

Activated Sterically Strained C=N Bond in *N*-Arylsulfonyl-*p*-quinonemono- and Diimines: IX.* Synthesis and Reactions of *N*-Tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine

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Abstract—*N*-Tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine as the other *N*-arylsulfonyl-1,4-benzoquinonimines with an activated C=N bond reacts along 1,2-addition path with alcohols and sodium azide and along 1,2-addition–elimination path with aromatic amines. The higher activity of the C=N bond in the *N*-arylsulfonyl-1,4-benzoquinonimines is not due to the electronic character of the substituent attached to the ring (Cl, CH₃) but to steric influence resulting in increase in the bond angle C=N–S.

We formerly studied 2,3,5,6-tetrachloroderivatives of *N*-arylsulfonyl-*p*-quinonimines that had chlorine atoms in both *ortho*-positions with respect to C=N bond and thus possessing activated sterically strained C=N bond (hereinafter “activated” C=N bond). A number of reactions with these bonds proceed unlike those with ordinary *N*-arylsulfonyl-*p*-quinonimines with no substituents in positions 3 and 5 [2–7].

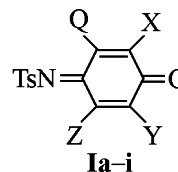
The most characteristic reactions of quinonimines with the activated C=N bond are reactions occurring at these bonds: 1,2-addition and 1,2-addition–elimination [2, 4, 6, 7].

The high activity of the C=N bond was explained by increase in the bond angle C=N–S due to the presence of substituents in both *ortho*-positions with respect to C=N bond. The X-ray diffraction analysis of *N*-4-chlorophenylsulfonyl-3,5-dimethyl-1,4-benzoquinonimine confirms the above statement: The angle C=N–S is equal to 132.7° [5].

The high activity of the C=N bond in the *N*-arylsulfonyl-2,3,5,6-tetrachloro-1,4-benzoquinonimines might be caused by the changed charge on the C⁴ carbon due to four electron-acceptor substituents in the quinoid ring. It was presumable that introduction into the ring of four electron-donor CH₃ groups

would change the activity of the C=N bond in *N*-arylsulfonyl-2,3,5,6-tetramethyl-1,4-benzoquinonimines.

The change of the electron density on a carbon atom may be followed by ¹³C NMR spectroscopy: The downfield shift of the signal (δ_C) evidences the reduced electron density on the carbon, and the upfield shift corresponds to the increase in the electron density [8]. We studied the ¹³C NMR spectra of *N*-tosyl-1,4-benzoquinonimines **Ib–i** with various substituents in the quinoid ring and compared them with the spectrum of *N*-tosyl-1,4-benzoquinonimine unsubstituted in the ring (**Ia**) (Table 1).

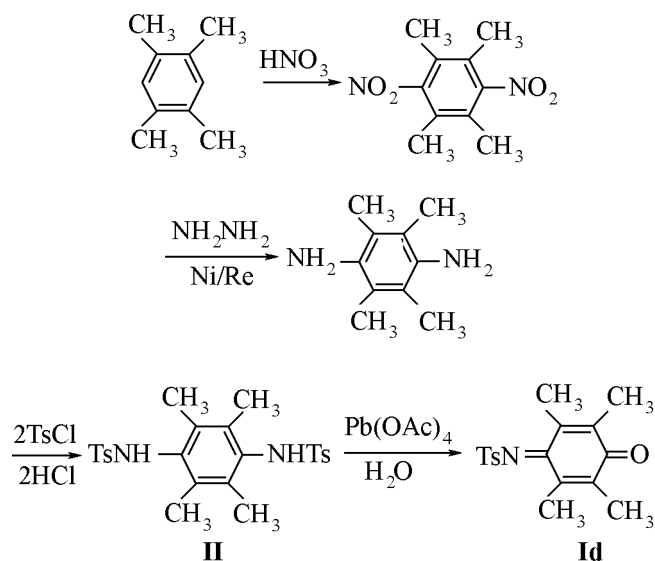


X = H (**a, c, e**), CH₃ (**b, d**), Cl (**f–i**); ¶ = H (**a, c**), CH₃ (**b, d**), Cl (**e–i**); Z = H (**a, b, g**), CH₃ (**c–f**), Cl (**h, i**); Q = H (**a, b, g, h**), CH₃ (**c–f**), Cl (**i**).

