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Reaction of *N*-Acyl- and *N*-[*N*-Arylsulfonylbenz(acet)imidoyl]-1,4-benzoquinone Monoimines with Hydrazoic Acid

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Abstract—*N*-Aroyl-3,5-dimethyl- and *N*-[*N*-arylsulfonylbenz(acet)imidoyl]-3,5-dimethyl-1,4-benzoquinone monoimines react with hydrazoic acid according to the 1,4-addition pattern. *N*-Acyl-2,3,5,6-tetrachloro-1,4-benzoquinone monoimines take up hydrazoic acid at the double C=N bond (1,2-addition).

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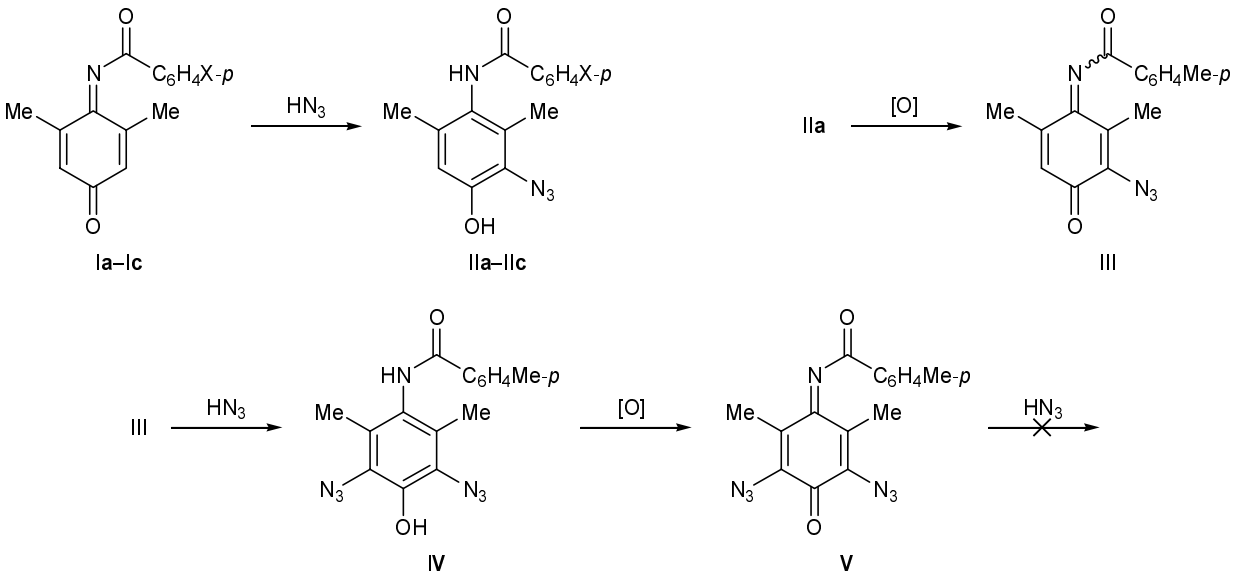
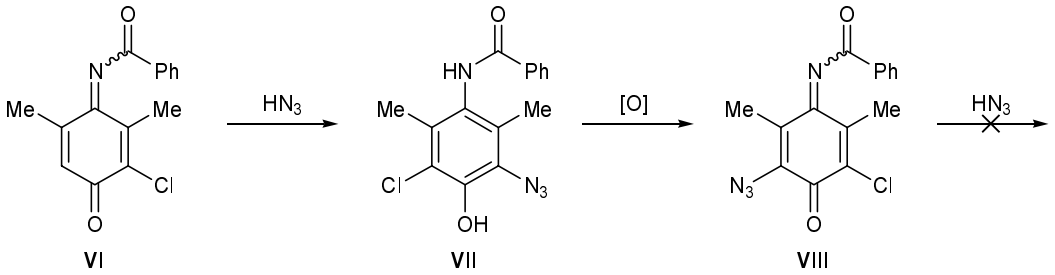
Reactions of *N*-aryl-1,4-benzoquinone imines with hydrazoic acid have been studied in detail [1, 2]. Addition of HN_3 to *N*-(4-tolyl)-1,4-benzoquinone imine, followed by oxidation of the adduct, leads to 2-azido-, 2,5- and 2,6-diazido-, and 2,3,6-triazido-1,4-benzoquinone monoimines. The addition of HN_3 to *N*-arylsulfonyl-1,4-benzoquinone mono- and diimines and *N*-arylsulfonyl-1,4-naphthoquinone monoimines was studied most thoroughly [3–7]. It was shown that HN_3 behaves like hydrogen chloride, i.e., its reaction with *N*-arylsulfonyl-1,4-benzoquinone monoimines follows the 1,4-addition pattern. The regioselectivity of reactions of *N*-arylsulfonyl-1,4-benzoquinone imines with HN_3 was discussed in [8]. In all cases, the reactions with quinone imines were carried out with sodium azide in acetic acid. If a chlorine atom was present in the quinoid ring of *N*,*N*′-bis(phenylsulfonyl)-1,4-benzoquinone diimine, the 1,4-addition was accompanied by nucleophilic replacement of the halogen atom by azido group [5].

