SYNTHESIS OF 4-AMINO-3-QUINOLINESULFONIC ACIDS AND 4-AMINOQUINOLINES *

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Abstract - The hydrolysis of 4-chloro-3-quinolinesulfonyl chloride (**1**) gives 4-chloro-3-quinolinesulfonic acid (**2**) or 1,4-dihydro-4-oxo-3-quinolinesulfonic acid (**3**). Compound (**2**) reacts with primary and secondary aliphatic or primary aromatic amines to give 4-amino-3-quinolinesulfonic acids (**4**). Desulfonation of quinolinesulfonic acids (**2**,**3**,**4**) yields 4(1*H*)-quinolinone or 4-aminoquinolines (**5**).

INTRODUCTION

4-Chloro-3-quinolinesulfonyl chloride (**1**) can be obtained easily by of chlorinolysis of thioquinanthrene.[1] Two reactive chlorine atoms allow introduction of amino groups converting sulfochloride (**1**) to a series of 4-chloro- and 4-amino-3-quinolinesulfamides. It was found that the chlorine of the chlorosulfonyl group is more reactive than the chlorine located at the γ-position of quinoline.[1] The aim of presented work is to find whether the reaction of compound (**1**) with alcohols can yield respective 4-substituted 3-quinolinesulfonates (4-chloro or 4-alkoxy). It appears, however, that the main product of the above mentioned reaction is sulfonic acid, i.e., 4-chloro-3-quinolinesulfonic acid (**2**) or 1,4-dihydro-4-oxo-3-quinolinesulfonic acid (**3**).

RESULTS AND DISCUSSION

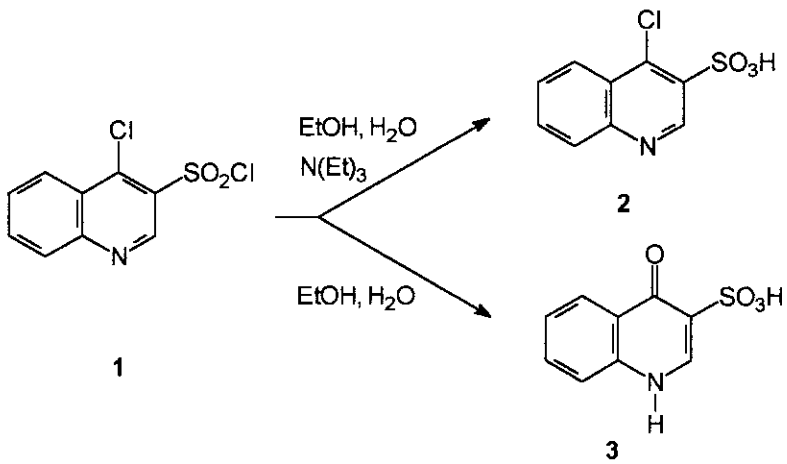
In a model reaction 4-chloro-3-quinolinesulfonyl chloride (**1**) was treated with ethanol. 4-Chloro-3-quinolinesulfonic acid (**2**) was the main product of such a reaction carried out in a large excess of refluxing 99.8% ethanol in the presence of triethylamine used as hydrochloride acceptor. The formation of the acid (**2**) was observed both in aqueous and anhydrous ethanolic solutions. Therefore, chlorosulfonyl group can hydrolyze by reacting directly with water or by the following sequence of reactions: $SO_2Cl \rightarrow SO_2OEt \rightarrow SO_3H$. It seems that the ester formed in the latter sequence could have been hydrolyzed as a result of exposure to atmospheric moisture. In fact, the mixture after evaporation of anhydrous ethanol and an

excess of triethylamine was highly hygroscopic.

4-Chloro-3-quinolinesulfonyl chloride (**1**), when treated with other alcohols (methanol, 1-propanol, 2-propanol, 2-methyl-2-propanol and cyclopentanol) yielded the same product, i.e., 4-chloro-3-quinoline-sulfonic acid (**2**).

It has been observed that a substitution of the chlorine in the γ-position can be prevented, provided that the reaction is performed under mild conditions (e.g., at temperatures below -70 °C).[1] Acidification of the reaction solution in the presence of an excess of amine should also be avoided because 4-quinolines are very reactive in acidic medium [2] which can be explained by protonation of the nitrogen atom activating the γ-position toward nucleophilic attack.[3-5]

Therefore, both chlorine atoms should undergo hydrolysis in reactions of sulfochloride (**1**) performed in aqueous ethanolic solution without triethylamine as hydrochloride acceptor. In fact, 1,4-dihydro-4-oxo-3-quinolinesulfonic acid (**3**) was isolated from such a reaction in the yield of 90%.

