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Fused Pyrimidines. IV.[1] The Reaction of 4-Alkylthio-2-chloroquinazolines with Alkylamines in Dimethylformamide[2]

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The reaction of 4-alkylthio-2-chloroquinazolines with alkylamines in dimethylformamide afforded 2,4-bis(alkylthio)-, 2-alkylamino-4-alkylthio-, 4-alkylamino-2-chloro-, 4-alkylamino-2-alkylthio-, and 2,4-bis(alkylamino)quinazolines. The relative easiness of displacement of the chlorine and/or the alkylthio group in 4-alkylthio-2-chloroquinazolines depended on the bulkiness rather than the basicity of the alkylamine. A possible reaction mechanism is discussed.

Keywords——amination; isomer; substitution reaction; mechanism; 2-alkylamino-4-alkylthioquinazoline; 4-alkylamino-2-alkylthioquinazoline; 4-alkylthio-2-chloroquinazoline

In the course of research on quinazoline derivatives showing herbicidal activity, the displacement reaction of 4-alkylthio-2-chloroquinazolines (**1**) with alkylamines has been studied.

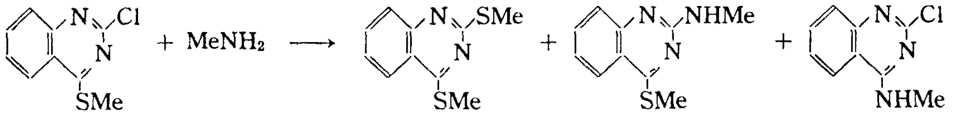
Curd *et al.* reported[3] that the reaction of 2-diethylaminoethylamine with 2-chloro-4-(4-chlorophenylthio)quinazoline gave 2,4-bis(4-chlorophenylthio)-, and 4-(2-diethylaminoethylamino)-2-(4-chlorophenylthio)quinazoline. On the other hand, they reported[4] that the reaction of 2-chloro-4-methylthioquinazoline with 4-chloroaniline gave 2-(4-chloroanilino)-4-methylthioquinazoline as a major product.

This paper deals with the migration of the chlorine *versus* the alkylthio group in derivatives of **1**. A possible reaction mechanism is discussed.

(i): The reaction of 2-chloro-4-methylthioquinazoline (**1a**) with methylamine in dimethylformamide at 20—25°C for 16 h gave **2a** as colorless needles (mp 78°C), **3a** as yellow needles (mp 131—132°C) and **4a** as colorless needles (mp 213°C).

Compound **2a** was confirmed to be 2,4-bis(methylthio)quinazoline[5] on the basis of its infrared (IR) and proton magnetic resonance (PMR) spectra. Similarly, **4a** was identified as 2-chloro-4-methylaminoquinazoline.[6]

The elemental analysis of **3a** was in good agreement with the calculated values for $C_{10}H_{11}N_3S$. The IR spectrum of **3a** revealed an NH band at 3200 cm^{-1}. The PMR spectrum of **3a** indicated the presence of a methylthio group (δ: 2.48, 3H, s), an *N*-methyl group (δ: 2.99, 3H, s), an imino group (δ: 5.32, 1H, br s), and four aromatic protons (δ: 6.82—7.82, 4H, m). On the basis of these data, the structure of **3a** was established to be 2-methylamino-4-methylthioquinazoline.



1a **2a** **3a** **4a**

Chart 1

(ii): On the other hand, when the same reaction was carried out at 80—85°C for 20 min, compound **4a** was obtained exclusively. Then, the reaction of **1a** with ethylamine in dimethylformamide at 80—85°C for 20 min gave **2a**, **3b** as yellow needles (mp 79—80°C), **4b** as colorless needles (mp 171°C), **5b** as colorless needles (149—150°C) and a trace amount of **6b** as colorless needles (mp 123—126°C).