In the quinonimine series under consideration the signal δ(C⁴) is the most shifted downfield in *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (**Id**), and the greatest upfield shift of the δ(C⁴) signal

* The preceding communication, see [1].

Scheme 1.



is observed in the spectrum of *N*-tosyl-2,3,5,6-tetrachloro-1,4-benzoquinonimine (**II**). Thus the electron density on the C^4 carbon in this series is the smallest in quinonimine **Id** and the greatest in quinonimine **II**.

We previously studied the chemical properties of quinonimines **Ic**, **e**, **f**, **i** containing an activated C=N bond. As compared with unsubstituted in the ring quinonimine **Ia** the electron density on the C^4 carbon is greater in tetrachloroderivative *p*-quinonimine **II**, slightly greater in *p*-quinonimines **Ie**, **f**, and slightly lower in *p*-quinonimine **Ic** [2-7].

The reactivity of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (**Id**) possessing the lowest electron density on the C^4 carbon was not studied earlier.

The synthesis of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (**Id**) is uncommon, since instead of benzoquinonediimine forms benzoquinonemonoimine (Scheme 1).

The nitration of 1,2,4,5-tetramethylbenzene cleanly provides dinitroarene [9]. Along the published procedure of dinitroarene reduction with tin(II) chloride we obtained the double salt of tin chloride and diaminodurene hydrochloride [9]. The attempt to acylate the salt with *p*-toluenesulfonyl chloride was unsuccessful. Therefore we prepared diaminodurene by reduction of dinitroarene with hydrazine hydrate on Raney nickel. Diaminodurene base was acylated with *p*-toluenesulfonyl chloride in ethyl ether in the presence of triethylamine.

N,N'-Ditosyl-2,3,5,6-tetramethyl-1,4-phenylenediamine (**II**) was oxidized with lead tetraacetate, and

Table 1. Chemical shifts (δ_C , ppm) of C^4 carbon in the ^{13}C NMR spectra of *N*-tosyl-1,4-benzoquinonimines (**Ia-i**)

Compd. no.	$\delta(C^4)$	$\Delta(C^4)$
Id	167.85	4.21
Ic	164.72	1.08
Ib	164.56	0.92
Ia	163.64	0
If	162.60	-1.04
Ie	162.20	-1.44
Ig	160.01	-3.63
Ih	156.03	-7.61
Ii	149.83	-13.81

instead of the expected *N,N'*-ditosyl-2,3,5,6-tetramethyl-1,4-benzoquinonediimine we obtained the product of its partial hydrolysis, *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonemonoimine (**Id**).

The composition and structure of compounds **Id**, **II** were proved by elemental analysis (Table 2) and IR, ^1H and ^{13}C NMR spectra.

In the IR spectrum of compound **Id** appear the absorption bands at the frequencies 1627, 1596, 1555, 1322, and 1161 cm^{-1} characteristic respectively of C=O, C=C, C=N, and SO_2 groups.

The IR spectrum of compound **II** contains absorption bands at 3280, 1133, and 1171 cm^{-1} characteristic respectively of NH and SO_2 groups.

The ^1H NMR spectrum of compound **Id** contains the following signals (δ , ppm): 7.26-7.88 d.d (4H, Ts), 2.44 s (3H, Ts), 2.02 d, 2.24 d (12H, CH_3 of quinoid ring).