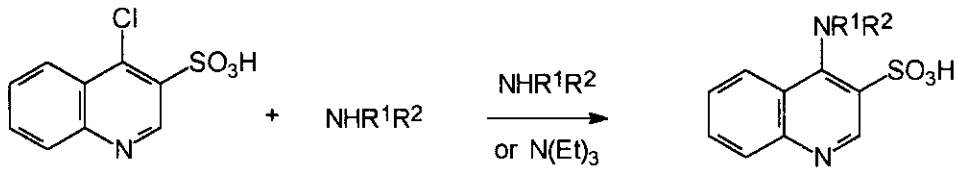


1,4-Dihydro-4-oxo-3-quinolinesulfonic acid (**3**) was previously obtained by condensation of o-formamido-acetophenone followed by sulfonylation. [6] The reported mp and NMR data of the product significantly differ from those measured by us. The observed mp (301-303 °C) is higher than reported one, [6] and the value of chemical shift (9.30 ppm) for the **H**-2 proton of quinoline ring measured in DMSO-d_6 was also not consistent with the reported value of 8.83 ppm (DMSO-d_6). However, the value of chemical shift measured in this work compares favorably with the chemical shifts of **H**-2 protons of other 4-substituted 3-quinolinesulfonic acids which are listed in the experimental section. Almost all these values are higher than 9 ppm.

Amination of 4-chloro-3-quinolinesulfonic acid (**2**)

4-Chloro-3-quinolinesulfonic acid (**2**) has a reactive chlorine atom which can be replaced by different groups in nucleophilic substitution reactions. The acid (**2**) was treated with ammonia, as well as primary

and secondary aliphatic and aromatic amines. In the reactions of ammonia and aliphatic amines an excess of amine was used as a hydrochloride acceptor, while triethylamine needed to be added for this purpose in reactions with aromatic amines. *N*-monosubstituted and *N,N*-disubstituted 4-amino-3-quinolinesulfonic acids were formed in the above mentioned reactions. The yields range from 71 to 83%. However, the acid (**2**) did not react with ammonia and *N*-methylaniline. It was found earlier that 4-chloro-3-quinolinesulfonamides are also unreactive toward ammonia and *N*-methylaniline.[1]



2 **4**

Table 1

Product			Yield
	R^1	R^2	[%]
4a	H	CH_3	81
4b	CH_3	CH_3	81
4c	H	CH_2CH_3	71
4d	CH_2CH_3	CH_2CH_3	75
4e	$-CH_2CH_2OCH_2CH_2-$		65
4f	H	Ph	68
4g	H	*p*-CH_3Ph	75
4h	H	*p*-NO_2Ph	81
4i	H	*p*-NH_2SO_2Ph	83

It is worth noting that the acid (**2**) reacts easily with very weak bases, e.g., nitroaniline. Apparently in this case, the activation of position 4 by the ortho effect of the neighboring sulfonyl group is effectively operative as described previously. [1,7]

All 4-substituted 3-quinolinesulfonic acids (**2**,**3**,**4**) obtained are relatively sparingly soluble in water, which enables their separation from aqueous solutions by acidifying of the respective sodium salt with hydrochloric acid.