Compound **4b** was confirmed to be 2-chloro-4-ethylaminoquinazoline by comparison of its spectral data with those of an authentic sample.[6]

The elemental analysis of **6b** was in good agreement with the calculated values for $C_{12}H_{16}N_4$. The IR spectrum of compound **6b** showed an NH band at 3250 cm^{-1}, and the presence of two ethylamino groups was apparent in the PMR spectrum . On the basis of these data. the structure of **6b** was assigned as 2,4-bis(ethylamino)quinazoline.[6]

Compounds **3b** and **5b** had the same molecular formula, $C_{11}H_{13}N_3S$, and their PMR spectra indicated the presence of an ethylamino group (δ: 1.12, 3.30—3.80 for **3b** and 1.22, 3.40—3.87 for **5b**) and a methylthio group (δ: 2.56 for **3b** and 2.57 for **5b**). However, the IR spectra and melting points of these compounds were different from each other. These results show that the structure of **3b** is isomeric with that of **5b**. In order to differentiate clearly the struutures of **3b** and **5b**, these compounds were desulfurized with Raney nickel. Compound **3b** afforded **10** as colorless needles (mp 96—97°C), while **5b** gave **11** as colorless needles (mp 149—150°C). The elemental analyses of both **10** and **11** were in good agreement with the calculated values for $C_{10}H_{11}N_3$. Compound **11** was confirmed to be 4-ethylaminoquinazoline[6] by unequivocal synthesis, *via* the reaction of 4-chloroquinazoline[7] (**12**) with ethylamine.

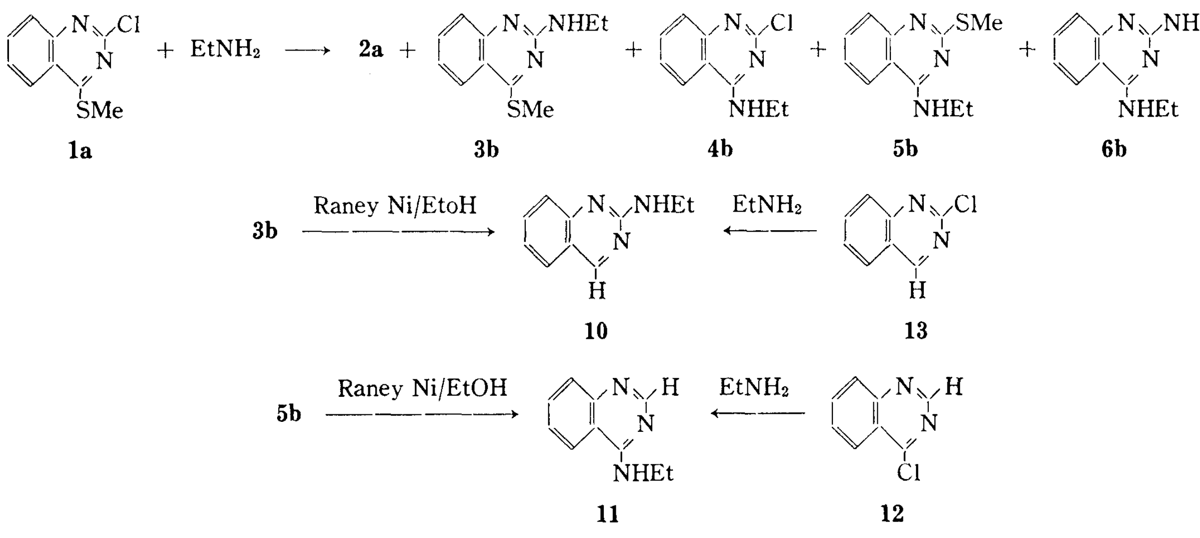


Chart 2

Consequently, the structure of **5b** was confirmed to be 4-ethylamino-2-methylthioquinazoline, and compounds **10** and **3b** were assigned as 2-ethylaminoquinazoline and 2-ethylamino-4-methylthioquinazoline, isomers of **11** and **5b**, respectively. The structure of **10** was also confirmed by synthesis from 2-chloroquinazoline[8] (**13**) and ethylamine.

