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Thiocyanation of *N*-Arylsulfonyl-, *N*-Aroyl-, and *N*-[(*N*-Arylsulfonyl)benzimidoyl]-1,4-benzoquinone Imines

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Abstract—Reactions of thiocyanate ion with *N*-aroyl-, *N*-arylsulfonyl-, and *N*-(*N*-arylsulfonylbenzimidoyl)-1,4-benzoquinone imines follow the 1,4-addition pattern, and the adducts undergo intramolecular cyclization to give the corresponding N-substituted 5-amino-1,3-benzoxathiol-2-ones as final products.

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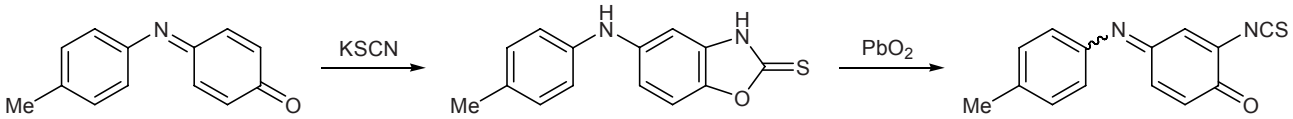
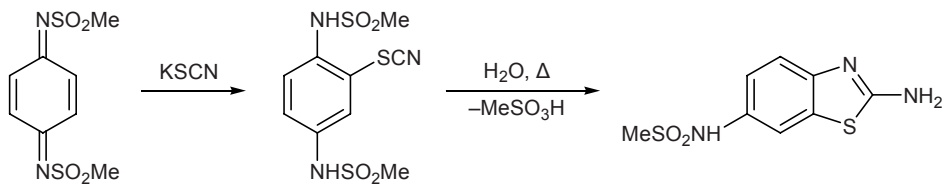
Thiocyanation of N-substituted *para*-quinone imines was the subject of a few publications [1, 2], and specificity of this reaction remains so far poorly studied. Thiocyanate ion $[NCS]^-$ is a soft ambident nucleophile possessing two reaction centers, nitrogen and sulfur atoms. Nucleophilic addition at the nitrogen atom gives the corresponding isothiocyanates (–N=C=S), while the addition involving the sulfur atom produces thiocyanates (–S–C≡N) [3]. The sulfur atom is known to be a stronger nucleophilic center than nitrogen. In most cases, addition reactions of thiocyanate ion involve the sulfur atom with formation of thiocyanates [4]. Depending on the product structure and reaction conditions, isomerization into the corresponding thermodynamically more stable isothiocyanate is possible.

The reaction of *N*-(4-methylphenyl)-1,4-benzoquinone imine with potassium and ammonium thiocyanates was reported to yield 5-(4-methylphenylamino)-1,3-benzoxazole-2-thione [1] (Scheme 1). The authors presumed that the first step in this process is addition of thiocyanate ion as nitrogen-centered nucleophile at the *ortho* position with respect to the carbonyl group. An indirect support for the proposed reaction path was opening of the five-membered heteroring in the product by the action of lead(IV) oxide as oxidant with formation of 2-isothiocyanato-*N*-(4-methylphenyl)-1,4-benzoquinone imine. Nucleophilic replacement of the chlorine atom in 2-chloro-*N*-(4-methylphenyl)-1,4-benzoquinone imine gave the corresponding thiocyanate, *N*-(4-methylphenyl)-2-thiocyanato-1,4-benzoquinone imine, which did not undergo intramolecular

Scheme 1.



Scheme 2.

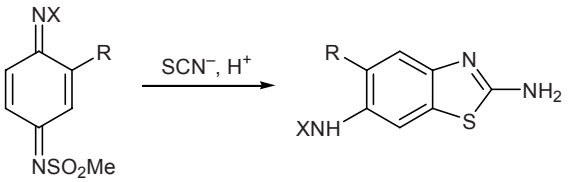


cyclization [1]. In this case, thiocyanate ion reacted as sulfur-centered nucleophile. Analogous reaction was reported for *N*,*N*′-bis(methylsulfonyl)-1,4-benzoquinone diimine [2]. Here, the product was *N*,*N*′-(2-thiocyanatobenzene-1,4-diyl)bis(methanesulfonamide) which was converted into *N*-(2-amino-1,3-benzothiazol-6-yl)sulfonamide upon subsequent aqueous treatment (Scheme 2).

The formation of *N*,*N*′-(2-thiocyanatobenzene-1,4-diyl)bis(methanesulfonamide) was confirmed by the IR data, namely by the presence in the IR spectrum of an absorption band at 2165 cm^{-1}, which is typical of thiocyanato group. We believe that the cyclization of *N*,*N*′-(2-thiocyanatobenzene-1,4-diyl)bis(methanesulfonamide) occurs with participation of water molecule and elimination of methanesulfonic acid and is accompanied by cleavage of the S–N bond in the quinone diimine. The latter process is fairly rarely observed in reactions of *N*-sulfonyl-1,4-quinone imines. In particular, analogous bond cleavage was reported in [5].

Unsymmetrically substituted 1,4-benzoquinone diimines, such as *N*-*tert*-butylcarbonyl-*N*′-methylsulfonyl-1,4-benzoquinone diimine and *N*-methoxycarbonyl-*N*′-methylsulfonyl-1,4-benzoquinone diimine reacted with thiocyanate ion to give the corresponding N^6-substituted 1,3-benzothiazole-2,6-diamines as a result of elimination of methylsulfonyl group [2] (Scheme 3). If a methyl or methoxy group was present in the *meta* position with respect to the methylsulfonylimino group, no addition of thiocyanate ion was observed, but the initial quinone diimine was reduced to the corresponding *p*-phenylenediamine. This pattern was rationalized in [2] in terms of steric hindrances. However, we believe that the R substituent cannot affect attack by thiocyanate ion on the quinoid ring at position *5*.

Scheme 3.



R = H, Me, MeO; X = *t*-BuCO. MeOCO.