Rajappa and Shenoy [7] studied the addition of HN_3 to an unsymmetrically substituted quinone diimine. As in the reaction with hydrogen chloride, in was believed that the first stage is protonation of the most basic

Scheme 1.



Scheme 2.



VI **VII** **VIII**

center in *N*-methylsulfonyl-*N*′-dichloroacetyl-1,4-benzoquinone diimine. Next followed attack by azide ion on the quinoid ring at the most electron-deficient carbon atom in the *ortho*-position with respect to the $MeSO_2N=$ group.

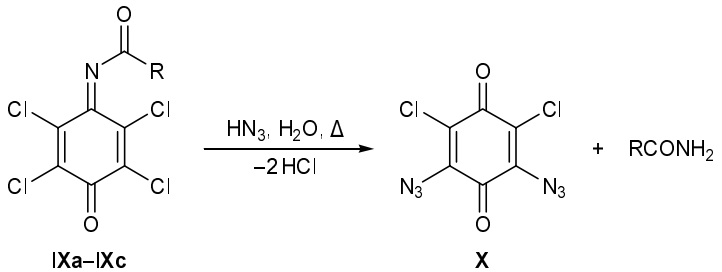
Nucleophilic replacement of the halogen by azido group was observed in reactions of *N*-arylsulfonyl-1,4-benzoquinone monoimines having a chlorine atom in the *ortho*-position with respect to the carbonyl group [4]. *N*-Arylsulfonyl-2,3,5,6-tetrachloro-1,4-benzoquinone monoimines in which the C=N bond is sterically strained reacted with HN_3 at room temperature according to the 1,2-addition pattern, yielding 4-azido-4-arylsulfonylamino-2,3,5,6-tetrachlorocyclohexa-2,5-dien-1-ones; at elevated temperature, both chlorine atoms in the *ortho*-position with respect to the carbonyl group were replaced. The 1,2-addition products were shown for the first time to undergo migration of the azido group from C^4 to C^2 with simultaneous nucleophilic replacement of the 2-chlorine atom [4]. *N*,*N*′-Bis(arylsulfonyl)-2,3,5,6-tetrachloro-1,4-benzoquinone diimines reacted with one or two HN_3 molecules to give 1,2-addition products at one or two C=N bonds, respectively [4].

N-Arylsulfonyl-1,4-naphthoquinone monoimines reacted with hydrazoic acid in a way similar to *N*-arylsulfonyl-1,4-benzoquinone monoimines. The chlorine atom in the *ortho*-position with respect to the carbonyl group in *N*-arylsulfonyl-2-chloro(or 2,3-dichloro)-1,4-naphthoquinone monoimines was replaced by azido group [4].