Compound **3b** showed a characteristic ultraviolet (UV) absorption maximum at 362 nm,while **5b** had an absorption at 328 nm. By

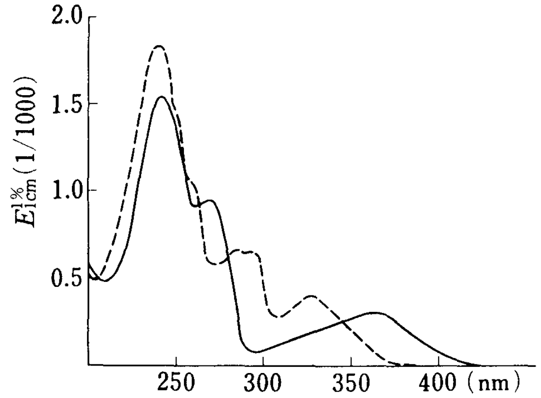
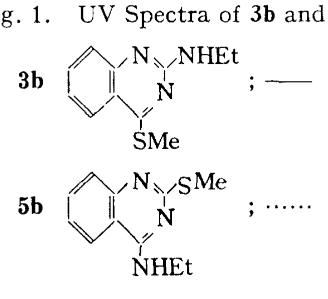


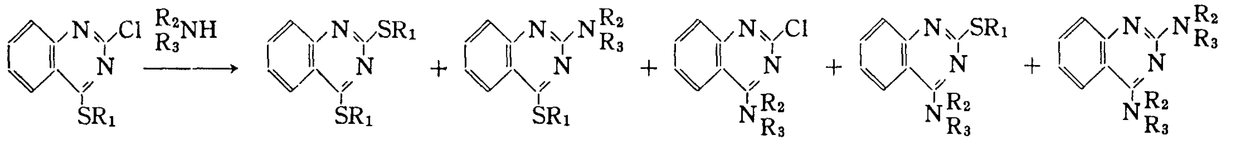
Fig. 1. UV Spectra of **3b** and **5b**



comparing their UV spectra in the region of about 300—400 nm, 2-alkylamino-4-alkylthioquinazoline (**3**) and 4-alkylamino-2-alkylthioquinazolines (**5**) can be easily and unequivocally differentiated.

To investigate the scope and limitations of this type of reaction, the study was extended to include the use of other alkylamines (*e.g.*, butyl-, *sec*-butyl-, *tert*-butyl-, dimethyl-, dipropyl-, and diisopropylamine). The results are summarized in Table I and II.

Table I. The Reaction of 4-Alkylthio-2-chloroquinazoline with Alkylamine



1 **2** **3** **4** **5** **6**

Starting materials R^1	R^2R^3NH	Reaction conditions time (h)	Temp (°C)	Products and yields (%)				
Me	$MeNH_2$	16	20—25	**2a** (5)	**3a** (21)	**4a** (65)	—	—
Me	$MeNH_2$	1/3	80—85	—	—	**4a** (92)	—	—
Me	$EtNH_2$	1/3	80—85	**2a** (2)	**3b** (38)	**4b** (54)	**5b** (3)	**6b** (1)
Me	$BuNH_2$	1/3	80—85	**2a** (6)	**3c** (34)	**4c** (33)	**5c** (9)	**6c** (3)
Me	*tert*-$BuNH_2$	3	Reflux	—	**3d** (25)	**4d** (1)	—	—
tert-Bu	*sec*-$BuNH_2$	1/3	80—85	—	**3e** (19)	—	—	—
Me	Me_2NH	1/3	80—85	**2a** (5)	**3f** (26)	**4f** (35)	**5f** (13)	**6f** (1)
Me	Pr_2NH	3	Reflux	—	**3g** (92)	—	—	—
Me	iso-Pr_2NH	3	Reflux	—	—	—	—	—

Table II. UV Absorption Maxima of 3 and 5 in EtOH

3	nm (ε_{max})	5	nm (ε_{max})
3a	365(2135)	—	
3b	362(6570)	**5b**	328(8320)
3c	365(5340)	**5c**	330(6870)
3d	366(4997)	—	
3e	366(5647)	—	
3f	370(4420)	**5f**	337(7400)
3g	377(4639)	—	

An alkylamine which is not bulky, such as methyl-, ethyl-, butyl- or dimethylamine, reacts with the chlorine at the 2-position and/or the methylthio group at the 4-position in the quinazoline ring to afford 2- and/or 4-alkylaminoquinazolines.