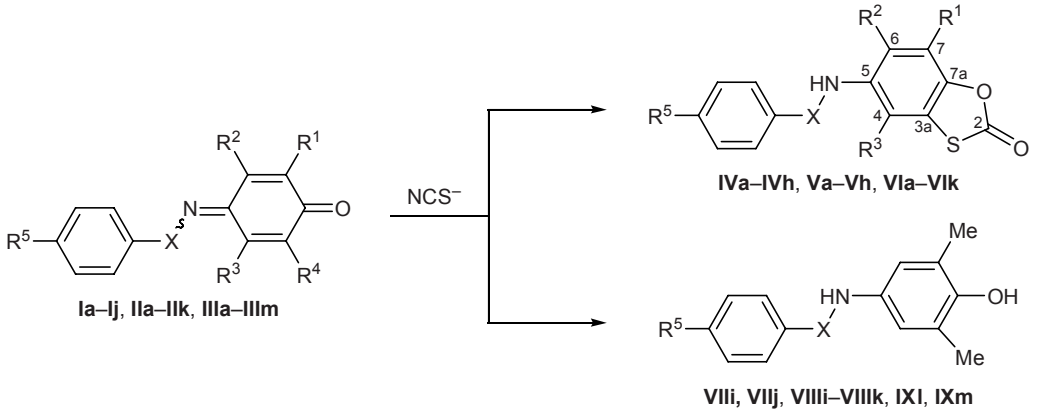
Taking into account that in the above nucleophilic addition reactions with various quinone imines thiocyanate ion acted as both nitrogen- and sulfur-centered nucleophile, it was interesting to elucidate factors determining the reactivity of thiocyanate ion. For this purpose, we examined reactions of thiocyanate ion with other *N*-substituted 1,4-benzoquinone imines. We presumed that convenient substrates could be *N*-arylsulfonyl-, *N*-aroyl-, and *N*-(*N*-arylsulfonylbenzimidoyl)-1,4-benzoquinone imines which are characterized by high reactivity and relatively high stability. Their redox potentials are greater than those of *N*-aryl-1,4-benzoquinone imines but lower than the corresponding values of analogous 1,4-benzoquinone diimines.

The reactions of *N*-arylsulfonyl-, *N*-aroyl-, and *N*-(*N*-arylsulfonylbenzimidoyl)-1,4-benzoquinone imines **Ia**–**Ij**, **IIa**–**IIk**, and **IIIa**–**IIIm** with potassium or ammonium thiocyanate were carried out in acetic acid. To obtain more detailed data, both unsubstituted quinone imines and those containing alkyl groups (Me, *i*-Pr) in different positions of the quinoid ring were involved. The results showed that most quinone imines, except for 2,6-disubstituted derivatives, reacted with thiocyanate ion according to a common scheme to give *N*-substituted 5-amino-1,3-benzoxathiol-2-ones **IV**–**VI**. Under the same conditions, 2,6-disubstituted 1,4-benzoquinone imines were reduced to the corresponding aminophenols **VII**–**IX**. These findings indicate that the reaction of thiocyanate ion with 1,4-benzoquinone imines is strictly regioselective and that it follows the 1,4-addition pattern (Scheme 4).

N-Substituted 5-amino-1,3-benzoxathiol-2-ones **IV**–**VI** were isolated as colorless crystalline substances. Their structure was confirmed by elemental analysis and IR and ^1H and ^{13}C NMR spectroscopy. The IR spectra of **IV**–**VI** contained a strong carbonyl absorption band in the region 1750–1810 cm^{-1}, while bands typical of initial quinone imines (ν C=O, 1640–1690 cm^{-1}, and ν C=N, 1580–1600 cm^{-1}) were absent. No signals assignable to NH_2 group were observed in the ^1H NMR spectra of **IV**–**VI**; this means that the products are not 2-aminobenzothiazole derivatives like those described in [2].

The structure of 5-amino-1,3-benzoxathiol-2-ones **IV**–**VI** is also consistent with the ^{13}C NMR spectra recorded for compounds **IVd**, **IVe**, **Vb**, and **Vg**). In particular, the ^{13}C NMR spectrum of **IVe** lacked signals from carbonyl and imino carbon atoms (δ_C 186.1 and 164.7 ppm, respectively, in the spectrum of the initial quinone imine). The number of signals in the aromatic region of the spectra of **IV** and **V** was larger by unity, as compared to the initial compounds, while one CH signal disappeared. In the ^{13}C NMR spectrum of 3,5-dimethyl-substituted benzoquinone imine **Ie**, carbon nuclei in positions *3*/*5* and *2*/*6* were magnet-

Scheme 4.



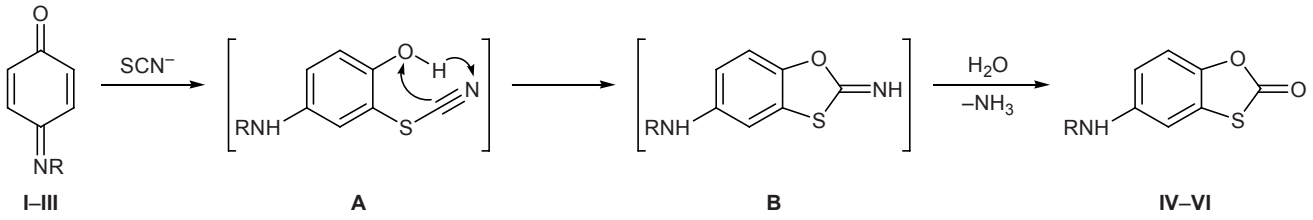
I, **IV**, **VII**, X = SO_2, $R^1 = R^2 = R^3 = R^4 = H$ (**a**); R^1 = Me, $R^2 = R^3 = R^4 = H$ (**b**); $R^1 = R^3$ = Me, $R^2 = R^4 = H$ (**c**, **d**); $R^1 = R^4 = H$, $R^2 = R^3$ = Me (**e**, **f**); $R^1 = R^2$ = Me, $R^3 = R^4 = H$ (**g**, **h**); $R^1 = R^4$ = Me, $R^2 = R^3 = H$ (**i**, **j**); R^5 = Me (**a**, **c**, **e**, **h**, **i**), H (**b**, **g**), MeO (**d**), Cl (**f**, **j**); **II**, **V**, **VIII**, X = CO, $R^1 = R^3$ = Me, $R^2 = R^4 = H$ (**a**–**c**); $R^1 = R^4 = H$, $R^2 = R^3$ = Me (**d**–**f**); $R^1 = R^2$ = Me, $R^3 = R^4 = H$ (**g**, **h**); $R^1 = R^4$ = Me, $R^2 = R^3 = H$ (**i**–**k**); R^5 = Me (**a**, **d**, **g**, **i**), MeO (**b**, **e**, **j**), Cl (**c**, **f**, **h**, **k**); **III**, **VI**, **IX**, X = C(=NSO_2Ar), Ar = 4-$MeOC_6H_4$ (**a**, **j**), 4-MeC_6H_4 (**b**, **d**–**g**, **i**, **l**), Ar = Ph (**c**), 4-ClC_6H_4 (**h**, **k**, **m**); $R^1 = R^2 = R^3 = R^4 = H$ (**a**); R^1 = Me, $R^2 = R^3 = R^4 = H$ (**b**); $R^1 = R^3$ = Me, $R^2 = R^4 = H$ (**c**, **d**); R^1 = *i*-Pr, $R^2 = R^4 = H$, R^3 = Me (**e**); R^1 = Me, $R^2 = R^4 = H$, R^3 = *i*-Pr (**f**); $R^1 = R^4 = H$, $R^2 = R^3$ = Me (**g**, **h**); $R^1 = R^2$ = Me, $R^3 = R^4 = H$ (**i**–**k**); $R^1 = R^4$ = Me, $R^2 = R^3 = H$ (**l**, **m**); R^5 = H.