In continuation of our studies on reactions of N-substituted *p*-quinone imines with hydrazoic acid, in the present work we examined the behavior of *N*-aroyl(acetyl)- and *N*-[*N*-arylsulfonylbenz(acet)imidoyl]-1,4-benzoquinone monoimines in the same reaction. Like *N*-arylsulfonyl-3,5-dimethyl-1,4-benzoquinone monoimines [4], *N*-aroyl-3,5-dimethyl-1,4-benzoquinone monoimines **Ia**–**Ic** reacted with HN_3 according to the 1,4-addition pattern, yielding *N*-aroyl-4-amino-2-azido-3,5-dimethylphenols **IIa**–**IIc** (Scheme 1), though 1,2-addition could be expected taking into account the presence of an activated sterically strained C=N bond [9]. By successive oxidation, addition of HN_3, and oxidation again we obtained *N*-4-toluoyl-2,6-diazido-3,5-dimethyl-1,4-benzoquinone monoimine **V** as final product which failed to react with HN_3.

Menafova [9] showed that *N*-arylsulfonyl-2,6-diazido-3,5-dimethyl-1,4-benzoquinone monoimines can be synthesized from *N*-arylsulfonyl-2-chloro-3,5-dimethyl-1,4-benzoquinone monoimines. The first stage in the synthesis is 1,4-addition of hydrazoic acid, the adduct is subjected to oxidation, and the final stage is nucleophilic substitution of the chlorine atom by azido group. In the reaction with *N*-benzoyl-2-chloro-3,5-dimethyl-1,4-benzoquinone monoimine (**VI**), only 1,4-addition of hydrazoic acid occurred. The resulting *N*-benzoyl-4-amino-6-azido-2-chloro-3,5-dimethyl-

Scheme 3.



IXa–IXc **X**

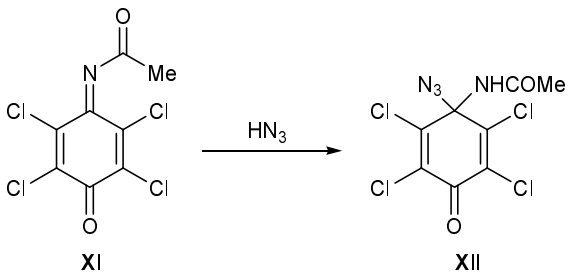
R = Ph (**a**), 4-MeC_6H_4 (**b**), 3-MeC_6H_4 (**c**).

phenol (**VII**) was oxidized to *N*-benzoyl-6-azido-2-chloro-3,5-dimethyl-1,4-benzoquinone imine (**VIII**) which failed to undergo nucleophilic replacement of the halogen atom, regardless of the conditions (Scheme 2). *N*-Aroyl-2,6-dimethyl-1,4-benzoquinone imines do not react with HN_3; therefore, the reaction of *N*-aroyl-1,4-benzoquinone imines with HN_3 is regioselective: it follows only the 1,4-addition pattern.

By reactions of *N*-aroyl-2,3,5,6-tetrachloro-1,4-benzoquinone imines **IXa**–**IXc** with HN_3 both at room temperature and on heating we obtained 2,6-diazido-3,5-dichloro-1,4-benzoquinone (**X**) as the only product (Scheme 3). Presumably, the initial stage is 1,2-addition of hydrazoic acid molecule at the activated sterically strained C=N bond [10]; the subsequent migration of the azido group from C^4 to C^2 is accompanied by nucleophilic substitution of the 2-chlorine atom, as was shown in [4]. Nucleophilic substitution of the second chlorine atom occurs in a similar way to give *N*-aroyl-2,6-diazido-3,5-dichloro-1,4-benzoquinone imine, and the latter undergoes hydrolysis by the action of atmospheric moisture.

The reaction with a smaller amount of hydrazoic acid also afforded 2,6-diazido-3,5-dichloro-1,4-benzoquinone (**X**) but in a lower yield, and unreacted initial quinone imine **IX** was present in the reaction mixture; we failed to isolate any intermediate product. We succeeded in isolating a stable intermediate 1,2-adduct, 4-acetylamino-4-azido-2,3,5,6-tetrachlorocyclohexa-2,5-dien-1-one (**XII**) only in the reaction of HN_3 with *N*-acetyl-2,3,5,6-tetrachloro-1,4-benzoquinone monoimine (**XI**) (Scheme 4); this result provides one more support to our assumption that the reaction of HN_3 with *N*-acyl-1,4-benzoquinone imines begins with 1,2-addition.

Scheme 4.