A bulky amine, such as *sec*-butyl-, *tert*-butyl- or dipropylamine, reacts with the chlorine at the 2-position to give 2-alkylamino-4-alkylthioquinazolines as a major product. Even bulkier amines, such as diisopropylamine, do not react with **1**.

The results of these studies show that the reaction of **1** with alkylamines is substantially affected by the bulkiness rather than the basicity of the alkylamines.

Some reactions were attempted in order to clarify the mechanism of formation of compounds **2**—**6**. The reaction of **2a** with butylamine in dimethylformamide at 80—85°C for 20 min gave mainly **5c** instead of **3c**. The reaction of **3c** with butylamine gave chiefly **6c**, but **4c** and **5c** did not react with butylamine.

On the basis of these results, a possible mechanism for the reaction of **1** with alkylamines is shown in Chart 3.

The herbicidal activities of the quinazoline derivatives are now under investigation and will be reported shortly.

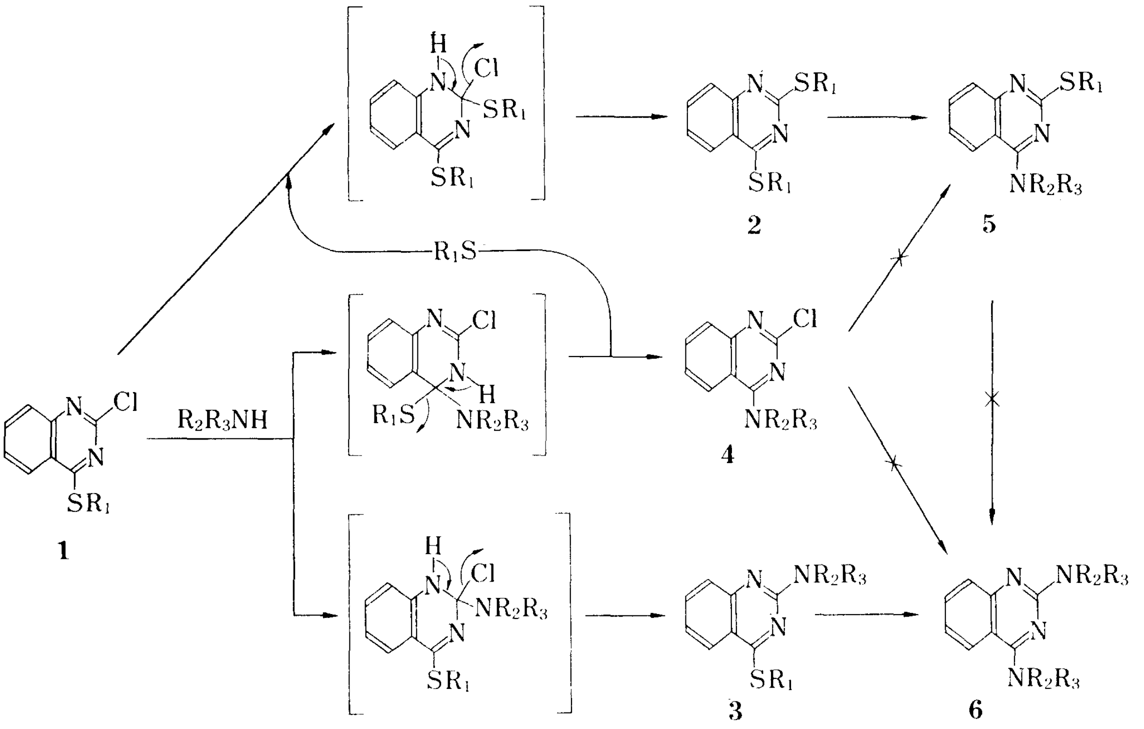


Chart 3

Experimental

Melting points were determined with a Yanagimoto micro melting point apparatus and are uncorrected. IR spectra were recorded by using a Hitachi EPI-3 spectrophotometer, and PMR spectra were taken with a Hitachi R-24 spectrometer; chemical shifts are expressed in ppm (δ) from tetramethylsilane as an internal standard. The following abbreviations are used: s, singlet; d, doublet; t, triplet; m, multiplet; br, broad. UV spectra were recorded with a Shimadzu UV-210 spectrophotometer. Silica gel (Kiesel gel, Merck) was used for column chromatography.