ically equivalent due to fast (on the NMR time scale) *Z*–*E* isomerization, whereas compound **IVe** displayed separate signals from the above carbon atoms. These data indicate aromatization of the quinoid fragment and addition of a carbon-containing substituent to position *2*(*6*) of the quinoid ring. The chemical shifts of carbon nuclei in the arylsulfonyl fragment remain almost the same as in the spectrum of initial 1,4-benzoquinone imine **Ie** (δ_C, ppm: **Ie**: 139.44, 126.56, 129.53, 143.69; **IVe**: 138.60, 126.58, 129.82, 143.22). Characteristically, two signals appeared in the ^{13}C NMR spectra of **IV**–**VI** in the region corresponding to sp^2-hybridized carbon atoms. The upfield signal (δ_C 111–120 ppm) was assigned to carbon atoms in the benzene ring in the *ortho* position with respect to the oxygen atom, and the downfield signal (δ_C 167–169 ppm) corresponds to the newly formed carbonyl group. The ^{13}C NMR spectra recorded without decoupling from protons, as well as ^1H–^{13}C heteronuclear correlation spectra (HETCOR and LR HETCOR), were also consistent with the assumed structure of compounds **IV**–**VI**.

Thus the NMR data for the initial compounds and addition products showed that thiocyanate ion reacts with quinone imines **I**–**III** as sulfur-centered nucleophile and that the addition is followed by intramolecular cyclization to N-substituted 5-amino-1,3-benzoxathiol-2-ones **IV**–**VI**. A plausible mechanism of the process is illustrated by Scheme 5. Presumably, in the initial step 1,4-addition of thiocyanate ion gives N-substituted 4-amino-2-thiocyanatophenol **A**. Intramolecular ring closure occurs as a result of electrophilic attack by the carbon atom of the thiocyanato group on the hydroxy oxygen atom and proton transfer to the nitrogen atom. Intermediate 5-amino-1,3-benzoxathiol-2-imine **B** undergoes hydrolysis with formation of final 5-amino-1,3-benzoxathiol-2-one **IV**–**VI** and liberation of ammonia. In fact, addition of alkali to the filtrate obtained after separation of the product and subsequent heating resulted in evolution of an equimolar amount of ammonia. The latter was separated by steam distillation into a receiver charged with distilled water, and the distillate was titrated with 0.1 N hydrochloric acid (the amount of liberated ammonia was determined only in experiments with potassium thiocyanate).

The crystalline and molecular structure of 4,7-dimethyl-5-(*p*-tolylsulfonylamino)-1,3-benzoxathiol-2-one (**IVc**) was determined by X-ray analysis (see figure). Molecule **IVc** is composed of two planar fragments. The first of these includes the *p*-tolyl substituent and S^1, and the second consists of the benzoxathiol ring system and O^4 and N^1 atoms attached thereto. The two fragments are almost orthogonal to each other: the corresponding dihedral angle is 106.0°. Such orientation makes conjugation between the benzene and benzoxathiol fragments impossible, and the C^8–N^1, N^1–S^1, and S^1–C^5 bonds [1.471(3), 1.673(2), and 1.804(3) Å, respectively] are appreciably longer than analogous standard bonds (1.390, 1.633, and 1.758 Å, respectively) [6]. Molecules **IVc** in crystal

Scheme 5.



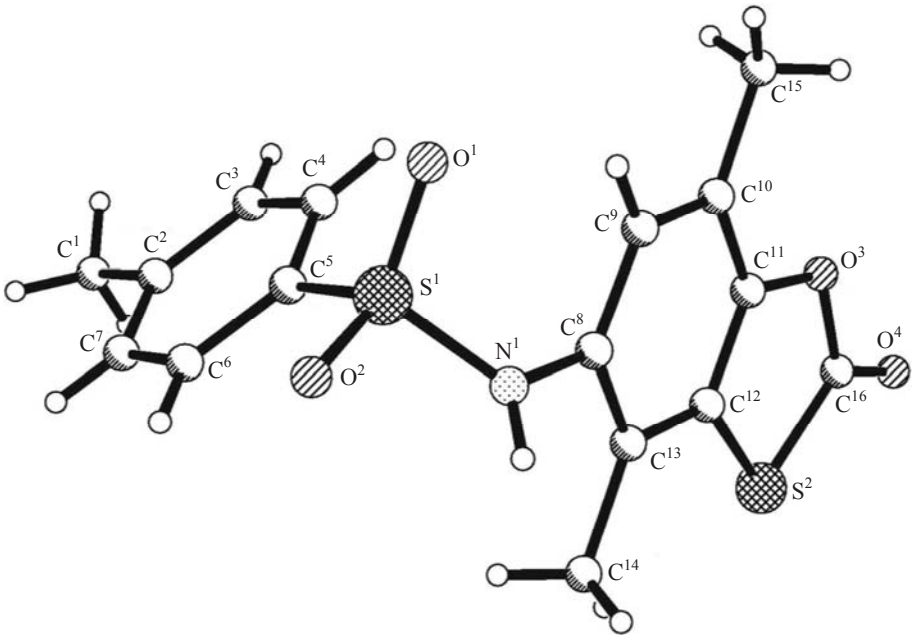
form centrosymmetric dimers via weak intermolecular hydrogen bonds $N^1–H^{1A}\cdots O^{2'}$ (1.5 – *x*, 0.5 – *y*, –*z*; N···O 2.41 Å, ∠NHO 145°). In addition, short intermolecular contact $S^2\cdots S^{2'}$ (2 – *x*, – *y*, 1 – *x*; 3.28 Å, sum of the corresponding van der Waals radii 3.68 Å [7]) was detected in crystal.