XI **XII**

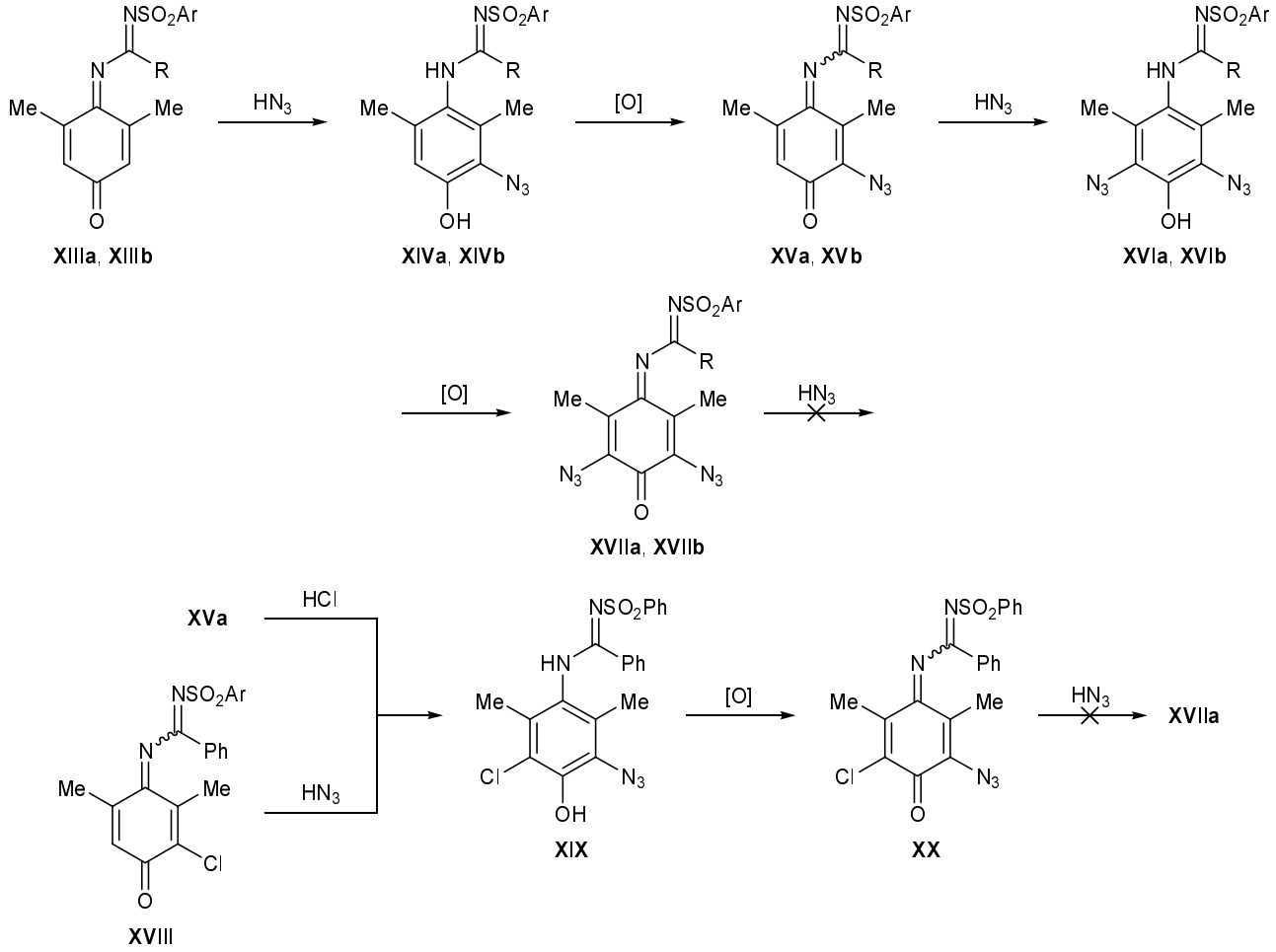
The structure of compounds **IIa**–**IIc**, **III**–**V**, **VII**, **VIII**, **X**, and **XII** was proved by the analytical data and IR and ^1H and ^{13}C NMR spectra. The IR spectra of azidophenols **IIa**–**IIc**, **IV**, and **VII** characteristically contained absorption bands due to stretching vibrations of the azido group at 2110–2180 cm^{-1} and those belonging to the C=O, NH, and OH groups in the regions 1650–1670, 3210–3290, and 3380–3490 cm^{-1}, respectively. Compound **XII** showed in the IR spectrum absorption bands corresponding to the acetyl and endocyclic carbonyl groups, azido group, and NH group at 1650, 1685, 2100, and 3180 cm^{-1}, respectively. In the ^{13}C NMR spectrum of **X** we observed two carbonyl carbon signals at δ_C 174.15 and 167.87 ppm and two signals from the carbon atoms attached to chlorine and azido group at δ_C 147.52 and 126.93 ppm, respectively. The ^{13}C NMR spectrum of **XII** contained signals from the carbonyl carbon atom (δ_C 169.41 ppm), sp^3-hybridized carbon atom (δ_C 75.34 ppm), C^2/C^6 (δ_C 143.51 ppm), C^2/C^5 (δ_C 133.09 ppm), and acetyl group (δ_C 17.38 and 169.17 ppm).