The ^{13}C and APT NMR spectra of compound **Id** are completely consistent with the assumed structure [$\delta(C^4)$, ppm]: 185.44 (C=O), 167.85 (C=N), 143.26 (C^4 , Ts), 142.11, 140.06 ($C^{2,3,5,6}$ of quinoid ring), 140.02 (C^1 , Ts), 129.40, 126.59 ($C^{2,3,5,6}$, Ts), 21.52 (CH_3 , Ts), 18.83, 12.80 (four CH_3 groups of quinoid ring). The magnetic equivalence of carbon atoms C^2 , C^6 and C^3 , C^5 of quinoid ring, and also of carbons in the CH_3 groups in positions 2,6 and 3,5 of quinoid ring evidences *Z,E* isomerization at the nitrogen atom in quinonimine **Id** that is fast according to the NMR time scale.

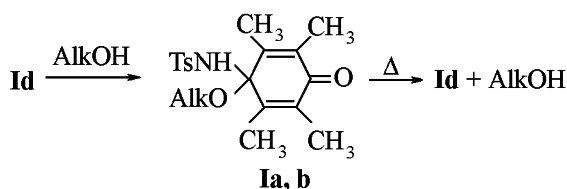
Reactions of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonemonoimine (**Id**) with alcohols occur as 1,2-addition affording the products of quinolid structure: 4-alkoxy-4-(tosylamido)-2,3,5,6-tetramethyl-2,5-cyclohexadien-2-ones (**IIIa**, **b**) (Scheme 2)

Table 2. Yields, melting points, and elemental analyses of compounds **Id**, **II**, **IIIa, b**, **IVa, b**, **V**, **VIa, b**, **VIIa, b**, **VIII**

Compd. no.	Yield, %	mp, °C (solvent for crystallization)	Found N, %	Formula	Calculated N, %
Id	36	134 (C ₇ H ₁₆)	4.40, 4.52	C ₁₇ H ₁₉ NO ₃ S	4.42
II	84	273 (decomp.) (AcOH)	5.73, 5.85	C ₂₄ H ₂₆ N ₂ O ₄ S ₂	5.96
IIIa	68	184 (C ₆ H ₆ +C ₆ H ₁₄)	3.95, 3.99	C ₁₈ H ₂₃ NO ₄ S	4.01
IIIb	72	148 (C ₆ H ₆ +C ₆ H ₁₄)	4.02, 4.07	C ₁₉ H ₂₅ NO ₄ S	3.86
IVa	25	92 (AcOH, reprecipitation)	5.45, 5.53	C ₁₇ H ₁₉ NO	5.53
IVb	40	73 (AcOH, reprecipitation)	4.50, 4.57	C ₁₆ H ₁₆ BrNO	4.40
V	86	60 (decomp.)	–	C ₁₇ H ₂₀ N ₄ O ₃ S	15.56
VIa+VIIa	70	– (AcOH)	2.92, 3.01	C ₂₃ H ₂₅ NO ₅ S ₂	3.05
VIb+VIIb	62	– (AcOH)	2.95, 3.06	C ₂₄ H ₂₇ NO ₅ S ₂	2.96
VIII	75	210 (C ₆ H ₆)	4.35, 4.40	C ₁₇ H ₂₁ NO ₃ S	4.39

similar to the products forming in reaction with alcohol from the other *N*-arylsulfonyl-1,4-benzoquinonimines with the activated C=N bond [2, 5].

The heating of compounds **IIIa, b** to melting point results in their dealkoxylation to yield the original quinonimine **Id**; similar process we have observed formerly with analogous products [2].

Scheme 2.

In the IR spectra of compounds **IIIa, b** are present the characteristic absorption bands of the C=O group in the quinolid ring in the region 1625–1630 cm⁻¹ and of NH group at 3180–3250 cm⁻¹.