In all the examined reactions, thiocyanation of *N*-substituted 1,4-benzoquinone imines **Ia**–**Ij**, **IIa**–**IIk**, and **IIIa**–**IIIm** followed a pattern different from that reported in [1] for thiocyanation of *N*-(4-methylphenyl)-1,4-benzoquinone imine. The substituent on the nitrogen atom in *N*-aryl-1,4-benzoquinone imines is a weak electron acceptor, and the redox potentials of such quinone imines [8] are lower than those of *N*-arylsulfonyl-, *N*-aroyl-, and *N*-(*N*-arylsulfonylbenzimidoyl)-1,4-benzoquinone imines [9]. Moreover, *N*-aryl-1,4-benzoquinone imines are the strongest electrophiles among the examined quinone imines. In most cases, the structure of thiocyanation products (thiocyanato or isothiocyanato derivatives) depends on the rate of thiocyanate–isothiocyanate isomerization which is determined in turn by the substrate structure and reaction conditions. The isomerization is strongly favored by increased electrophilicity of the substrate [4].

Thus our results, in combination with the data of [4], demonstrated that thiocyanate ion acts as sulfur-centered nucleophile toward all the examined quinone imines. The reaction with *N*-aryl-1,4-benzoquinone imines may be accompanied by isomerization of initially formed thiocyanate into the corresponding isothiocyanate. The rate of isomerization of thiocyanates derived from N-substituted 1,4-benzoquinone imines **I**–**III** is considerably lower than the rate of their cyclization into benzoxathiole derivatives **IV**–**VI**.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded from solutions in DMSO-d_6 or acetone-d_6 (compound **IVc**) on a Varian VXR-300 spectrometer (300 MHz); the



Structure of the molecule of *N*-(4,7-dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methylbenzenesulfonamide (**IVc**) according to the X-ray diffraction data.

chemical shifts were determined relative to tetramethylsilane. The IR spectra were measured in KBr on a UR-20 instrument. Analytical thin-layer chromatography was performed on Silufol UV-254 plates; samples were applied from solutions in chloroform, plates were eluted with benzene–hexane (10 : 1), and spots were visualized under UV light.

X-Ray analysis of a single crystal of compound **IVc** was performed at 293 K on an Xcalibur-3 diffractometer (MoK_α irradiation, CCD detector, graphite monochromator, ω-scanning, $2\theta_{max} = 50°$). Monoclinic crystals, $C_{16}H_{15}NO_4S_2$, with the following unit cell parameters (293 K): a = 19.527(4), b = 18.949(4), c = 9.625(4) Å; β = 90.71(4)°; V = 3560.9(19) Å^3; M_r = 349.41; Z = 8; space group $C2/c$; d_{calc} = 1.304 g/cm^3; μ(MoK_α) = 0.316 mm^{-1}; $F(000)$ = 1456. Total of 16748 reflections were measured, 3127 of which were independent (R_{int} = 0.041). The structure was solved by the direct method using SHELXTL software package [10]. The positions of hydrogen atoms were determined by dif-ference synthesis of electron density and were refined according to the riding model ($U_{iso} = n U_{eq}$ for non-hydrogen atom to which the given hydrogen atom is attached; n = 1.5 for methyl group, n = 1.2 for other hydrogen atoms). The structure was refined with respect to F^2 by full-matrix least-squares procedure in anisotropic approximation for non-hydrogen atoms to wR_2 = 0.108 (16384 reflections) and R_1 = 0.040 [1978 reflections with $F > 4\sigma(F)$, S = 0.968]. The complete set of crystallographic data, including coordinates of atoms, was deposited to the Cambridge Crystallographic Data Centre (entry no. CCDC 681168).

Quinone imines **Ia**–**Id** and **Ig**–**Ij** were synthesized as described in [11] by oxidation of the corresponding aminophenols with sodium dichromate in acetic acid. Quinone imines **Ie**, **If**, **IIa**–**IIk**, and **IIIa**–**IIIm** were synthesized according to the procedure reported in [12] by oxidation of the corresponding aminophenols with lead tetraacetate in acetic acid. The properties of compounds **Ia** [11], **Ib** [13], **Ic** [14], **Id**, **IIb**, **IIc**, **IIId**–**IIIf** [15], **Ie** [16], **Ig**, **Ih** [17], **Ii**, **Ij** [18], **IIa** [19], **IId**, **IIi** [20], **IIg** [21], **IIIg**, **IIIh**, **IIIl**, and **IIIm** [22] were reported previously.

4-Chloro-*N*-(2,6-dimethyl-4-oxocyclohexa-2,5-dien-1-ylidene)benzenesulfonamide (If). Yield 64%, mp 105–106°C. Found, %: N 4.28, 4.49; S 10.01, 10.54. $C_{14}H_{12}ClNO_3S$. Calculated, %: N 4.52; S 10.35.

***N*-(2,6-Dimethyl-4-oxocyclohexa-2,5-dien-1-ylidene)-4-methoxybenzamide (IIe).** Yield 40%, mp 106–107°C. Found, %: N 5.17, 5.36. $C_{16}H_{15}NO_3$. Calculated, %: N 5.20.

4-Chloro-*N*-(2,6-dimethyl-4-oxocyclohexa-2,5-dien-1-ylidene)benzamide (IIf). Yield 44%, mp 88–89°C. Found, %: N 5.15, 5.38. $C_{15}H_{12}ClNO_2$. Calculated, %: N 5.12.

4-Chloro-*N*-(2,3-dimethyl-4-oxocyclohexa-2,5-dien-1-ylidene)benzamide (IIh). Yield 51%, mp 130–131°C. Found, %: N 5.18, 4.96. $C_{15}H_{12}ClNO_2$. Calculated, %: N 5.12.

***N*-(3,5-Dimethyl-4-oxocyclohexa-2,5-dien-1-ylidene)-4-methoxybenzamide (IIj).** Yield 31%, mp 97.5–99°C. Found, %: N 5.29, 5.35. $C_{16}H_{15}NO_3$. Calculated, %: N 5.20.

4-Chloro-*N*-(3,5-dimethyl-4-oxocyclohexa-2,5-dien-1-ylidene)benzamide (IIk). Yield 56%, mp 119–121°C. Found, %: N 5.05, 4.91. $C_{15}H_{12}ClNO_2$. Calculated, %: N 5.12.

***N*-(Cyclohexa-2,5-dien-1-ylidene)-*N′*-(4-methoxyphenylsulfonyl)benzimidamide (IIIa).** Yield 67%, mp 116–117°C. Found, %: N 7.40, 7.17; S 8.19, 8.30. $C_{20}H_{16}N_2O_4S$. Calculated, %: N 7.36; S 8.43.

***N*-(3-Methylcyclohexa-2,5-dien-1-ylidene)-*N′*-(4-methylphenylsulfonyl)benzimidamide (IIIb).** Yield 47%, mp 104–105°C. Found, %: N 7.21, 7.29; S 8.66, 8.38. $C_{21}H_{18}N_2O_3S$. Calculated, %: N 7.40; S 8.44.