Repeated HN_3 addition–oxidation sequence with *N*-[*N*-Arylsulfonylbenz(acet)imidoyl]-3,5-dimethyl-1,4-benzoquinone imines **XIIIa** and **XIIIb** as substrates leads through intermediate products **XIV**–**XVI** to *N*-[*N*-arylsulfonylbenz(acet)imidoyl]-2,6-diazido-3,5-dimethyl-1,4-benzoquinone imines **XVIIa** and **XVIIb**, respectively (Scheme 5). Our attempt to synthesize compound **XVIIa** by independent method, from *N*-(*N*-phenylsulfonylbenzimidoyl)-2-chloro-3,5-dimethyl-1,4-benzoquinone imine (**XVIII**) (as it was reported for *N*-arylsulfonyl-1,4-benzoquinone imines [9]), was unsuccessful. *N*-Arylsulfonyl-2,6-diazido-3,5-dimethyl-1,4-benzoquinone imines were obtained from *N*-arylsulfonyl-2-chloro-3,5-dimethyl-1,4-benzoquinone imines via 1,4-addition of HN_3 molecule in the first stage and subsequent oxidation of the adduct and nucleophilic substitution of the halogen atom at elevated temperature [9].

The first stage in the reaction with benzoquinone imine **XVIII** is also 1,4-addition of HN_3 with formation of compound **XIX**. However, the oxidation of **XIX** gives benzoquinone imine **XX** in which the halogen atom is not replaced by the action of HN_3 (Scheme 5). This fact, as well as the inactivity of HN_3 with respect to *N*-[*N*-arylsulfonylbenz(acet)imidoyl]-2,3,5,6-tetrachloro-, -2,6-dichloro-3,5-dimethyl-, and -2,6-dimethyl-1,4-benzoquinone imines, indicates that *N*-[*N*-arylsulfonylbenz(acet)imidoyl]-1,4-benzoquinone imines, being structural analogs of *N*-aroyl-1,4-benzoquinone imines [10], are similar to the latter in chemical properties and that they differ considerably from *N*-arylsulfonyl-1,4-benzoquinone imines.

The structure of the newly synthesized compounds was proved by the analytical data and IR and ^1H NMR spectra; the structure of compound **XX** was also con-

Scheme 5.



Ar = R = Ph (**a**); Ar = 4-MeC_6H_4, R = Me (**b**).

firmed by the ^{13}C NMR data and independent synthesis. The IR spectra of *N*-[*N*-arylsulfonylbenz(acet)-imidoyl]-1,4-benzoquinone imines **XVa**, **XVb**, **XVIIa**, **XVIIb**, and **XX** having an azido group contained absorption bands in the regions 1630–1660 (C=O), 2100–2120 (N_3), and 1150–1180 cm^{-1} (SO_2). Compound **XX** displayed in the ^{13}C NMR spectrum signals from the carbonyl carbon atom (δ_C 174.25 ppm), quinone imine C=N fragment (δ_C 164.09 ppm), and benzimidoyl group (δ_C 153.16 ppm); also, six signals from aromatic carbon atoms attached to a proton (δ_C 133.89, 132.66, 129.20, 128.86, 128.35, and 126.96 ppm; the signals were assigned using the APT pulse sequence), two signals from aromatic carbon atoms attached to the SO_2 group and C=N carbon atom (δ_C 141.32, 131.16 ppm), four signals from carbon atoms in the quinoid ring (δ_C 143.46, 138.62, 137.09, 136.64 ppm), and two signals from methyl carbon atoms (δ_C 16.75, 13.75 ppm) were present.

