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SYNTHESIS OF 2-ARYLQUINOXALINES: TRIARYLSTIBANE-CATALYZED OXIDATIVE CYCLIZATION OF α-HYDROXY KETONES WITH 1,2-DIAMINES UNDER AEROBIC CONDITIONS

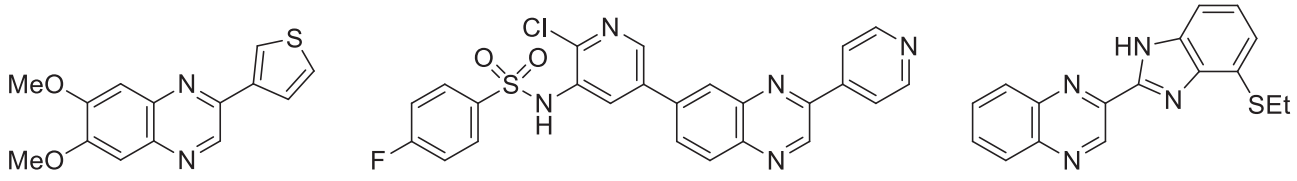
Mio Matsumura,[1] Rie Takada,[1] Yuu Ukai,[1] Mizuki Yamada,[1] Yuki Murata,[2] Naoki Kakusawa,[2] and Shuji Yasuike[1]*

[1] School of Pharmaceutical Sciences, Aichi Gakuin University, 1-100 Kusumoto-cho, Chikusa-ku, Nagoya 464-8650, Japan. [2] Faculty of Pharmaceutical Sciences, Hokuriku University, Kanagawa-machi, Kanazawa 920-1181, Japan

Abstract – The reaction of α-hydroxy ketones with 1,2-diamines in the presence of triphenylstibane (10 mol%) as catalyst led to the formation of 2-arylquinoxalines in moderate to good yield under aerobic conditions. This reaction is the first example of oxidative cyclization catalyzed by organoantimony compounds.

INTRODUCTION

Quinoxaline is an important building block for the synthesis of functional organic materials,[1,2] and biologically active compounds.[3] For examples, 2-arylquinoxalines are known for biological activities such as kinase inhibitor[4] and adenosine receptor antagonist[5] (Figure 1). Consequently, many methods have been developed for the synthesis of 2-arylquinoxalines. Among these methods, the oxidative cyclization of α-hydroxy ketones with 1,2-diamines in the presence of a transition metal or inorganic catalyst such as MnO_2,[6] $Pd(OAc)_2$,[7] HgI_2,[8] $CuCl_2$,[9] manganese oxide octahedral molecular sieves,[10] $NbCl_5$,[11] $Ga(ClO_4)_3$,[12] and CaO[13] are known.



Tyrosine kinase inhibitor PI3K/mTOR dual inhibitor Adenosine receptor antagonist

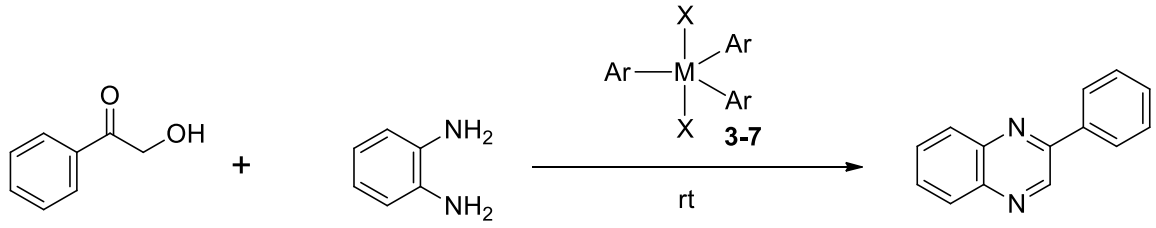
Figure 1. Examples of biologically active compound containing 2-arylquinoxaline skeleton.