2-Chloro-4-methylthioquinazoline (1a)——A mixture of 2,4-dichloroquinazoline[9] (2.0 g, 10.0 mmol) and methylmercaptan sodium salt (*ca.* 15% in water, 4.7 g, 10.0 mmol) in acetone (30 ml) was stirred at 10—15°C for 1 h. The resulting mixture was poured into ice-water, and extracted with dichloromethane (50 ml × 3). The extract was dried over magnesium sulfate, and concentrated. The residue was recrystallized from acetonitrile to give 1.7 g of **1a** as colorless needles, mp 119—120°C, (lit.,[4] mp 122°C).

2-Chloro-4-*tert*-butylthioquinazoline (1e)——A mixture of 2,4-dichloroquinazoline (2.0 g, 10.0 mmol) and *tert*-butylmercaptan potassium salt (1.3 g, 10.2 mmol) in acetone (30 ml) was worked up as described for **1a** to give 1.6 g of **1e** as pale yellow needles, mp 114—115°C. *Anal.* Calcd for $C_{12}H_{14}ClN_3$: C, 61.14; H, 5.99; N, 17.83. Found: C, 61.01; H, 5.49; N, 17.56.

Reaction of 1a with Methylamine——A mixture of **1a** (4.2 g, 20.0 mmol), methylamine (40% in water, 1.7 g, 21.9 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was stirred at 20—25°C for 16 h. The resulting mixture was poured into ice-water, and extracted with dichloromethane (50 ml × 3). The extract was washed with water, dried over magnesium sulfate and concentrated. The residue was subjected to column chromatography on silica gel. Elution with acetone–hexane (1: 2) gave 0.11 g of 2,4-bis(methylthio)quinazoline (**2a**) as colorless needles (mp 78°C), 0.86 g of 2-methylamino-4-methylthioquinazoline (**3a**) as yellow needles (mp 131—132°C), and 2.5 g of 2-chloro-4-methylaminoquinazoline (**4a**) as colorless needles (mp 213°C). **3a**: *Anal.* Calcd for $C_{10}H_{11}N_3S$: C, 58.31; H, 5.25; N, 20.46. Found: C, 58.39; H, 5.20; N, 20.52. PMR ($CDCl_3$): 2.48 (3H, s, SCH_3), 2.99 (3H, d, NCH_3), 5.32 (1H, br s, NH), 6.82—7.82 (4H, m, Ar–H). IR (Nujol): 3200 cm^{-1} (NH).

Reaction of 1a with Ethylamine——A mixture of **1a** (4.2 g, 20.0 mmol), ethylamine (70% in water, 1.6 g, 24.9 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was stirred at 80—85°C for 20 min. The resulting mixture was worked up as described for the reaction of **1a** with methylamine to give 0.05 g of **2a**, 1.7 g of 2-ethylamino-4-methylthioquinazoline (**3b**) as yellow needles (mp 79—80°C), 2.24 g of 2-chloro-4-ethylaminoquinazoline (**4b**) as colorless needles (mp 171°C), 0.13 g of 4-ethylamino-2-methylthioquinazoline (**5b**) as colorless needles (mp 149—151°C), and 0.04 g of 2,4-bis(ethylamino)quinazoline (**6b**) as colorless needles (mp 123—126°C). **3b**: *Anal.* Calcd for $C_{11}H_{13}N_3S$: C, 60.24; H, 5.97; N, 19.16. Found: C, 60.35; H, 5.83; N, 19.09. PMR ($CDCl_3$): 1.21 (3H, t, CH_3), 2.56 (3H, s, SCH_3), 3.30—3.80 (2H, m, CH_2), 5.24 (1H, br s, NH), 6.95—8.01 (4H, m, Ar–H). IR (Nujol): 3230 cm^{-1} (NH). **5b**: *Anal.* Calcd for $C_{11}H_{13}N_3S$: C, 60.24; H, 5.97; N, 19.16. Found: C, 60.40; H, 5.91; N, 19.37. PMR ($CDCl_3$): 1.22 (3H, t, CH_3), 2.57 (3H, s, SCH_3), 3.40—3.87 (2H, m, CH_2), 6.19 (1H, br s, NH), 7.10—7.82 (4H, m, Ar–H). IR (Nujol): 3220 cm^{-1} (NH).