***N*-(2,5-Dimethylcyclohexa-2,5-dien-1-ylidene)-*N′*-(phenylsulfonyl)benzimidamide (IIIc).** Yield 80%, mp 120–121°C. Found, %: N 7.24, 7.57; S 8.19, 8.54. $C_{21}H_{18}N_2O_3S$. Calculated, %: N 7.40; S 8.47.

***N*-(2,3-Dimethylcyclohexa-2,5-dien-1-ylidene)-*N′*-(4-methylphenylsulfonyl)benzimidamide (IIIi).** Yield 86%, mp 146–147°C. Found, %: N 6.95, 7.23; S 8.47, 8.20. $C_{22}H_{20}N_2O_3S$. Calculated, %: N 7.14; S 8.17.

***N*-(2,3-Dimethylcyclohexa-2,5-dien-1-ylidene)-*N′*-(4-methoxyphenylsulfonyl)benzimidamide (IIIj).** Yield 85%, mp 150.5–152°C. Found, %: N 7.00, 7.08; S 7.59, 7.77. $C_{22}H_{20}N_2O_4S$. Calculated, %: N 6.86; S 7.85.

***N′*-(4-Chlorophenylsulfonyl)-*N*-(2,3-dimethylcyclohexa-2,5-dien-1-ylidene)benzimidamide (IIIk).** Yield 79%, mp 160–161°C. Found, %: N 6.85, 6.87; S 7.90, 8.12. $C_{21}H_{17}ClN_2O_3S$. Calculated, %: N 6.78; S 7.77.

Reaction of quinone imines I–III with potassium or ammonium thiocyanate (*general procedure*). Potassium or ammonium thiocyanate, 4 mmol, was added to a solution of 2 mmol of quinone imine **I**–**III** in 15 ml of glacial acetic acid, the mixture was stirred,

left to stand for 12 h, and diluted with water until complete precipitation of the product. The precipitate was filtered off, washed with water, and recrystallized from acetic acid.

4-Methyl-*N*-(2-oxo-1,3-benzoxathiol-5-yl)benzenesulfonamide (IVa). Yield 52%, mp 177–178°C. ^{1}H NMR spectrum, δ, ppm: 2.34 s (3H, CH_3), 7.46 d (1H, 7-H, J = 7.8 Hz), 7.35–7.65 d.d (4H, $C_6\mathbf{H}_4CH_3$, J = 7.8 Hz), 7.53–7.56 d.d (1H, 6-H, J = 7.8, 1.5 Hz), 7.55 d (1H, 4-H, J = 1.5 Hz), 10.39 br.s (1H, NH). Found, %: N 4.26, 4.39; S 19.76, 20.04. $C_{14}H_{11}NO_4S_2$. Calculated, %: N 4.36; S 19.95.

***N*-(7-Methyl-2-oxo-1,3-benzoxathiol-5-yl)benzenesulfonamide (IVb).** Yield 66%, mp 184–185°C. ^{1}H NMR spectrum, δ, ppm: 2.23 s (3H, CH_3), 6.92 d (1H, 6-H, J = 1.5 Hz), 7.36 d (1H, 4-H, J = 1.8 Hz), 7.54–7.80 m (5H, Ph), 10.44 br.s (1H, NH). Found, %: N 4.14, 4.26; S 19.70, 20.05. $C_{14}H_{11}NO_4S_2$. Calculated, %: N 4.36; S 19.95.

***N*-(4,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methylbenzenesulfonamide (IVc).** Yield 82%, mp 157.5–158°C. ^{1}H NMR spectrum, δ, ppm: 2.04 s and 2.25 s (3H each, 4-CH_3, 7-CH_3), 2.41 s (3H, $C\mathbf{H}_3C_6H_4$), 6.93 s (1H, 6-H), 7.37–7.58 d.d (4H, C_6H_4, J = 8.1 Hz), 8.57 br.s (1H, NH). Found, %: N 3.72, 3.85; S 18.13, 18.58. $C_{16}H_{15}NO_4S_2$. Calculated, %: N 4.01; S 18.35.

***N*-(4,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methoxybenzenesulfonamide (IVd).** Yield 78%, mp 153.5–155°C. ^{1}H NMR spectrum, δ, ppm: 1.93 s and 2.22 s (3H each, 4-CH_3, 7-CH_3), 3.82 s (3H, MeO), 6.83 s (1H, 6-H), 7.08–7.56 d.d (4H, C_6H_4, J = 9.0 Hz), 9.62 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 14.89 and 16.16 (4-CH_3, 7-CH_3), 55.45 (MeO), 113.94 ($C^{3'}$), 119.18 (C^7), 122.55 (C^{3a}), 126.52 (C^5), 127.68 (C^6), 128. 46 ($C^{2'}$), 131.37 ($C^{1'}$), 131.45 ($C^{4'}$), 143.76 (C^{7a}), 162.05 ($C^{4'}$), 167.88 (C^2). Found, %: N 3.86, 3.98; S 17.02, 17.46. $C_{16}H_{15}NO_5S_2$. Calculated, %: N 3.83; S 17.55.

***N*-(4,6-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methylbenzenesulfonamide (IVe).** Yield 72%, mp 163–164°C. ^{1}H NMR spectrum, δ, ppm: 1.93 s and 2.01 s (3H each, 4-CH_3, 6-CH_3), 2.40 s (3H, $C\mathbf{H}_3C_6H_4$), 7.22 s (1H, 7-H), 7.39–7.56 d.d (4H, C_6H_4, J = 8.1 Hz), 9.53 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 17.68 and 18.45 (3-CH_3, 6-CH_3), 21.00 (**Me**C_6H_4), 111.23 (C^7), 120.48 (C^{3a}), 126.44 ($C^{2'}$), 129.65 ($C^{3'}$), 130.51 (C^5), 132.39 (C^4), 138.09 (C^6), 138.51 ($C^{1'}$), 143.05 ($C^{4'}$), 145.67 (C^{7a}), 168.14 (C^2). Found, %: N 3.89, 3.97; S 18.05, 18.64. $C_{16}H_{15}NO_4S_2$. Calculated, %: N 4.01; S 18.35.

4-Chloro-*N*-(4,6-dimethyl-2-oxo-1,3-benzoxathiol-5-yl)benzenesulfonamide (IVf). Yield 80%, mp 144–146°C. ^{1}H NMR spectrum, δ, ppm: 1.97 s and 2.03 s (3H each, 4-CH_3, 6-CH_3), 7.23 (1H, 7-H), 7.68 s (4H, 4-ClC_6H_4), 9.71 br.s (1H, NH). Found, %: N 3.78, 4.15; S 17.35, 17.69. $C_{15}H_{12}ClNO_4S_2$. Calculated, %: N 3.79; S 17.34.