EXPERIMENTAL

The IR spectra were recorded in KBr on a UR-20 spectrometer. The ^1H NMR spectra were measured on a Varian VXR-300 instrument (300 MHz) relative to tetramethylsilane; DMSO-d_6 (**IIa**, **IIc**), $CDCl_3$ (**III**, **V**, **VIII**, **XII**, **XVa**, **XVb**, **XVIIa**, **XVIIb**, **XX**), and acetone-d_6 (**XIVb**) were used as solvents. The ^{13}C NMR spectra were obtained on a Varian VXR-300 spectrometer at 75.4 MHz from solutions in hexamethylphosphoramide (**X**) and chloroform-*d* (**XII**, **XX**); the chemical shifts were measured relative to tetramethylsilane.

N-Aroyl-1,4-quinone imines **Ia**–**Ic** were synthesized according to the procedure described in [10]; compound **Ia**: yield 68%, mp 119–120°C. Found, %: N 5.27, 5.42. $C_{16}H_{15}NO_2$. Calculated, %: N 5.53; compound **Ib**: yield 72%, mp 115–116°C. Found, %: N 5.08, 5.17. $C_{15}H_{12}ClNO_2$. Calculated, %: N 5.12;

compound **Ic**: yield 75%, mp 117–118°C. Found, %: N 4.17, 4.25. $C_{15}H_{12}BrClNO_2$. Calculated, %: N 4.40.

Quinone imine **VI** was synthesized by hydrochlorination of *N*-benzoyl-3,5-dimethyl-1,4-benzoquinone imine (**XXI**) and subsequent oxidation according to the procedure described in [11]. Yield 75%, mp 97–99°C. Found, %: N 5.08, 5.19. $C_{15}H_6ClNO_2$. Calculated, %: N 5.23. Yield of **XXI** 63%, mp 80–81°C. Found, %: N 5.89, 5.94. $C_{15}H_{13}NO_2$. Calculated, %: N 5.86.

Quinone imines **XIIIa**, **XIIIb**, **XVIII**, **IXa**–**IXc**, and **XI** were prepared as described in [10–12].

Reaction of *N*-aroyl-1,4-benzoquinone imines Ia–Ic, III, VI, IXa–IXc, and XI and *N*-[*N*-arylsulfonylbenz(acet)imidoyl]-1,4-benzoquinone imines XIIIa, XIIIb, XVa, XVb, and XVIII with hydrazoic acid (*general procedure*). Sodium azide, 0.007 mol, was added to a solution of 0.005 mol of quinone imine **Ia**–**Ic**, **III**, **VI**, **IXa**–**IXc**, **XIIIa**, **XIIIb**, **XVa**, **XVb**, or **XVIII** in 10 ml of acetic acid. The mixture was stirred for 1 h at room temperature, and it turned dark red. The precipitate was filtered off, washed with water, dried in air, and recrystallized from benzene. If no solid separated from the solution, a few milliliters of water were added to the reaction mixture.

Compound **IIa**. Yield 89%, mp 145–146°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 2.01 s (3H, 5-Me), 2.06 s (3H, 3-Me), 2.37 s (3H, C**H**$_3$C$_6$H$_4$), 6.65 s (1H, 6-H), 7.30–7.89 d.d (4H, C$_6$H$_4$), 9.54 s (1H, NH), 10.17 s (1H, OH). Found, %: C 64.46, 64.91; H 5.36, 5.48. $C_{16}H_{16}N_4O_2$. Calculated, %: C 64.85; H 5.44.

Compound **IIb**. Yield 91%, mp 136–137°C (decomp.). Found, %: C 56.71, 56.80; H 3.95, 4.10. $C_{15}H_{13}ClN_4O_2$. Calculated, %: C 56.88; H 4.14.