On the other hand, pentavalent organoantimony compounds such as stibane oxide,[14] bromodiphenylstibane,[15] dibromotriphenylstiborane,[16] and stibane imides[17] are known to be effective for achieving the oxidation of thiols, benzyl alcohols, benzopinacol, and diaryl-α-ketoalcohols. In these reactions, however, it is inevitably necessary to add a stoichiometric or excess amount of the antimony reagent and/or the base to ensure a smooth reaction. Recently, we also reported triarylstibane to be a useful catalyst for the oxidation of benzoin to the corresponding benzyl derivative under mild conditions.[18] As a continuation of our studies on the promotion of oxidative catalytic reactions by organoantimony reagents, we now report the one-pot tandem oxidative cyclization of α-hydroxy ketones with 1,2-diamines to afford the corresponding 2-arylquinoxalines under aerobic conditions.

RESULTS AND DISCUSSION

Previously, we reported that benzoin compounds were oxidized to the corresponding benzyl derivatives in the presence of a catalytic amount of triphenylstibane (**3a**).[18] The oxidation of 2-hydroxyacetophenone (**1a**) by using triphenylstibane (**3a**) was performed before the oxidative cyclization of **1a** with *o*-phenylendiamine (**2a**). However, the oxidation of **1a** with **3a** resulted in a complex mixture and did not afford phenylglyoxal in dichloromethane at room temperature under aerobic conditions. Therefore, we first studied the effect of solvents, to search for suitable catalysts and reaction conditions for the oxidative cyclization of **1a** with **2a**. The results are shown in Table 1. Initially, we performed the reaction between **1a** and **2a** using 10 mol% of triphenylstibane (**3a**) as catalyst at room temperature under aerobic conditions to compare the effect of solvent (entries 1-5). The expected 2-phenylquinoxaline (**8**) was obtained in an excellent yield when toluene and dichloromethane were employed as the solvent, whereas tetrahydrofuran (THF), acetonitrile, and ethanol gave inferior results. Next, several antimony catalysts were also screened in toluene under the same conditions (entries 2, 6-13). Trivalent organoantimony catalysts with electron-donating and -withdrawing groups on the aromatic ring, *p*-methoxyphenyl **3b** and *p*-trifluoromethylphenyl **3c**, produced **8** in satisfactory yields (entries 6, 7). On the other hand, the 2,6-dimethoxyphenyl and mesityl derivatives (**3d**, **e**), which are sterically hindered, and the inorganic reagent (**3f**) were inactive with a low yield despite longer reaction times (entries 8-10). Moreover, the pentavalent organoantimony catalysts (**3g-i**) also produced inferior results (entries 11-13). As a result, triphenylstibane (**3a**) proved to be superior to the other antimony catalysts (**3b-i**) in terms of the reaction time, yield of **8**, and the fact that this catalyst is commercially available. A remarkable decrease in the yield of the product **8** under an argon atmosphere indicated that the presence of oxygen in the reaction medium was essential for the reaction to proceed (entry 14). The progressive addition of pure dioxygen shortened the reaction time (entry 15).

Table 1. Screening of reaction conditions[a]