***N*-(6,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-benzenesulfonamide (IVg).** Yield 66%, mp 201.5–203°C. ^{1}H NMR spectrum, δ, ppm: 1.88 s and 2.17 s (6H, 6-CH_3, 7-CH_3), 7.24 s (1H, 4-H), 7.52–7.66 m (5H, Ph), 9.78 s (1H, NH). Found, %: N 4.20, 4.06; S 19.00, 19.05. $C_{15}H_{13}NO_4S_2$. Calculated, %: N 4.18; S 19.12.

***N*-(6,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methylbenzenesulfonamide (IVh).** Yield 81%, mp 171–173°C. ^{1}H NMR spectrum, δ, ppm: 1.89 s and 2.18 s (6H, 6-CH_3, 7-CH_3), 2.37 s (3H, $C\mathbf{H}_3C_6H_4$), 7.24 s (1H, 4-H), 7.35–7.53 d.d (4H, C_6H_4, J = 8.1 Hz), 9.67 br.s (1H, NH). Found, %: N 3.77, 3.98; S 18.20, 18.68. $C_{16}H_{15}NO_4S_2$. Calculated, %: N 4.01; S 18.35.

***N*-(4,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methylbenzamide (Va).** Yield 63%, mp 199–201°C. ^{1}H NMR spectrum, δ, ppm: 2.17 s and 2.34 s (6H, 4-CH_3, 7-CH_3), 2.39 s (3H, **Me**C_6H_4), 7.23 s (1H, 6-H), 7.34–7.89 d.d (4H, C_6H_4, J = 8.1 Hz), 10.03 s (1H, NH). Found, %: N 4.40, 4.71; S 10.05, 11.04. $C_{17}H_{15}NO_3S$. Calculated, %: N 4.47; S 10.23.

***N*-(4,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methoxybenzamide (Vb).** Yield 57%, mp 207–208°C. ^{1}H NMR spectrum, δ, ppm: 9.92 s (1H, NH), 7.22 s (1H, 6-H), 7.06–7.97 d.d (4H, C_6H_4, J = 8.4 Hz), 3.84 s (3H, MeO), 2.34 s and 2.17 s (3H each, 4-CH_3, 7-CH_3). ^{13}C NMR spectrum, δ_C, ppm: 15.02 and 16.68 (4-CH_3, 7-CH_3), 55.33 (MeO), 113.49 ($C^{3'}$), 119.19 (C^7), 122.40 (C^{3a}), 125.87 (C^4), 126.10 ($C^{1'}$), 127.34 (C^6), 129.45 ($C^{2'}$), 133.35 (C^5), 143.62 (C^{7a}), 161.84 ($C^{4'}$), 164.92 (C=O), 168.14 (C^2). Found, %: N 4.01, 4.32; S 9.38, 9.99. $C_{17}H_{15}NO_4S$. Calculated, %: N 4.25; S 9.73.

4-Chloro-*N*-(4,7-dimethyl-2-oxo-1,3-benzoxathiol-5-yl)benzamide (Vc). Yield 69%, mp 227–228°C. ^{1}H NMR spectrum, δ, ppm: 10.17 s (1H, NH), 7.62–8.00 d.d (4H, C_6H_4, J = 8.4 Hz), 7.24 s (1H, 6-H), 2.34 s and 2.18 s (6H, 4-CH_3, 7-CH_3). Found, %: N 4.23, 4.41; S 9.34, 9.95. $C_{16}H_{12}ClNO_3S$. Calculated, %: N 4.20; S 9.61.

***N*-(4,6-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methylbenzamide (Vd).** Yield 62%, mp 201–203°C. ^{1}H NMR spectrum, δ, ppm: 2.18 s and 2.24 s (6H, 4-CH_3, 6-CH_3), 2.39 s (3H, **Me**C_6H_4), 7.35 s (1H, 7-H), 7.35–7.91 d.d (4H, C_6H_4, J = 8.1 Hz), 9.85 s (1H, NH). Found, %: N 4.48, 4.69; S 10.25, 10.42. $C_{17}H_{15}NO_3S$. Calculated, %: N 4.47; S 10.23.

***N*-(4,6-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methoxybenzamide (Ve).** Yield 64%, mp 188–189.5°C. ^{1}H NMR spectrum, δ, ppm: 2.18 s and 2.24 s (6H, 4-CH_3, 6-CH_3), 3.84 s (3H, MeO), 7.07–7.99 d.d (4H, C_6H_4, J = 9.0 Hz), 7.34 s (1H, 7-H), 9.77 s (1H, NH). Found, %: N 4.07, 4.39; S 9.54, 9.87. $C_{17}H_{15}NO_4S$. Calculated, %: N 4.25; S 9.73.

4-Chloro-*N*-(4,6-dimethyl-2-oxo-1,3-benzoxathiol-5-yl)benzamide (Vf). Yield 76%, mp 194–195°C. ^{1}H NMR spectrum, δ, ppm: 10.02 s (1H, NH), 7.63–8.03 d.d (4H, C_6H_4, J = 8.4 Hz), 7.36 s (1H, 7-H), 2.24 s and 2.19 s (6H, 4-CH_3, 6-CH_3). Found, %: N 4.19, 4.57; S 9.59, 9.68. $C_{16}H_{12}ClNO_3S$. Calculated, %: N 4.20; S 9.61.

***N*-(6,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-4-methylbenzamide (Vg).** Yield 53%, mp 193–194°C. ^{1}H NMR spectrum, δ, ppm: 2.16 s and 2.31 s (6H, 6-CH_3, 7-CH_3), 2.39 s (3H, **Me**C_6H_4), 7.34–7.90 d.d (4H, C_6H_4, J = 7.8 Hz), 7.55 s (1H, 4-H), 10.02 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 13.15 and 14.36 (6-CH_3, 7-CH_3), 21.03 (**Me**C_6H_4), 118.66 (C^7), 119.34 (C^4), 121.00 (C^{3a}), 127.72 ($C^{2'}$), 128.96 ($C^{3'}$), 131.35 (C^5), 133.50 (C^6), 133.55 ($C^{1'}$), 141.69 (C^{7a}), 144.76 ($C^{4'}$), 165.56 (C=O), 169.24 (C^2). Found, %: N 4.25, 4.53; S 10.07, 10.63. $C_{17}H_{15}NO_3S$. Calculated, %: N 4.47; S 10.23.