Compound **IIc**. Yield 87%, mp 142–143°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 2.01 s (3H, 5-Me), 2.06 s (3H, 3-Me), 6.66 s (1H, 6-H), 7.72–7.91 d.d (4H, C$_6$H$_4$), 9.71 s (1H, NH), 10.69 s (1H, OH). Found, %: C 49.75, 49.83; H 3.41, 3.54. $C_{15}H_{13}BrN_4O_2$. Calculated, %: C 49.88; H 3.63.

Compound **IV**. Yield 82%, mp 136–137°C (decomp.). Found, %: C 56.99, 60.15; H 4.40, 4.59. $C_{16}H_{15}N_7O_2$. Calculated, %: C 56.97; H 4.45.

Compound **VII**. Yield 87%, mp 143–144°C (decomp.). Found, %: C 56.69, 56.78; H 4.05, 4.13. $C_{15}H_{13}ClN_7O_2$. Calculated, %: C 56.87; H 4.11.

Compound **X**. Yield 78–81%, mp 153–154°C. Found, %: C 27.64, 27.85; Cl 27.18, 27.62. $C_6Cl_2N_6O_2$. Calculated, %: C 27.82; Cl 27.37.

Compound **XII**. Yield 73%, mp 119–120°C. ^{1}H NMR spectrum, δ, ppm: 2.04 s (3H, Me), 6.17 s (1H, NH). Found, %: C 29.05, 29.15; H 1.03, 1.14. $C_8H_{14}Cl_4N_4O_2$. Calculated, %: C 29.12; H 1.22.

Compound **XIVa**. Yield 68%, mp 177–180°C (decomp.). Found, %: C 59.61, 59.78; H 4.29, 4.41. $C_{21}H_{19}N_5O_3S$. Calculated, %: C 59.84; H 4.54.

Compound **XIVb**. Yield 81%, mp 168–170°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 2.01 s (3H, 5-Me), 2.05 s (3H, 3-Me), 2.41 s (3H, **Me**C$_6$H$_4$), 2.58 s (3H, MeC=N), 3.04 br.s (1H, OH), 6.68 s (1H, 6-H), 7.50–7.73 d.d (4H, C$_6$H$_4$), 9.05 br.s (1H, NH). Found, %: C 54.51, 54.62; H 4.93, 5.01. $C_{17}H_{19}N_5O_3S$. Calculated, %: C 54.68; H 5.13.

Compound **XVIa**. Yield 64%, mp 172–173°C (decomp.). Found, %: C 54.31, 54.42; H 3.60, 4.01. $C_{21}H_{18}N_8O_3S$. Calculated, %: C 54.54; H 3.92.

Compound **XVIb**. Yield 59%, mp 120–121°C (decomp.). Found, %: C 49.10, 49.15; H 4.40, 4.49. $C_{17}H_{18}N_8O_3S$. Calculated, %: C 49.27; H 4.38.

Compound **XIX**. Yield 91%, mp 168–169°C (decomp.). Found, %: C 54.92, 55.29; H 3.75, 3.89. $C_{21}H_{18}ClN_5O_3S$. Calculated, %: C 55.32; H 3.98.

Oxidation of aminophenols IIa–IIc, IV, VII, XIVa, XIVb, XVIa, XVIb, and XIX (*general procedure*). Lead tetraacetate, 0.0044 mol, was added under stirring at room temperature to a suspension of 0.004 mol of aminophenol **IIa**–**IIc**, **IV**, **VII**, **XIVa**, **XIVb**, **XVIa**, **XVIb**, or **XIX** in 7 ml of acetic acid. When the exothermic reaction was complete, the mixture was cooled, and a red solid precipitated. A few drops of ethylene glycol was added, the mixture was stirred for 5 min, and the precipitate was filtered off, washed with methanol, dried in air, and recrystallized from acetic acid.

Compound **III**. Yield 63%, mp 102–104°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 2.12 s (3H, 5-Me), 2.43 s (3H, 3-Me), 2.43 s (3H, **Me**C$_6$H$_4$), 6.47 s (1H, 6-H), 7.26–7.72 d.d (4H, C$_6$H$_4$). Found, %: C 65.01, 65.28; H 4.62, 4.74. $C_{16}H_{14}N_4O_2$. Calculated, %: C 65.31; H 4.76.