1a **2a** **8**

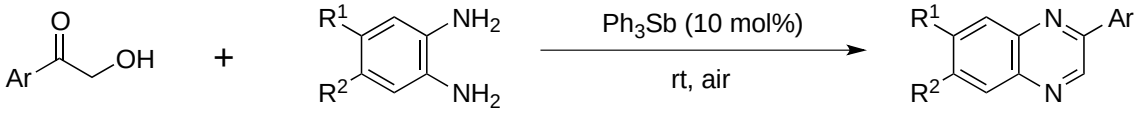
Entry	Catalyst	M	Ar	X	Solvent	Atmosphere	Time (h)	Yield (%)[b]
1	**3a**	Sb	C_6H_5	---	CH_2Cl_2	air	5	80
2	**3a**	Sb	C_6H_5	---	toluene	air	3	83
3	**3a**	Sb	C_6H_5	---	THF	air	24	52
4	**3a**	Sb	C_6H_5	---	MeCN	air	24	46
5	**3a**	Sb	C_6H_5	---	MeOH	air	24	36
6	**3b**	Sb	4-$MeOC_6H_4$	---	toluene	air	2	78
7	**3c**	Sb	4-$CF_3C_6H_4$	---	toluene	air	4	80
8	**3d**	Sb	2,6-$(MeO)_2C_6H_4$	---	toluene	air	24	43
9	**3e**	Sb	mesityl	---	toluene	air	24	41
10	**3f**	Sb	Br	---	toluene	air	24	32
11	**3g**	Sb	C_6H_5	C_6H_5	toluene	air	24	54
12	**3h**	Sb	C_6H_5	OAc	toluene	air	12	65
13	**3i**	Sb	C_6H_5	Cl	toluene	air	12	64
14	**3a**	Sb	C_6H_5	---	toluene	argon	24	31
15	**3a**	Sb	C_6H_5	---	toluene	O_2	0.5	75
16[c]	**3a**	Sb	C_6H_5	---	toluene	air	24	48
17	**4**	N	C_6H_5	---	toluene	air	24	20
18	**5**	P	C_6H_5	---	toluene	air	24	36
19	**6**	As	C_6H_5	---	toluene	air	24	34
20	**7**	Bi	C_6H_5	---	toluene	air	24	51

[a]All reaction were carried out using **1a** (1 mmol), **2a** (1.2 mmol), catalyst (10 mol%) and solvent (6 mL). [b]Isolated yield. [c]Ph_3Sb (5 mol%) was used.

Reactions under aerobic conditions are simple and easy to operate. It is apparent that the reaction is sensitive to catalyst loading, as decreasing the loading of triphenylstibane from 10 mol% to 5 mol% gave a significantly reduced yield of **8** (48%, entry 16). Finally, the reaction of **1a** with **2a** was repeated to compare the reactivity with 10 mol% of pnictogen reagents as the catalyst (entries 2, 17-20). The results clearly showed that triphenylstibane (**3a**) is the most effective catalyst to prepare the expected product (**8**). Thus, the most favorable reaction conditions were determined to be as follows: 2-hydroxyacetophenone (**1a**) was treated with *o*-phenylendiamine (**2a**) with the use of triphenylstibane (**3a**) as catalyst in toluene or dichloromethane at room temperature under aerobic conditions.

The sufficiency and generality of this protocol was evaluated by examining the reactions of various α-hydroxy ketones (**1a-h**) and *o*-phenylendiamines (**2a-h**), and the results are summarized in Table 2. The reaction solvent was selected based on the solubility of **1** and **2**. α-Hydroxy ketones (**1a-f**) bearing electron-donating or –withdrawing group on the aromatic ring gave the corresponding 2-arylquinoxalines (**9**-**13**) in moderate to good yields (entries 1-5). The reaction of the naphthyl and heteroaryl derivatives (**1g**, **h**) also obtained the expected products **14** and **15**, respectively (entries 6, 7). Unfortunately, the reaction of hydroxyacetone and methyl glycolate resulted in a complex mixture and did not afford the desired quinoxalines. The results indicate that this oxidative cyclization has high substrate specificity and is effective for converting α-hydroxy ketones containing an aryl group into the corresponding 2-arylquinoxalines. Various *o*-phenylendiamines (**2b-h**) were reacted with 2-hydroxyacetophenone (**1a**) under the same reaction conditions. The diamine (**2b-e**) with electron-donating and -withdrawing group afforded the product in moderate to good yields (entries 8-11). The reaction of 2,3-diaminonaphthalene (**2f**) with **1a** was also gave the benzo[*g*]quinoxaline (entry 12). The reactions of **2b**, **e**, **f**, all of which were least soluble in the solvent required prolonged reaction times. The mono-substituted diamines (**2g** and **2h**) afforded mixtures of two regioisomers, of which the 6-substituted isomer (**b**) was the major product (entries 13, 14). When the aliphatic, instead of aromatic, diamine was used the reaction of **1a** with (±)-*trans*-1,2-diaminocyclohexane (**23**) gave the corresponding hexahydroquinoxaline (**24**) in 24% yield (Scheme 1). The product (**24**) was unstable for purification by chromatography despite using amino-functionalized silica and aluminum oxide, and therefore isolated in a disappointing yield.