4-Chloro-*N*-(6,7-dimethyl-2-oxo-1,3-benzoxathiol-5-yl)benzamide (Vh). Yield 77%, mp 210–211°C. ^{1}H NMR spectrum, δ, ppm: 2.16 s and 2.32 s (6H, 6-CH_3, 7-CH_3), 7.55 s (1H, 4-H), 7.62–8.01 d.d (4H, C_6H_4, J = 8.4 Hz), 10.16 s (1H, NH). Found, %: N 4.05, 4.22; S 9.62, 9.98. $C_{16}H_{12}ClNO_3S$. Calculated, %: N 4.20; S 9.61.

***N*′-(4-Methoxyphenylsulfonyl)-*N*-(2-oxo-1,3-benzoxathiol-5-yl)benzimidamide (VIa).** Yield 74%, mp 178–180°C. ^{1}H NMR spectrum, δ, ppm: 3.82 s (3H, MeO), 6.99–7.46 d.d (4H, C_6H_4, J = 8.7 Hz), 7.40–7.71 m (5H, Ph), 7.58–7.61 d.d (1H, 6-H, J = 8.4, 1.5 Hz), 7.64 d (1H, 7-H, J = 8.4 Hz), 7.94 d (1H, 4-H, J = 1.5 Hz), 10.67 br.s (1H, NH). Found, %: N 6.38, 6.43; S 14.28, 14.57. $C_{21}H_{16}N_2O_5S_2$. Calculated, %: N 6.36; S 14.56.

***N*-(7-Methyl-2-oxo-1,3-benzoxathiol-5-yl)-*N*′-(4-methylphenylsulfonyl)benzimidamide (VIb).** Yield 49%, mp 147–148°C. ^{1}H NMR spectrum, δ, ppm: 2.22 s (3H, 7-CH_3), 2.37 s (3H, C**H**$_3C_6H_4$), 7.31–7.52 d.d (4H, C_6H_4, J = 7.5 Hz), 7.37 br.s (1H, 6-H), 7.55–7.64 m (5H, Ph), 7.66 br.s (1H, 4-H), 10.66 s (1H, NH). Found, %: N 6.47, 6.49; S 14.46, 14.50. $C_{22}H_{18}N_2O_4S_2$. Calculated, %: N 6.39; S 14.62.

***N*-(4,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-*N*′-(phenylsulfonyl)benzimidamide (VIc).** Yield 60%, mp 207–208.5°C. ^{1}H NMR spectrum, δ, ppm: 2.12 s and 2.28 s (3H each, 4-CH_3, 7-CH_3), 7.19 br.s (1H, 6-H), 7.39–7.58 m (10H, Ph), 10.43 br.s (1H, NH). Found, %: N 6.38, 6.55; S 14.07, 14.58. $C_{22}H_{18}N_2O_4S_2$. Calculated, %: N 6.39; S 14.62.

***N*-(4,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-*N*′-(4-methylphenylsulfonyl)benzimidamide (VId).** Yield 63%, mp 204–205°C. ^{1}H NMR spectrum, δ, ppm: 2.13 s and 2.28 s (3H each, 4-CH_3, 7-CH_3), 2.32 s (3H, C**H**$_3C_6H_4$), 7.18–7.38 d.d (4H, C_6H_4, J = 7.2 Hz), 7.22 br.s (1H, 6-H), 7.46–7.57 m (5H, Ph), 10.35 s (1H, NH). Found, %: N 6.00, 6.23; S 14.12, 14.83. $C_{23}H_{20}N_2O_4S_2$. Calculated, %: N 6.19; S 14.17.

***N*-(7-Isopropyl-2-oxo-1,3-benzoxathiol-5-yl)-*N*′-(4-methylphenylsulfonyl)benzimidamide (VIe).** Yield 68%, mp 164–166°C. ^{1}H NMR spectrum, δ, ppm: 1.15 d (6H, **Me**$_2$CH, J = 6.6 Hz), 2.15 s (3H, 4-CH_3), 2.32 s (3H, C**H**$_3C_6H_4$), 3.04–3.17 m (1H, Me_2C**H**), 7.22–7.49 d.d (4H, C_6H_4, J = 7.8 Hz), 7.23 br.s (1H, 6-H), 7.42–7.64 m (5H, Ph), 10.31 s (1H, NH). Found, %: N 5.67, 5.92; S 12.88, 13.37. $C_{25}H_{24}N_2O_4S_2$. Calculated, %: N 5.83; S 13.34.

***N*-(4-Isopropyl-7-methyl-2-oxo-1,3-benzoxathiol-5-yl)-*N*′-(4-methylphenylsulfonyl)benzimidamide (VIf).** Yield 56%, mp 188–190°C. ^{1}H NMR spectrum, δ, ppm: 1.18 d (6H, **Me**$_2$CH, J = 6.9 Hz), 2.31 s (6H, C**H**$_3C_6H_4$, 7-CH_3), 3.17–3.31 m (1H, Me_2C**H**), 7.18–7.32 d.d (4H, C_6H_4, J = 7.8 Hz), 7.20 br.s (1H, 6-H), 7.45–7.58 m (5H, Ph), 10.38 br.s (1H, NH). Found, %: N 5.38, 5.43; S 13.11, 13.58. $C_{25}H_{24}N_2O_4S_2$. Calculated, %: N 5.83; S 13.34.

***N*-(4,6-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-*N*′-(4-methylphenylsulfonyl)benzimidamide (VIg).** Yield 73%, mp 223–224°C. ^{1}H NMR spectrum, δ, ppm: 2.19 s and 2.26 s (3H each, 4-CH_3, 6-CH_3), 2.30 s (C**H**$_3C_6H_4$), 7.16–7.24 d.d (4H, C_6H_4, J = 8.1 Hz), 7.37 br.s (1H, 7-H), 7.45–7.59 m (5H, Ph), 10.25 br.s (1H, NH). Found, %: N 6.21, 6.44; S 14.20, 14.67. $C_{23}H_{20}N_2O_4S_2$. Calculated, %: N 6.19; S 14.17.

***N′*-(4-Chlorophenylsulfonyl)-*N*-(4,6-dimethyl-2-oxo-1,3-benzoxathiol-5-yl)benzimidamide (VIh).** Yield 78%, mp 228–230°C. ^{1}H NMR spectrum, δ, ppm: 2.19 s and 2.26 s (3H, 4-CH_3, 6-CH_3), 7.35–7.43 d.d (4H, C_6H_4, J = 8.7 Hz), 7.37 br.s (1H, 7-H), 7.46–7.60 m (5H, Ph), 10.39 s (1H, NH). Found, %: N 5.91, 6.32; S 13.51, 13.92. $C_{22}H_{17}ClN_2O_4S_2$. Calculated, %: N 5.92; S 13.56.

