Ox}}$, J mol$^{-1}$ K$^{-1}$ | $-\Delta H_{\mathrm{ads}}^{\mathrm{L}}$, kJ/mol | $-\Delta S_{\mathrm{ads}}^{\mathrm{L}}$, J mol$^{-1}$ K$^{-1}$ | $k$, mol cm$^{-2}$ min$^{-1}$ | $E_{\mathrm{a}}$, kJ/mol |
|---|---|---|---|---|---|---|---|---|
| | | | | Mg–DMF | | | | |
| 343 | 2.3 | 0.084 | 22 ± 2 | 57 ± 5 | 23 ± 1 | 89 ± 7 | $2.3\times10^{-6}$ | 63 ± 4 |
| 353 | 1.9 | 0.067 | | | | | $3.9\times10^{-6}$ | |
| 363 | 1.5 | 0.054 | | | | | $7.3\times10^{-6}$ | |
| | | | | Zn–DMF | | | | |
| 323 | 6.7 | 0.174 | 44 ± 1 | 121 ± 3 | 17 ± 1 | 68 ± 1 | $1.2\times10^{-5}$ | 52 ± 4 |
| 333 | 3.9 | 0.143 | | | | | $2.1\times10^{-5}$ | |
| 343 | 2.7 | 0.119 | | | | | $4.0\times10^{-5}$ | |
| | | | | Cd–DMF | | | | |
| 353 | 10.8 | 0.144 | 32 ± 4 | 69 ± 7 | 31 ± 1 | 101 ± 2 | $3.0\times10^{-7}$ | 113 ± 2 |
| 363 | 8.4 | 0.107 | | | | | $8.7\times10^{-7}$ | |
| 369 | 6.7 | 0.091 | | | | | $15.6\times10^{-7}$ | |
| | | | | Cd–DMSO | | | | |
| 353 | 10.9 | 0.6 | 34 ± 3 | 72 ± 7 | 76 ± 1 | 218 ± 3 | $1.6\times10^{-6}$ | 73 ± 1 |
| 363 | 8.2 | 0.3 | | | | | $3.2\times10^{-6}$ | |
| 369 | 6.6 | 0.2 | | | | | $4.7\times10^{-6}$ | |

**Table 2.** Oxidation rates of magnesium [5], zinc [3], and cadmium with 3,5-di-*tert*-butyl-1,2-benzoquinone in various solvents ($C_{\mathrm{I}}$ 0.15 M, $C_{\mathrm{L}}$ 10 M)

| Solvent | $D_N$(SbCl$_5$), kJ/mol [7] | $V_{\mathrm{Mg}}^{343\,\mathrm{K}}\times10^6$, g cm$^{-2}$ min$^{-1}$ | $V_{\mathrm{Zn}}^{323\,\mathrm{K}}\times10^4$, g cm$^{-2}$ min$^{-1}$ | $V_{\mathrm{Cd}}^{363\,\mathrm{K}}\times10^5$, g cm$^{-2}$ min$^{-1}$ |
|---|---|---|---|---|
| Diglyme | 100.8 | 0.0 | 0.0 | 0.2 |
| DMF | 111.3 | 7.5 | 0.7 | 3.0 |
| Dimethylacetamide | 116.7 | 3.2 | 3.0 | 3.2 |
| DMSO | 125.8 | 0.8 | 4.0 | 13.0 |
| Pyridine | 139.1 | 0.2 | 0.4 | 16.0 |
| Hexametapol | 162.9 | 0.0 | 0.0 | 0.1 |

Analogous dependence was established for the reaction of *o*-quinone with cadmium. Table 2 lists the reaction rates of cadmium, zinc [3], and magnesium [5] with 3,5-di-*tert*-butyl1,2-benzoquinone in various solvents.

Certain aspects of the problem concerning such extremal dependences have been considered in [22]. Note that the maximum rate of metal oxidation with 3,5-di-*tert*-butyl-1,2-benzoquinone in the series Mg–Cd–Zn shifts from DMF to a stronger donor, pyridine. For calcium and barium, which are more active metals, solvents of moderate basicity, such as ethyl acetate and THF, are optimal. Therefore, the more active is the metal, the less basic medium is needed for its fastest oxidation.

## EXPERIMENTAL

The ESR spectra were measured on a Bruker ER-200D-SRC radiospectrometer (working frequency 9.5 HHz) with an ER-4111VT thermostating block at the Analytical Center, Institute of Organometallic Chemistry, Russian Academy of Sciences. The *g* factors were determined using diphenylpicrylhydrazyl as reference.

The following metals were used: Mg, MCh-1 grade, State Standard 804-53; Zn, Technical Specifications MKhP 112-40; Cd, Technical Specifications MKhP ORU 35-55; Ba, TsM Technical Specifications 4774-54; and Ca (distilled), Technical Specificaions 95.123-76. Zinc and cadmium wires (diameter 0.5 mm) were made of the above metals and used without additional treatment.

Kinetic measurements were performed by the resistometric method [23] modified for operations with easily oxidized and hydrolyzed compounds.

Organic solvents were purified and dried according to [24]. Before use they were degassed by repeated freeze–pump–thaw cycles.

3,5-Di-*tert*-butyl-1,2-benzoquinone was prepared according to [25]. The melting point of the obtained compound (389 K) is consistent with published data.

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