Table 2. Synthesis of 2-arylquinoxalines[a]

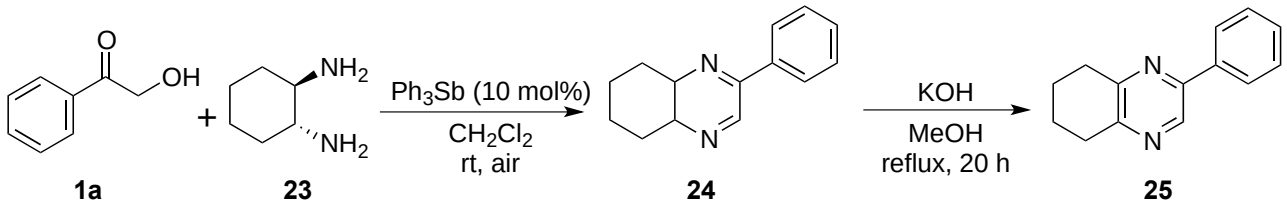


1a-h **2a-h** **9-22**

Entry	**1**	Ar	**2**	R^1	R^2	Solvent	Time (h)	Product	Yield (%)[b]
1	**1b**	4-$MeOC_6H_4$	**2a**	H	H	toluene	3	**9**	74
2	**1c**	4-MeC_6H_4	**2a**			toluene	3	**10**	70
3	**1d**	4-BrC_6H_4	**2a**			CH_2Cl_2	5	**11**	72
4	**1e**	4-CNC_6H_4	**2a**			CH_2Cl_2	5	**12**	60
5	**1f**	4-$CF_3C_6H_4$	**2a**			toluene	3	**13**	68
6	**1g**	2-naphthyl	**2a**			toluene	3	**14**	71
7	**1h**	2-thienyl	**2a**			toluene	4	**15**	74
8	**1a**	C_6H_5	**2b**	MeO	MeO	CH_2Cl_2	24	**16**	50
9	**1a**		**2c**	Me	Me	toluene	6	**17**	72
10	**1a**		**2d**	Br	Br	toluene	3	**18**	75

11	**1a**	**2e**	CN	CN	CH_2Cl_2	48	**19**	59
12	**1a**	**2f**	naphthyl		CH_2Cl_2	24	**20**	80
13	**1a**	**2g**	MeO	H	CH_2Cl_2	5	**21a**[c]	13[d]
							21b[c]	42[d]
14	**1a**	**2h**	CF_3	H	toluene	3	**22a**[c]	27[d]
							22b[c]	52[d]

[a] All reaction were carried out using **1** (1 mmol), **2** (1.2 mmol), **3a** (10 mol%) and solvent (6 mL). [b] Isolated yield. [c] **21a**: 7-methoxy-2-phenylquinoxaline, **21b**: 6-methoxy-2-phenylquinoxaline, **22a**: 2-phenyl-7-trifluoromethyl-quinoxaline, **22b**: 2-phenyl-6-trifluoromethylquinoxaline. [d] Determined by ^{1}H NMR.



Scheme 1. Synthesis of hexahydro- and tetrahydroquinoxaline (**24, 25**)

Furthermore, we examined the one-pot two-step reaction for the preparation of tetrahydroquinoxaline (**25**) from **1a** and **23** by using a Ph_3Sb-catalyzed cyclization and the aromatization[19] that was previously published by Raw *et al*. This method involved the addition of KOH and MeOH after the completion of the cyclization step to the same reaction flask, after which the reaction mixture was heated under reflux for 20 h to afford **25** in 45% yield.