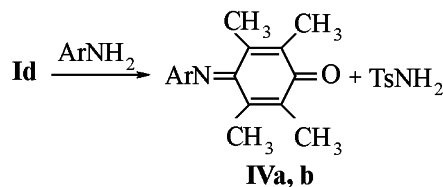
In the ¹H NMR spectrum of compound **IIIa** were observed the following signals: broadened singlet of a proton from NH group (5.22 ppm), three-proton singlet of CH₃O group (2.84 ppm), two doublets (1.62 and 1.73 ppm) from 6 protons of the two magnetically equivalent CH₃ groups in positions 2, 6 and from 6 protons of the two magnetically equivalent CH₃ groups in positions 3, 5 of the quinolid ring, and also from the protons in the tosyl group (δ, ppm): 7.12–7.50 d.d (4H), 2.40 s (3H, CH₃). Thus the spectrum is in total agreement with the assumed structure.

In the ¹H NMR spectrum of compound **IIIb** are present analogous resonances (δ, ppm): 7.20–7.49

d.d (4H, Ts), 4.92 br.s (1H, NH), 2.95 q (2H, OCH₂), 2.42 s (3H, CH₃ in Ts), 1.72 d, 1.62 d (12H, CH₃ of quinoid ring), 1.10 t (3H, CH₃ of Et).

In the ¹³C NMR spectrum of compound **IIIa** in a strong field was observed a characteristic signal of sp³-hybridized carbon atom in the quinolid structure [δ(C⁴) 84.60 ppm]; the other signals are fully consistent with the assumed structure (δ_C, ppm): 184.12 (C=O), 146.39 (C^{3,5} of quinolid ring), 143.59 (C⁴, Ts), 136.86 (C¹, Ts), 135.97 (C^{2,6} of quinolid ring), 129.16, 127.53 (C^{2,3,5,6}, Ts), 49.67 (CH₃O), 21.49 (CH₃, Ts), 13.80, 11.42 (CH₃ of quinolid ring).

In reaction of quinonimine **Id** with aromatic amines we failed to isolate the products of 1,2-addition: occurred 1,2-addition-elimination affording *N*-aryl-2,3,5,6-tetramethyl-1,4-benzoquinonimines (**IVa, b**) (Scheme 3).

Scheme 3.

Ar = 4-CH₃C₆H₄ (a), 4-BrC₆H₄ (b).

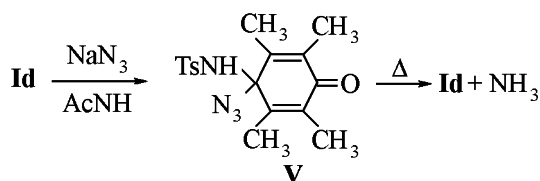
IR spectra of compounds **IVa, b** contain the characteristic absorption bands of C=O, C=C, and C=N groups of quinoid ring in the regions 1640–1630, 1615–1610, and 1590–1580 cm⁻¹ respectively.

In the ¹H NMR spectrum of compound **IVa** appear the signals corresponding to the assumed structure

(δ , ppm): 6.63–7.13 d.d (4H, 4-CH₃C₆H₄), 2.34 (3H, 4-CH₃C₆H₄), 2.24 br.s, 1.98 br.d, 1.50 br.s (12H, CH₃ in quinoid ring). It should be noted that the protons of methyl groups attached to positions 3 and 5, 2 and 6 of the quinoid ring are nonequivalent unlike those in *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinoneminoimine (**Id**). The dynamic *Z,E*-isomerization process at the nitrogen in quinonimine **IVa** is evidenced by the broadened signals of the protons corresponding the methyl groups at the quinoid ring.

The hydrazoic acid adds to quinonimine **Id** in 1,2-position, as with the other quinonimines with the activated C–N group [4]. The resulting product of quinolid structure, 4-azido-4-(tosylamido)-2,3,5,6-tetramethyl-2,5-cyclohexadien-2-one (**V**) is unstable and at heating eliminates a molecule of hydrazoic acid to recover the original quinonimine **Id**. (Scheme 4). We observed such a process for the first time.