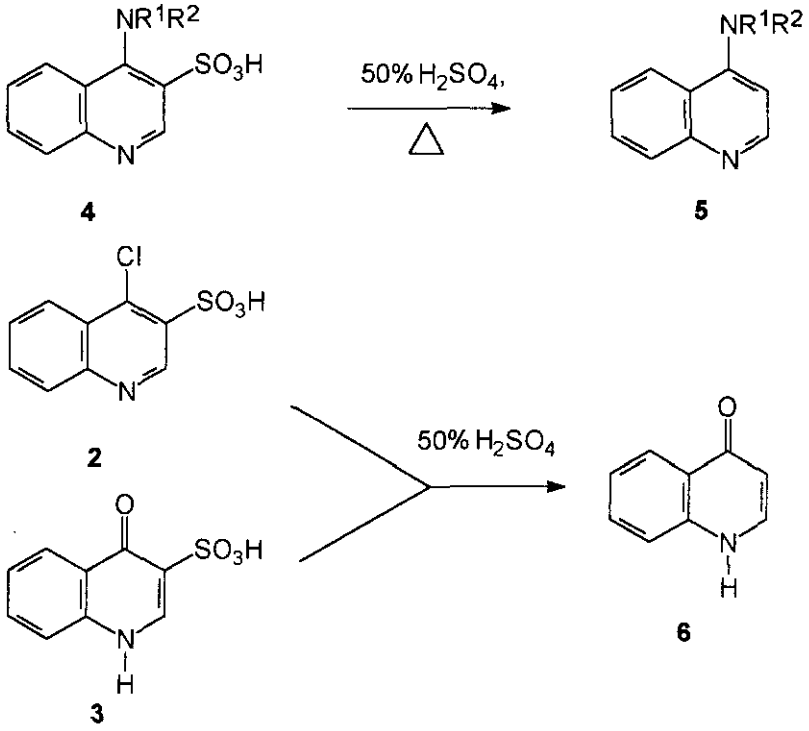
Desulfonation of 4-amino-3-quinolinesulfonic acid (**4**)

4-Aminoquinolines are synthesized by reactions of 4-chloroquinolines with amines under high pressure [2] or using the so-called phenolic method. [8-10] Biological activity of these compounds stimulated interest in

developing new and more attractive synthetic routes.[11] A series of alkyl, aryl and alkyl - aryl 4-aminoquinolines was synthesized already from 4-quinolinesulfonic acids [12] or from quinoline *N*-oxide *via* the reaction of 1-(4-quinolyl)pyridinium tosylate with amines.[13] Recently it was found that 4-aminoquinolines inhibit the replication of human immunodeficiency virus (HIV). [14]

Desulfonation of the acids (**2-4**) yielding 4-substituted quinolines provides a new synthetic route to 4-aminoquinolines (**5**) and 4(1*H*)-quinolinone (**6**).

The recorded spectra of 4-substituted quinolinesulfonic acids obtained indicate probable desulfonation, i.e., the signal of desulfonated fragments is displayed with 100% intensity, while the molecular peaks of the acids (**3**) and (**4**) cannot be observed within EI MS.



In fact, treatment with 50% sulfuric acid yielded the expected products of desulfonation, i.e., 4-aminoquinolines (**5**) (80-95% yields) and 4(1*H*)-quinolinone (**6**) (74%). In the reactions 4-chloro-3-quinolinesulfonic acid (**2**) initially undergoes hydrolysis yielding the acid (**3**) which is further desulfonated to give quinolone (**6**). The reaction failed, however, to give pure 4-aminoquinoline derivatives of *p*-nitroaniline (**5h**) and sulfanilamide (**5i**). Although the MS analyses prove the homogeneity of samples obtained, the compounds cannot be characterized by sharp mp and color of the samples depends upon the purification procedure. It is believed that both 4-aminoquinolines (**5h**) and (**5i**) can form a mixture of stable 4-amine - 4-iminic tautomers. The occurrence of the tautomeric forms of 4-aminoquinoline - 4-iminoquinoline was previously observed and confirmed by means of ^{1}H NMR spectroscopy by Renault and Cartron.[15]

Table 2

	Product		Yield
	R^1	R^2	[%]
5a	H	CH_3	80
5b	CH_3	CH_3	85
5c	H	CH_2CH_3	92
5d	CH_2CH_3	CH_2CH_3	80
5e	$-CH_2CH_2OCH_2CH_2-$		85
5f	H	Ph	95
5g	H	*p*-CH_3Ph	94
5h	H	*p*-NO_2Ph	90
5i	H	*p*-NH_2SO_2Ph	81

EXPERIMENTAL

Melting points were determined in open capillary tubes on a Boetius mp apparatus and are uncorrected. The ^{1}H NMR spectra were recorded on a Bruker MSL 300 (300 MHz) spectrometer with tetramethylsilane as the internal standard. Chemical shifts are reported in ppm (δ) and *J* values in Hz. EIMS were run on a LKB GC 2091 spectrometer at 70 eV and 15 eV.