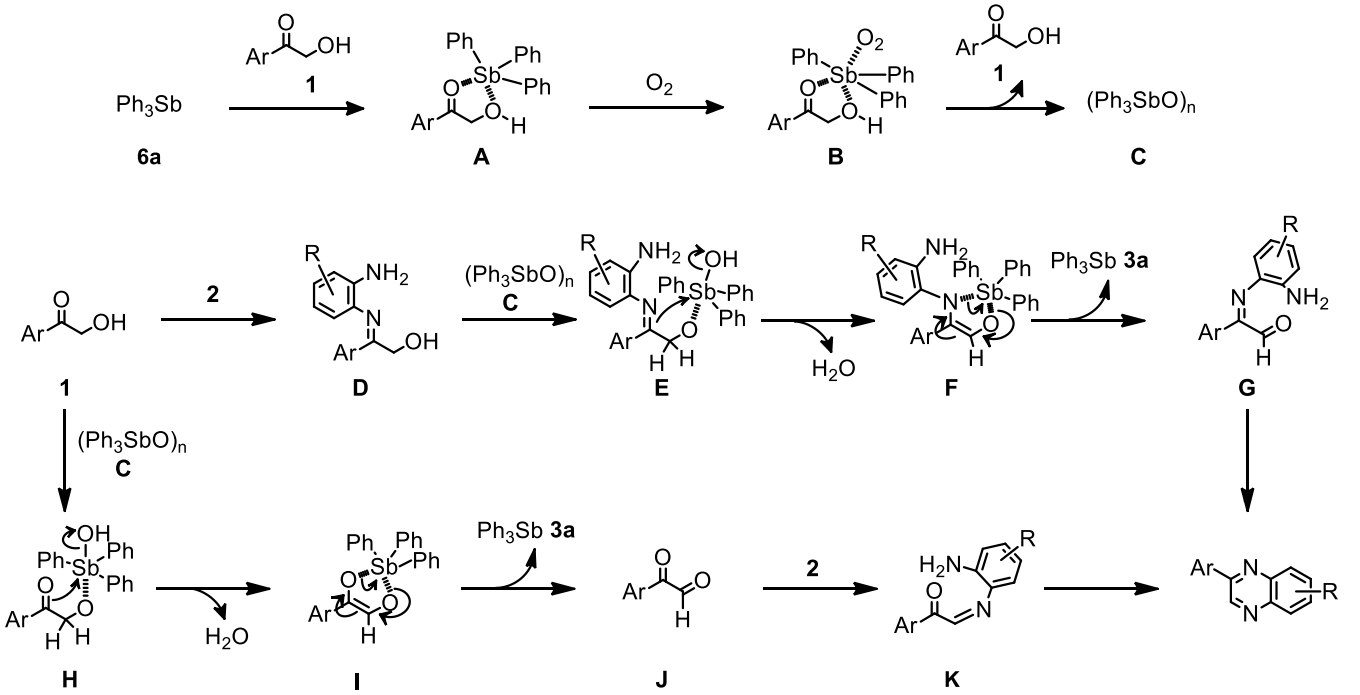


Figure 2. Possible mechanism

The reaction mechanism of this oxidative cyclization is unclear at present. The catalytic cycle of this reaction would be similar to that of the oxidation of diaryl-α-keto-alcohols reported by us.[18] Furthermore, it is known that organoantimony compounds formed intermolecular non-bonding interaction between the antimony atom and the nitrogen (N) and/or oxygen (O) atoms in other molecules.[20] Possible mechanisms of the reaction between α-hydroxy arylketones and *o*-phenylendiamines in the formation of quinoxalines are depicted in Figure 2. The initial step of this reaction involves the oxidation of **3a** to the SbPh-oxo species (oxide) (**C**) with aerobic oxygen.[18] Actually, the reaction of **1a** with **2a** by using a catalytic amount of tripehenylstibane oxide (Ph_3SbO) brought about the same cyclization to afford **8** in high yield (84%). On the other hand, **1** is transformed into α-iminoalcohol **D** by diamine (**2**). The oxide (**C**) formed as mentioned above may react with **D** to produce pentavalent antimony intermediates **E** and **F** which undergo reductive antimony(III) elimination to provide α-iminoketone **G** and regenerate **3a**. Finally, cyclodehydration occurs in the transformation from **G** to afford 2-arylquinoxalines. Another pathway through **1** is also oxidized by antimony oxide **C** and generates arylglyoxal **J** *via* **H** and **I**. **J** readily react with diamine (**2**) to give intermediates **K** and/or **G**, which undergo cyclodehydration to form the 2-arylquinoxalines. We probed the mechanism of this transformation by using NMR spectroscopy. The spectra clearly showed an increase in the products as the starting materials decreased. However, the proposed intermediate species **A**~**K**, including **G** and **J**, could not be observed on the NMR time scale.

CONCLUSION

In conclusion, we found triphenylstibane to be a new class of catalyst for the synthesis of 2-arylquinoxalines, and it is the first examine of oxidative cyclization catalyzed by organoantimony compounds. The reaction procedure is an experimentally simple oxidative cyclization of α-hydroxy ketones with 1,2-diamines that can be conducted either toluene or dichloromethane at room temperature under aerobic conditions. The antimony reagents, such as triphenylstibane have low toxicity,[21] therefore hold great promise for organic synthetic reagents. The synthetic application of this reaction and a detailed investigation of the reaction mechanisms are in progress.