Scheme 4.



Previously we observed that such quinolid structures with chlorine atoms in 2 and 6 positions underwent C⁴–C²-migration of azido group with simultaneous nucleophilic substitution of chlorine [4]. In our case the nucleophilic substitution of CH₃ group is impossible and therefore occurs elimination of HN₃ molecule.

In the IR spectrum of compound **V** appear characteristic absorption bands of NH, N₃, C=O « SO₂ groups at 3233, 2082, 1635, 1341 and 1170 cm⁻¹.

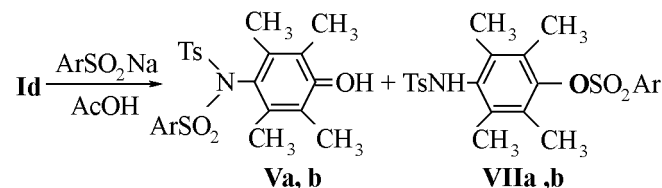
In the ¹H NMR spectrum of compound **V** appear the following signals: broadened singlet of the NH group proton (5.16 ppm), two doublets (1.71 and 1.79 ppm) from the 12 protons of the four CH₃ groups attached to quinolid ring, and the resonances from tosyl group protons at 7.48–7.69 d.d (4H) and 2.43 s (3H, CH₃) ppm in full conformity to the assumed structure.

In the ¹³C NMR spectrum of compound **V** in a strong field was observed a characteristic signal of sp³-hybridized carbon atom in the quinolid structure [δ (C⁴) 94.19 ppm]; the other signals are fully consistent with the assumed structure (δ _C, ppm): 180.90 (C=O), 146.68 (C⁴, Ts), 134.90 (C¹, Ts), 129.95

127.73 (C^{2,3,5,6}, Ts), 129.43, 127.38 (C^{2,3,5,6} of quinolid ring), 24.23 (CH₃, Ts), 15.23, 11.47 (CH₃ of quinolid ring).

The reaction of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (**Id**) with arylsulfonic acids occurs in two directions: 6,1- and 1,6-addition affording a mixture of *N*-tosyl-*N*-arylsulfonyl-2,3,5,6-tetramethyl-1,4-aminophenols (**Va, b**) and *N*-tosyl-*O*-arylsulfonyl-2,3,5,6-tetramethyl-1,4-aminophenols (**VIIa, b**) in 1 : 1 ratio (Scheme 5).

Scheme 5.



Ar = C₆H₅ (a), 4-CH₃C₆H₄ (b).

Commonly arylsulfonic acids react with *N*-arylsulfonyl-1,4-benzoquinonimines along the scheme of 1,4- or 1,6-addition, and in the presence of chlorine in 2 and 6 position of the quinoid ring occurs nucleophilic substitution of chlorine by the arylsulfonic rest. The 1,6-addition of arylsulfonic acid to *N*-arylsulfonyl-1,4-benzoquinonimines possessing isopropyl or methoxy groups in 2 and 6 positions [10].

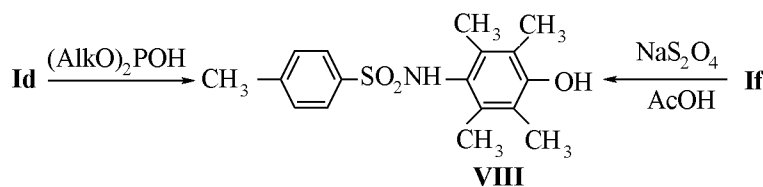
With *N*-arylsulfonyl-2,3,5,6-tetrachloro-1,4-benzoquinonimines only 1,6-addition was observed [11]. In the case under consideration (Scheme 5) occurred unusual combination of two simultaneous processes: 6,1- and 1,6-addition.