4-Chloro-3-quinolinesulfonic acid (**2**): A mixture of 4-chloro-3-quinolinesulfonyl chloride (**1**) (786 mg, 3 mmol) and triethylamine (1.016 g, 9 mmol) in 99.8% ethanol (10 mL) and water (1 mL) was refluxed for 6 h. The mixture was cooled and, then, volatile components of the reaction mixture were distilled off under vaccum. The residue was treated with 2% aqueous sodium hydroxide (10 mL), and the insoluble part was removed with chloroform (2 x 7 mL). The aqueous solution was acidified with 20% hydrochloric acid and filtered off to give 620 mg of the pure product (**2**) (85%). mp 315-316 °C (decomp). EI MS (m/z): 243(M^+,100). ^{1}H NMR (DMSO-d_6) δ: 7.99-8.04(m, 1H, **H**-6); 8.28-8.30(m, 1H, **H**-8); 8.14-8.16(m, 1H, **H**-7); 8.53- 8.56(m, 1H, **H**-5); 9.51(s, 1H, **H**-2). *Anal.* Calcd for $C_9H_6NO_3ClS$: C 44.36, H 2.48, N 5.75, Cl 14.55. Found: C 44.21, H 2.41, N 5.84, Cl 14.68.

The acid (**2**) is moisture-sensitive and undergoes slowly hydrolysis to the acid (**3**). The acid (**2**) can be purified by dissolution in 5% aqueous sodium hydroxide and precipitation with 20% hydrochloric acid.

1,4-Dihydro-4-oxo-3-quinolinesulfonic acid (**3**): A solution of **1** (786 mg, 3 mmol) in 96% ethanol (20 mL) was refluxed for 3 h. The mixture was cooled down and the precipitated solid was filtered off to give 843 mg of the product (**3**) (90%). mp 301-303 °C (decomp) (50% aqueous ethanol). EI MS (m/z): 145(M-SO_3, 100). ^{1}H NMR (DMSO-d_6) δ: 7.84-7.90 (m, 1H, **H**-6); 8.13-8.19(m, 2H, **H**-7, **H**-8);

8.45-8.48(m, 1H, **H**-5); 9.30(s, 1H, **H**-2); 10.96(s, 1H, N-**H**). *Anal.* Calcd for $C_9H_7NO_4S$: C 48.00, H 3.13, N 6.22, S 14.24. Found: C 48.23, H 3.19, N 6.45, S 14.91.

4-Amino-3-quinolinesulfonic acides (**4**). General procedure:

A mixture of 4-chloro-3-qunolinesulfonic acid (**2**) (487 mg, 2 mmol), aliphatic amine (1 mL or 3 mL 40% aqueous solution) or aromatic amine (2.2 mmol) and triethylamine (1 mL, 7.17 mmol) in water (10 mL) was refluxed for 2 h (aliphatic amine) or 10 h (aromatic amine). The solution was cooled down and volatile components of the reaction mixture were distilled off under vaccum. Water (3 mL) was added and the solution was acidified with 20% hydrochloric acid. The precipitate was filtered off to give the product (**4**). The solid was air-dried and finally recrystallized from ethanol and water-ethanol. Yields of compounds (**4**) are collected in Table 1. The same reaction with 25% ammonia (5 mL) was failed only unaffected acid (**2**) was isolated in 90% yield.

4-Methylamino-3-quinolinesulfonic acid (**4a**): mp 274-275 °C (decomp). EI MS (15 eV), (m/z) 158 (M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 3.64(d, *J*=5.6 Hz, 1H, NHC**H**$_3$); 7.59-7.65(m, 1H, **H**-6); 7.85-7.96(m, 2H, **H**-7, **H**-8); 8.61-8.64(m, 1H, **H**-5); 8.67(s, 1H, **H**-2); 9.27(m, 1H, N**H**CH$_3$); 13.54(s, 1H, SO_3**H**). *Anal.* Calcd for $C_{10}H_{10}N_2O_3S$: C 50.41, H 4.23, N 11.76, S 13.46. Found: C 50.20, H 4.11, N 11.96, S 13.71.