***N*-(6,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-*N′*-(4-methylphenylsulfonyl)benzimidamide (VIi).** Yield 82%, mp 201–202°C. ^{1}H NMR spectrum, δ, ppm: 2.10 s and 2.26 s (3H each, 6-CH_3, 7-CH_3), 2.32 s (3H, C**H**$_3$C$_6$H$_4$), 7.20–7.36 d.d (4H, C_6H_4, J = 7.8 Hz), 7.44–7.58 m (5H, Ph), 7.47 br.s (1H, 4-H), 10.38 s (1H, NH). Found, %: N 6.07, 6.38; S 14.00, 14.73. $C_{23}H_{20}N_2O_4S_2$. Calculated, %: N 6.19; S 14.17.

***N*-(6,7-Dimethyl-2-oxo-1,3-benzoxathiol-5-yl)-*N′*-(4-methoxyphenylsulfonyl)benzimidamide (VIj).** Yield 73%, mp 186–187°C. ^{1}H NMR spectrum, δ, ppm: 2.10 s and 2.26 s (3H each, 6-CH_3, 7-CH_3), 3.78 s (3H, MeO), 6.91–7.38 d.d (4H, C_6H_4, J = 8.7 Hz), 7.41–7.59 m (5H, Ph), 7.42 br.s (1H, 4-H), 10.35 br.s (1H, NH). Found, %: N 6.01, 6.18; S 13.22, 13.65. $C_{23}H_{20}N_2O_5S_2$. Calculated, %: N 5.98; S 13.69.

***N′*-(4-Chlorophenylsulfonyl)-*N*-(6,7-dimethyl-2-oxo-1,3-benzoxathiol-5-yl)benzimidamide (VIk).** Yield 71%, mp 244–245°C. ^{1}H NMR spectrum, δ, ppm: 2.11 s and 2.27 s (6H, 6-CH_3, 7-CH_3), 7.38 br.s (1H, 4-H), 7.46 s (4H, C_6H_4), 7.48–7.59 m (5H, Ph), 10.55 s (1H, NH). Found, %: N 5.77, 6.21; S 13.02, 13.53. $C_{22}H_{17}ClN_2O_4S_2$. Calculated, %: N 5.92; S 13.56.

***N*-(4-Hydroxy-3,5-dimethylphenyl)-4-methylbenzenesulfonamide (VIIi).** ^{1}H NMR spectrum, δ, ppm: 2.03 s (6H, 3-CH_3, 5-CH_3), 2.33 s (3H, C**H**$_3$-C$_6$H$_4$), 6.61 s (2H, 2-H, 6-H), 7.31–7.56 d.d (4H, C_6H_4, J = 8.4 Hz), 8.08 br.s (1H, NH), 9.60 s (1H, OH).

4-Chloro-*N*-(4-hydroxy-3,5-dimethylphenyl)-benzenesulfonamide (VIIj). Yield 79%, mp 148–149°C. ^{1}H NMR spectrum, δ, ppm: 2.04 s (6H, 3-CH_3, 5-CH_3), 6.61 s (2H, 2-H, 6-H), 7.61–7.67 d.d (4H, C_6H_4, J = 8.7 Hz), 8.14 br.s (1H, NH), 9.75 s (1H, OH). Found, %: N 4.53, 4.69; S 10.11, 10.35. $C_{14}H_{14}ClNO_3S$. Calculated, %: N 4.49; S 10.28.

***N*-(4-Hydroxy-3,5-dimethylphenyl)-4-methylbenzamide (VIIIi).** Yield 73%, mp 217–218°C. ^{1}H NMR spectrum, δ, ppm: 2.16 s (6H, 3-CH_3, 5-CH_3), 2.37 s (3H, C**H**$_3$C$_6$H$_4$), 7.29 s (2H, 2-H, 6-H), 7.29–7.84 d.d (4H, C_6H_4, J = 8.1 Hz), 8.03 br.s (1H, NH), 9.74 s (1H, OH). Found, %: N 5.51, 5.70. $C_{16}H_{17}NO_2$. Calculated, %: N 5.49.

***N*-(4-Hydroxy-3,5-dimethylphenyl)-4-methoxybenzamide (VIIIj).** Yield 72%, mp 194–196°C. ^{1}H NMR spectrum, δ, ppm: 2.16 s (6H, 3-CH_3, 5-CH_3), 3.83 s (3H, MeO), 7.03–7.92 d.d (4H, C_6H_4, J = 9.0 Hz), 7.28 s (2H, 2-H, 6-H), 8.00 br.s (1H, NH), 9.73 s (1H, OH). Found, %: N 5.10, 5.28. $C_{16}H_{17}NO_3$. Calculated, %: N 5.16.

4-Chloro-*N*-(4-hydroxy-3,5-dimethylphenyl)-benzamide (VIIIk). Yield 69%, mp 230–231°C. ^{1}H NMR spectrum, δ, ppm: 2.16 s (6H, 3-CH_3, 5-CH_3), 7.29 s (3H, 2-H, 6-H), 7.58–7.95 d.d (4H, C_6H_4, J = 8.7 Hz), 8.02 br.s (1H, NH), 9.56 s (1H, OH). Found, %: N 4.98, 5.16. $C_{15}H_{14}ClNO_2$. Calculated, %: N 5.08.

***N*-(4-Hydroxy-3,5-dimethylphenyl)-*N′*-(4-methylphenylsulfonyl)benzimidamide (IXl).** Yield 85%, mp 190–191°C. ^{1}H NMR spectrum, δ, ppm: 2.05 s (6H, 3-CH_3, 5-CH_3), 2.36 s (3H, C**H**$_3$C$_6$H$_4$), 7.10 s (2H, 2-H, 6-H), 7.31–7.58 d.d (4H, C_6H_4, J = 7.8 Hz), 7.45–7.55 m (5H, Ph), 8.30 s (1H, NH), 10.27 s (1H, OH). Found, %: N 7.12, 7.31; S 8.02, 8.23. $C_{22}H_{22}N_2O_3S$. Calculated, %: N 7.10; S 8.13.

***N′*-(4-Chlorophenylsulfonyl)-*N*-(4-hydroxy-3,5-dimethylphenyl)benzimidamide (IXm).** Yield 80%, mp 185–186°C. ^{1}H NMR spectrum, δ, ppm: 2.05 s (6H, 3-CH_3, 5-CH_3), 7.07 s (2H, 2-H, 6-H), 7.57–7.93 d.d (4H, C_6H_4, J = 8.4 Hz), 7.49–7.62 m (5H, Ph), 8.31 s (1H, NH), 10.40 s (1H, OH). Found, %: N 6.59, 6.80; S 7.65, 7.83. $C_{21}H_{19}ClN_2O_3S$. Calculated, %: N 6.75; S 7.73.

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