Desulfurization of 3b with Raney Nickel——A mixture of **3b** (0.5 g, 2.3 mmol) and Raney nickel (2.0 g) in ethanol (30 ml) was refluxed for 3 h. The reaction mixture was filtered, and the residue was washed with ethanol. The filtrate and washings were combined, and concentrated. The residue was recrystallized from acetonitrile to give 0.15 g (37.7%) of 2-ethylaminoquinazoline (**10**), mp 96—97°C. *Anal.* Calcd for $C_{10}H_{11}N_3$: C, 69.34; H, 6.40. Found: C, 69.21; H, 6.56. PMR ($CDCl_3$): 2.30 (3H, t, CH_3), 3.56, 3.68 (2H, qq, CH_2), 5.72 (1H, br s, NH), 7.50—7.90 (4H, m, Ar–H), 9.00 (1H, s, Ar–H).

Desulfurization of 5b with Raney Nickel——Compound **5b** was worked up as described for the desulfurization of **3b** to give 0.22 g (55.3%) of 4-ethylaminoquinazoline (**11**) as colorless needles, mp 149—150°C. PMR ($CDCl_3$): 1.32 (3H, t, CH_3), 3.68, 3.76 (2H, qq, CH_2), 6.38 (1H, br s, NH), 7.25—8.04 (4H, m, Ar–H), 8.72 (1H, s, Ar–H).

Reaction of 1a with Butylamine——A mixture of **1a** (4.2 g, 20.0 mmol), butylamine (1.5 g, 20.5 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was stirred at 80—85°C for 20 min. The resulting mixture was wroked up as described for the reaction of **1a** with ethylamine to give 0.13 g of **2a**, 1.7 g of 2-butylamino-4-methylthioquinazoline (**3c**) as yellow needles (mp 59°C), 1.56 g of 4-butylamino-2-chloroquinazoline[6] (**4c**) as colorless needles (mp 115—116°C), 0.45 g of 4-butylamino-2-methylthioquinazoline (**5c**) as colorless needles (mp 99—100°C) and 0.16 g of 2,4-bis(butylamino)quinazoline[6] (**6c**) as a yellow oil (n_d^{20} 1.5631). **3c**: *Anal.* Calcd for $C_{13}H_{17}N_3S$: C, 63.12; H, 6.93; N, 16.99. Found: C, 63.08; H, 6.95; N, 16.92. PMR ($CDCl_3$): 0.55—1.95 (7H, m, $CH_2CH_2CH_3$), 2.53 (3H, s, SCH_3), 3.49 (2H, q, CH_2), 5.40 (1H, br s, NH), 6.85—7.28 (1H, m, Ar–H), 7.35—7.65 (2H, m, Ar–H), 7.65—8.00 (1H, d, Ar–H). IR (Nujol): 3250 cm^{-1} (NH). **4c**: *Anal.* Calcd for $C_{12}H_{14}N_3Cl$: C, 61.15; H, 5.99; N, 17.83. Found: C, 60.96; H, 5.93; N, 17.54. PMR ($CDCl_3$): 0.65—1.12 (3H, m, CH_3), 1.12—2.04 (4H, m, CH_2CH_2), 3.47—4.00 (2H, m, CH_2), 6.75 (1H, br s, NH), 7.21—8.18 (4H, m, Ar–H). IR (Nujol): 3250 cm^{-1} (NH). **5c**: *Anal.* Calcd for $C_{13}H_{17}N_3S$: C, 63.12; H, 6.93; N, 16.99. Found: C, 63.23; H, 7.01; N, 16.81. PMR ($CDCl_3$): 0.68—1.12 (3H, br s, CH_3), 0.68—1.90 (4H, m, CH_2CH_2), 2.58 (2H, s, SCH_3), 3.37—3.82 (2H, br q, CH_2), 5.98 (1H, br s, NH), 7.05—7.49 (1H, m, Ar–H), 7.49—7.80 (3H, m, Ar–H). IR (Nujol): 3250 cm^{-1} (NH). **6c**: PMR ($CDCl_3$): 0.59—2.25 (14H, m, $CH_2CH_2CH_3 \times 2$), 3.13—3.89 (4H, m, $CH_2 \times 2$), 5.50 (1H, br s, NH), 6.65—7.26 (2H, m, Ar–H, NH), 7.30—7.60 (2H, br d, Ar–H), 7.72—8.10 (1H, br d, Ar–H). IR (Nujol): 3300 cm^{-1} (NH).