4-Dimethylamino-3-quinolinesulfonic acid (**4b**): mp 204-207 °C. EI MS (15 eV), (m/z) 172(M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 3.73(s, 6H, C**H**$_3$). 7.71-7.76(m, 1H, **H**-6); 7.99-8.01(m, 2H, **H**-7, **H**-8); 8.39-8.42(m, 1H, **H**-5); 9.13(s, 1H, **H**-2). *Anal.* Calcd for $C_{11}H_{12}N_2O_3S$: C 52.37, H 4.79, N 11.10, S 12.71. Found: C 52.45, H 4.52, N 10.91, S 12.54.

4-Ethylamino-3-quinolinesulfonic acid (**4c**): mp 255-257 °C (decomp). EI MS (15 eV), (m/z) 172(M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 1.36(t, *J*=7.1 Hz, 3H, NHCH$_2$C**H**$_3$); 4.05(m, 2H, NHC**H**$_2$CH$_3$); 7.59-7.64 (m, 1H, **H**-6); 7.84-7.96(m, 2H, **H**-7, **H**-8); 8.50-8.53(m, 1H, **H**-5); 8.67(s, 1H, **H**-2); 9.19(m, 1H, N**H**CH$_2$CH$_3$). *Anal.* Calcd for $C_{11}H_{12}N_2O_3S$ x 1/2 H_2O: C 50.52, H 4.98, N 10.72, S 12.25. Found: C 50.21, H 5.15, N 10.51, S 12.39.

4-Diethylamino-3-quinolinesulfonic acid (**4d**): mp 235-236 °C (decomp). EI MS (15 eV), (m/z) 200 (M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 1.88(t, *J*=7.0 Hz, 6H, 2 x CH$_2$C**H**$_3$); 4.04(q, *J*=7.1 Hz, 4H, 2 x C**H**$_2$CH$_3$); 7.67-7.73(m, 1H, **H**-6); 7.92-8.00(m, 2H, **H**-7, **H**-8); 8.19-8.22(m, 1H, **H**-5); 9.16(s, 1H, **H**-2); 13.62(s, 1H, SO_3**H**). *Anal.* Calcd for $C_{13}H_{16}N_2O_3S$: C 55.70, H 5.75, N 9.99, S 11.44. Found: C 55.80, H 5.55, N 9.74, S 11.62.

4-Morpholino-3-quinolinesulfonic acid (**4e**): mp 232-234 °C. EI MS (15 eV), (m/z) 214(M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 3.87-3.90(m, 4H, -C**H**$_2$NC**H**$_2$-); 4.09-4.12(m, 4H, -C**H**$_2$OC**H**$_2$-). 7.68-7.72(m, 1H, **H**-6); 7.96-7.98(m, 2H, **H**-7, **H**-8); 8.27-8.30(m, 1H, **H**-5); 9.18(s, 1H, **H**-2). *Anal.* Calcd for

$C_{13}H_{14}N_2O_4S$: C 53.05, H 4.79, N 9.52, S 10.89. Found: C 52.89, H 4.64, N 9.31, S 11.03.

4-Anilino-3-quinolinesulfonic acid (**4f**): mp 262-263 °C. EI MS (15 eV), (m/z) 220(M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 7.29-7.53(m, 7H, C_6**H**$_5$, and **H**-5, **H**-6); 7.86-7.91(m, 1H, **H**-7); 7.99-8.01(m, 1H, **H**-8); 9.04(s, 1H, **H**-2); 10.55(s, 1H, N**H**Ph). *Anal.* Calcd for $C_{15}H_{12}N_2O_3S$: C 59.99, H 4.03, N 9.33, S 10.67. Found: C 60.11, H 4.21, N 9.51, S 10.52.

4-(*p*-Toluidino)-3-quinolinesulfonic acid (**4g**): mp 270-271 °C. EI MS (15 eV), (m/z) 234(M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 2.35(s, 3H, PhC**H**$_3$); 7.18-7.29(m, 4H, C_6**H**$_4$); 7.36-7.41(m, 1H, **H**-6); 7.52-7.55 (m, 1H, **H**-5); 7.85-7.90(m, 1H, **H**-7); 7.97-8.00(m, 1H, **H**-8); 9.01(s, 1H, **H**-2); 10.52(s, 1H, N**H**PhCH$_3$); 14.40(s, 1H, SO_3**H**). *Anal.* Calcd for $C_{16}H_{14}N_2O_3S$: C 61.13, H 4.49, N 8.91, S 10.20. Found: C 61.28, H 4.32, N 8.78, S 10.34.