In the IR spectra of the mixture of compounds **VIa, VIIa** and **VIb, VIIb** are present the absorption bands of OH, NH, and SO₂ in the regions 3540–3520, 3280–3260, 1380–1370 and 1175–1170 cm⁻¹ respectively.

In the ¹H NMR spectrum of the mixture of compounds **VIa, VIIa** were observed the following signals (δ , ppm): 9.38 br.s (1H, OH), 8.51 br.s (1H, NH), 7.93–7.35 m (18H, ArSO₂ and Ts), 2.45 s, 2.39 s (6H, CH₃ in Ts), 2.02 s (6H, CH₃ *ortho*-position to OSO₂Ph in compound **VIIa**), 1.84 s (6H, CH₃ *meta* to OSO₂Ph of compound **VIIa**), 1.82 s (6H, CH₃ *meta* to OH of compound **VIa**), 1.59 s (6H, CH₃ *ortho* to OH of compound **VIa**).

In the ¹H NMR spectrum of the mixture of compounds **VIb, VIIb** were observed the following signals (δ , ppm): 9.30 br.s (1H, OH), 8.74 br.s (1H,

Scheme 6.



Alk = CH₃, *i*-C₃H₇.

NH), 7.40–7.91 four d.d (16H, Ts), 2.51 s, 2.43 s (6H, CH₃ in Ts of compound **VIIIb**), 2.49 s (6H, CH₃ in Ts of compound **VIb**), 2.12 s (6H, CH₃ *ortho* to OTs of compound **VIIIb**), 1.96 s (6H, CH₃ *meta* to OTs of compound **VIIIb**), 1.95 s (6H, CH₃ *meta* to OH of compound **VIb**), 1.72 s [6H, CH₃ *ortho* to OH of compound **VIb**).

The reaction of quinonimine **Id** with dialkyl phosphites resulted only in its reduction to *N*-tosyl-2,3,5,6-tetramethyl-1,4-aminophenol (**VIII**). Aminophenol **VIII** also formed on treatment of quinonimine **Id** with sodium dithionite in acetic acid (Scheme 6).

The reduction of quinonimine **Id** with dialkyl phosphites may be ascribed to its high redox potential.

Similar process was also observed for *N*-arylsulfonyl-2,3,5,6-tetrachloro-1,4-benzoquinonimines; however in this case were obtained the phosphorylation products of quinonimines formed along 6,1- and 1,2-addition [6].

IR spectra of compound **VIII** contain the absorption bands at 3520, 3278, 1327 and 1168 cm⁻¹ corresponding to OH, NH, and SO₂ groups respectively.

¹H NMR spectrum of compound **VIII** (δ, ppm): 9.00 s (1H, OH), 8.03 s (1H, NH), 7.72–7.49 d.d (4H, Ts), 2.38 s (3H, CH₃ in Ts), 1.99 (6H, CH₃ *meta* to OH), 1.78 s (6H, CH₃ *ortho* to OH) is consistent with the assumed structure.

The results of the present research and that performed before [1–7] show that the chief cause governing the direction of the reactions with *N*-arylsulfonyl-1,4-benzoquinonimines possessing two substituents in *ortho*-position to the C=N bond is not the electronic factors but the steric influence leading to decrease in the C=N–S angle.

EXPERIMENTAL

IR spectra of compounds synthesized were recorded on spectrophotometer UR-20 from KBr pellets. ¹H and ¹³C NMR spectra were registered on spectrometer Varian VXR-300 at operating frequencies 300

and 75.4 MHz respectively; TMS was applied as external reference. Compounds **Id**, **IIIa, b**, **IVa, b**, **V** were recorded in CDCl₃ solution, compounds **VIa, b**, **VIIa, b**, **VIII** in DMSO-*d*₆. The characteristics of compounds obtained are listed in Table 2.