Reaction of 1a with *tert*-Butylamine——A mixture of **1a** (4.2 g, 20.0 mmol), *tert*-butylamine (2.0 g, 27.4 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was refluxed for 3 h. The resulting mixture was worked up as described for the reaction of **1a** with ethylamine to give 1.2 g of 2-*tert*-butylamino-4-methylthioquinazoline (**3d**) as yellow needles (mp 87—89°C), followed by 0.43 g of 2-chloro-4-*tert*-butylaminoquinazoline (**4d**) as colorless needles (mp 139—141°C), and 2.8 g of **1a** was recovered. **3d**: *Anal.* Calcd for $C_{13}H_{17}N_3S$: C, 63.12; H, 6.93; N, 16.99. Found: C, 62.57; H, 6.66; N, 16.85. PMR ($CDCl_3$): 1.32 (9H, s, $CH_3 \times 3$), 2.38 (3H, s, SCH_3), 5.45 (1H, s, NH), 6.69—7.00 (1H, m, Ar–H), 7.26—7.47 (2H, m, Ar–H), 7.50—7.76 (1H, s, Ar–H). **4d**: *Anal.* Calcd for $C_{12}H_{14}N_3Cl$: C, 61.15; H, 5.99; N, 17.83. Found: C, 60.83; H, 5.86; N, 17.90. PMR ($CDCl_3$): 1.61 (9H, br s, $CH_3 \times 3$), 6.15 (1H, br s, NH), 7.32—8.00 (4H, m, Ar–H). IR (Nujol): 3290 cm^{-1} (NH).

Reaction of 1e with *sec*-Butylamine——A mixture of **1e** (4.8 g, 20.1 mmol), *sec*-butylamine (2.0 g, 27.4 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was stirred at 80—85°C for 20 min. The resulting mixture was worked up as described for the reaction of **1a** with ethylamine to give 1.0 g of 2-*sec*-butylamino-4-*tert*-butylthioquinazoline (**3e**) as yellow needles (mp 85—86°C), and 3.4 g of **1e** was recovered. **3e**: *Anal.* Calcd for $C_{16}H_{23}N_3S$: C, 66.39; H, 8.01; N, 14.52. Found: C, 66.39; H, 8.15; N, 14.63. PMR ($CDCl_3$): 0.96 (3H, t, CH_3), 1.25 (3H, d, CH_3), 1.56 (2H, m, CH_2), 1.69 (9H, s, $CH_3 \times 3$), 4.23 (1H, m, CH), 5.15 (1H, br d, NH), 7.03—7.49 (1H, m, Ar–H), 7.55—7.70 (1H, m, Ar–H), 7.96—8.19 (1H, br d, Ar–H). IR (Nujol): 3260 cm^{-1} (NH).