Compound **V**. Yield 74%, mp 142–143°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 2.02 s (3H, 3-Me), 2.02 s (3H, 5-Me), 2.42 s (3H, **Me**C$_6$H$_4$), 7.26–7.69 d.d (4H, C$_6$H$_4$). Found, %: C 57.12, 57.37; H 3.84, 3.96. $C_{16}H_{13}N_7O_2$. Calculated, %: C 57.31; H 3.88.

Compound **VIII**. Yield 71%, mp 138–139°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 1.98 s (3H,

5-Me), 2.23 s (3H, 3-Me), 7.48–7.85 m (5H, C_6H_5). Found, %: C 56.97, 57.11; H 3.38, 3.47. $C_{15}H_{11}ClN_4O_2$. Calculated, %: C 57.23; H 3.50.

Compound **XVa**. Yield 79%, mp 154–155°C. ^{1}H NMR spectrum, δ, ppm: 1.94 s (3H, 5-Me), 2.09 s (3H, 3-Me), 6.51 s (1H, 6-H), 7.43–7.78 m (5H, C_6H_5C=N), 7.51–7.98 m (5H, $C_6H_5SO_2$). Found, %: C 60.01, 60.19; H 3.86, 3.98. $C_{21}H_{17}N_5O_3S$. Calculated, %: C 60.13; H 4.09.

Compound **XVb**. Yield 83%, mp 142–143°C. ^{1}H NMR spectrum, δ, ppm: 1.99 s (3H, 5-Me), 2.13 s (3H, 3-Me), 2.34 s (3H, MeC=N), 2.42 s (3H, **Me**C_6H_4), 6.28–7.77 d.d (4H, C_6H_4), 6.47 s (1H, 6-H). Found, %: C 54.65, 54.80; H 4.32, 4.70. $C_{17}H_{17}N_5O_3S$. Calculated, %: C 54.98; H 4.61.

Compound **XVIIa**. Yield 86%, mp 143–145°C. ^{1}H NMR spectrum, δ, ppm: 1.97 s (3H, 3-Me), 1.97 s (3H, 5-Me), 7.41–7.75 m (5H, C_6H_5C=N), 7.50–7.96 m (5H, $C_6H_5SO_2$). Found, %: C 54.18, 54.13; H 3.39, 3.52. $C_{21}H_{16}N_8O_3S$. Calculated, %: C 54.78; H 3.50.

Compound **XVIIb**. Yield 83%, mp 141–142°C. ^{1}H NMR spectrum, δ, ppm: 2.01 s (3H, 3-Me), 2.01 s (3H, 5-Me), 2.31 s (3H, MeC=N), 2.42 s (3H, **Me**C_6H_4), 7.27–7.76 d.d (4H, C_6H_4). Found, %: C 49.15, 49.28; H 3.86, 4.02. $C_{17}H_{16}N_8O_3S$. Calculated, %: C 49.51; H 3.91.

Compound **XX**. Yield 85%, mp 154–155°C. ^{1}H NMR spectrum, δ, ppm: 1.96 s (3H, 5-Me), 2.26 s (3H, 3-Me), 7.42–7.76 m (5H, C_6H_5C=N), 7.52–7.98 m (5H, $C_6H_5SO_2$). Found, %: C 55.37, 55.59; H 3.43, 3.60. $C_{21}H_{16}ClN_5O_3S$. Calculated, %: C 55.57; H 3.55.

***N*-(*N*-Phenylsulfonylbenzimidoyl)-4-amino-2-azido-6-chloro-3,5-dimethylphenol (XIX).** A stream of gaseous hydrogen chloride was passed through a solution of 0.07 mol of *N*-(*N*-phenylsulfonylbenzimidoyl)-2-azido-3,5-dimethyl-1,4-benzoquinone imine (**XVa**) in 30 ml of anhydrous chloroform until the mixture turned colorless and a solid precipitated. The precipitate was filtered off, washed with chloroform, and recrystallized from benzene. The product was identical to a sample obtained as described above from quinone imine **XVIII**.

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