Diaminodurene. To a suspension of dinitrodurene in 15 ml of ethanol was added 0.2 g of Raney nickel and then was added dropwise 4 ml of hydrazine hydrate. The mixture was heated to 60–70°C and gradually turned transparent and colorless. On completion of the reduction the catalyst was filtered off, and ethanol was evaporated on a water bath. The diaminodurene obtained was washed with water and dried. mp 155°C [9].

***N,N'*-Ditosyl-2,3,5,6-tetramethyl-1,4-phenylenediamine (II).** To a solution of 5 mmol of diaminodurene in 20 ml of ethyl ether was added 10.5 mmol of *p*-toluenesulfonyl chloride and then 10 mmol of triethylamine. The precipitated triethylamine hydrochloride was filtered off, the filtrate was evaporated in a vacuum. The colorless precipitate formed was washed with water, filtered, dried, and recrystallized from glacial acetic acid.

***N*-Tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (Id).** To a suspension of 2 mmol of *N,N'*-ditosyl-2,3,5,6-tetramethyl-1,4-phenylenediamine (**II**) in 5 ml of glacial acetic acid was added 4.06 g of lead tetraacetate. The solution turned orange. Then was added several drops of ethylene glycol, the mixture was stirred, and water was added dropwise till formed orange precipitate of compound **Id**. The precipitate was filtered off, washed with water, dried, and recrystallized from heptane.

***N*-Tosyl-2,3,5,6-tetramethyl-1,4-aminophenol (VIII).** To a solution of 5 mmol of quinonimine **Id** in 25 ml of boiling glacial acetic acid was added by portions 0.5 g of sodium dithionite till the solution was colorless. On cooling to the solution was added several milliliters of water till complete precipitation of reaction product **VIII**. The precipitate was filtered off, washed with water, dried, and recrystallized from benzene.

Reaction of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (Id) with alcohols. In 5 ml of an appropriate alcohol was refluxed for 0.5 h 0.5 mmol of quinonimine Id. The alcohol was evaporated, the arising crystalline reaction product was recrystallized from a benzene–hexane mixture.

Reaction of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (Id) with alkyl phosphites. To 1 ml of dialkylphosphite heated to 100°C was added 0.5 mmol of quinonimine. The solution obtained was heated to 150°C. The color of the solution turned from orange to pale yellow. The solution was cooled and left standing for a week. The separated colorless crystalline product VIII was washed with butanol and recrystallized from benzene, mp 210°C. Yields in reaction with dimethyl phosphite 85%, with diisopropyl phosphite 79%.

Reaction of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (Id) with aromatic amines. To a solution of 0.5 mmol of quinonimine in 5 ml of chloroform was added 0.5 mmol of an aromatic amine. The mixture was stirred by magnetic stirrer for 2 h and left overnight. The formed thick mass was dissolved in a little of acetic acid, and to the solution obtained was dropwise added water till separation of a precipitate. Compound IVa, b was purified by reprecipitation from acetic acid.

Reaction of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (Id) with sodium azide. To a suspension of 0.5 mmol of quinonimine Id in 3 ml of acetic acid was added 0.15 g of NaN₃, and the mixture was stirred by magnetic stirrer. Gradually the bright orange color of the solution turned to pale yellow. The precipitated colorless crystalline product V was filtered off and washed with acetic acid.

Reaction of *N*-tosyl-2,3,5,6-tetramethyl-1,4-benzoquinonimine (Id) with sodium arylsulfonates. To a boiling solution of 0.5 mmol of quinonimine in

3 ml of acetic acid was added 0.6 mmol of sodium arylsulfonate, and the boiling was continued for 10 min. The bright orange color of the solution turned to pale yellow. On cooling precipitated the colorless crystalline reaction product containing a mixture of isomers VI and VII in 1:1 ratio. The precipitate was filtered off, washed with water, and recrystallized from acetic acid. The characteristics of the isomer mixtures VIa, VIIa, and VIb, VIIb are given in Table 2.

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