Reaction of 1a with Dimethylamine——A mixture of **1a** (4.2 g, 20.0 mmol), dimethylamine (50% in water, 2.5 g, 27.8 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was stirred at 80—85°C for 20 min. The resulting mixture was worked up as described for the reaction of **1a** with ethylamine to give 0.11 g of **2a**, 1.2 g of 2-dimethylamino-4-methylthioquinazoline (**3f**) as yellow needles (mp 65—66°C), 1.45 g of 2-chloro-4-dimethylaminoquinazoline (**4f**) as colorless needles mp 67°C (lit.;[6] mp 67.5°C), 0.6 g of 4-dimethylamino-2-methylthioquinazoline (**5f**) as colorless needles (mp 100—101°C) and 0.04 g of 2,4-bis(dimethylamino)quinazoline (**6f**) as a yellow oil.[6] **3f**: *Anal.* Calcd for $C_{11}H_{13}N_3S$: C, 60.24; H, 5.97; N, 19.16. Found: C, 59.92; H, 5.57; N, 18.83. PMR ($CDCl_3$): 2.58 (3H, s, SCH_3), 3.24 (6H, s, $NCH_3 \times 2$), 6.88—7.29 (1H, m, Ar–H), 7.45—7.65 (2H, m, Ar–H), 7.69—7.93 (1H, br d, Ar–H). **5f**: *Anal.* Calcd for $C_{11}H_{13}N_3S$: C, 60.24; H, 5.97; N, 19.16. Found: C, 60.01; H, 6.06; N, 18.91. PMR ($CDCl_3$): 2.58 (3H, s, SCH_3), 3.29 (6H, s, $NCH_3 \times 2$), 7.03—7.48 (1H, m, Ar–H), 7.53—7.73 (2H, m, Ar–H), 7.80—8.03 (1H, br d, Ar–H). **6f**: *Anal.* Calcd for $C_{12}H_{16}N_4$: C, 66.64; H, 7.46. Found: C, 66.26; H, 7.78.

Reaction of 1a with Dipropylamine——A mixture of **1a** (4.2 g, 20.0 mmol), dipropylamine (2.3 g, 22.8 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was refluxed for 3 h. The resulting mixture was poured into ice-water and extracted with chloroform (50 ml × 3). The extract was washed with satd. sodium chloride aq., dried over magnesium sulfate and concentrated. The residue was recrystallized from *n*-hexane to give 5.0 g (90%) of 2-dipropylamino-4-methylthioquinazoline (**3g**) as yellow needles (mp 52—54°C). *Anal.* Calcd for $C_{15}H_{21}N_3S$: C, 65.41; H, 7.69; N, 15.26. Found: C, 65.44; H, 7.61; N, 15.16. PMR ($CDCl_3$): 0.98 (6H, t, CH_3), 1.79 (4H, m, $CH_2 \times 2$), 2.66 (3H, s, SCH_3), 3.76 (4H, m, $CH_2 \times 2$), 7.09—7.39 (1H, m, Ar–H), 7.62—7.82 (2H, m, Ar–H), 7.90—8.15 (1H, br d, Ar–H).

Reaction of 1a with Diisopropylamine——A mixture of **1a** (4.2 g, 20.0 mmol), diisopropylamine (2.3 g, 22.8 mmol) and triethylamine (2.1 g, 21.0 mmol) in dimethylformamide (30 ml) was worked up as described for the reaction of **1a** with ethylamine, but **1a** was recovered quantitatively.

Reaction of 2a with Butylamine——A mixture of **2a** (2.2 g, 10.0 mmol) and butylamine (2.0 g, 27.4 mmol) in dimethylformamide (30 ml) was stirred at 80—85°C for 3 h. The reaction mixture was poured into ice-water and extracted with chloroform (30 ml × 3). The extract was washed with water, dried over magnesium sulfate and concentrated. The residue was recrystallized from acetonitrile to give 1.9 g (76.9%) of **5c** as colorless needles, mp 100°C.

Reaction of 3c with Butylamine——Compound **3c** (2.5 g) was worked up as described for the reaction of **2a** with butylamine to give 2.6 g (92.9%) of **6c** as a yellow oil.

Reaction of 5c with Butylamine——Compound **5c** (2.5 g) was worked up as described for the reaction of **3c** with butylamine, but **5c** was recovered.

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