4-(*p*-Nitroanilino)-3-quinolinesulfonic acid (**4h**): mp 263-265 °C (decomp). EI MS (15 eV), (m/z) 265 (M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 7.45-7.50(m, 1H, **H**-6); 7.59-7.68(m, 4H, C_6**H**$_4$); 7.92-7.99(m, 1H, **H**-7); 8.07-8.13(m, 2H, **H**-5, **H**-8); 9.18(s, 1H, **H**-2); 10.48(s, 1H, N**H**PhNO_2). *Anal.* Calcd for $C_{15}H_{11}N_3O_5S$: C 52.17, H 3.21, N 12.17, S 9.28. Found: C 52.10, H 3.47, N 12.50, S 9.42.

4-Sulfanilamino-3-quinolinesulfonic acid (**4i**): mp 300-301 °C (decomp). EI MS (15 eV), (m/z) 300 (M-SO_3, 100%). ^{1}H NMR (DMSO-d_6) δ: 7.38-7.41 and 7.79-7.83(m, 4H, C_6**H**$_4$); 7.41(s, 2H, N**H**$_2$); 7.49-7.55(m, 1H, **H**-6); 7.61-7.64(m, 1H, **H**-8); 7.93-7.99(m, 1H, **H**-7); 8.08-8.10(m, 1H, **H**-5); 9.18(s, 1H, **H**-2); 10.41(s, 1H, N**H**Ph). *Anal.* Calcd for $C_{15}H_{13}N_3O_5S_2$: C 47.49, H 3.43, N 11.08, S 16.89. Found: C 47.48, H 3.61, N 11.20, S 16.73.

4-Aminoquinolines (**5**). General procedure:

A solution of 4-amino-3-quinolinesulfonic acids (**4**) (1 mmol) in 50% sulfuric acid (10 mL) was refluxed for 5 h. After cooling to rt the solution was alkalized with 10% aqueous sodium hydroxide. The amines obtained (**5**) were filtered off or extracted with chloroform (2 x 5 mL). Crystalline 4-aminoquinolines (**5**) were recrystallized from ethanol and oily amines (**5**) were purified by extraction with hot hexane. Yields of compounds (**5**) are collected in Table 2.

4-Methylaminoquinoline (**5a**): mp 226-228 °C, lit.,[13,16] mp 229-230 °C.

4-Dimethylaminoquinoline (**5b**): oil, lit.,[16] oil.

4-Ethylaminoquinoline (**5c**): mp 189-190 °C, lit.,[17] mp 189-190 °C.

4-Diethylaminoquinoline (**5d**): oil, lit.,[18] oil.

4-Morpholinoquinoline (**5e**): mp 78-80 °C. EI MS (15 eV), (m/z) 214(M^+, 100%). ^{1}H NMR ($CDCl_3$) δ: 3.21-3.24(m, 4H, -C**H**$_2$NC**H**$_2$-); 3.97-3.99(m, 4H, -C**H**$_2$OC**H**$_2$-); 6.85(d, *J*=5.0 Hz, 1H, **H**-3); 7.46-7.52 (m, 1H, **H**-6); 7.63-7.69(m, 1H, **H**-7); 8.00-8.08(m, 2H, **H**-5, **H**-8); 8.75(d, *J*=5.0 Hz, 1H, **H**-2). *Anal.*

Calcd for $C_{13}H_{14}N_2O$: C 72.87, H 6.59, N 13.07. Found: C 72.93, H 6.42, N 12.86.

4-Phenylaminoquinoline (**5f**): mp 199-200 °C, lit.,[19] mp 196-197 °C.

4-(*p*-Methylphenyl)aminoquinoline (**5g**): mp 178-180 °C, lit.,[13] mp 180-181 °C.

4-Oxo-1,4-dihydroquinoline (**6**): A solution of 4-chloro-3-quinolinesulfonic acid (**2**) or 1,4-dihydro-4-oxo-3-quinolinesulfonic acid (**3**) (3 mmol) in 50% sulfuric acid (5 mL) was refluxed for 3 h. After cooling to rt, solution was neutralized with aqueous 20% sodium hydroxide to pH 3-5. The precipitate was filtred off and recrystallized from water to give 360 mg (74%) of 4-oxo-1,4-dihydroquinoline (**6**) as a hydrate with the mp 100-101 °C (lit., [20] mp 100 °C).

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