EXPERIMENTAL

Unless otherwise indicated, chemicals and solvents were purchased from commercial suppliers and used without further purification. All chromatographic separations were accomplished with Silica Gel 60N (Kanto Chemical Co., Inc.) or NH Silica Chromatorex DM2035 (Fuji Silysia Chemcal, Ltd.). Thin-layer chromatography (TLC) was performed with MERCK TLC Silicagel 60 F254 and MERCK TLC aluminum oxide F254, neutral. Melting points were taken on a Yanagimoto micro melting point hot-stage apparatus. ^{1}H NMR (TMS: δ: 0.00 ppm as an internal standard) and ^{13}C NMR ($CDCl_3$: δ: 77.00 ppm as an

internal standard) spectra were recorded on a JEOL JNM- AL400 (400 MHz and 100 MHz) spectrometers in $CDCl_3$. Mass spectra (MS) were obtained on a JEOL JMS-SX-102A instrument.

General procedure for the synthesis of 2-arylquinoxaline derivatives (8-22 and 24). Triphenylstibane (35.5 mg, 0.1 mmol, 10 mol%) and diamine **2** (1.2 mmol) were added to a solution of α-hydroxy ketone **1** (1 mmol) in toluene (6 mL) under air. The solution was stirred at room temperature and monitored by TLC. The reaction mixture was concentrated under reduced pressure and the residue was purified by column chromatography (CH_2Cl_2) on silica gel. The products were confirmed by comparison of mp, NMR data, and MS spectra with that in the literature. **8**[22]: Yellow needles (from MeOH), mp 74-76 ˚C (lit. 75-78 ˚C), **9**[22]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 100-102 ˚C (lit. 97-100 ˚C), **10**[22]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 93-96 ˚C (lit. 93-96 ˚C), **11**[22]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 135-137 ˚C (lit. 138 ˚C), **12**[22]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 191-193 ˚C (lit. 193-195 ˚C), **13**[23]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 145.5-146.5 ˚C (lit. 142-143 ˚C), **14**[24]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 140-142.5 ˚C (lit. 141-142 ˚C), **15**[24]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 120-121.5 ˚C (lit. 118-119 ˚C), **16**[25]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 130-133 ˚C (lit. 134 ˚C), **17**[26]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 126-128 ˚C (lit. 127-129 ˚C), **20**[25]: Colorless needles (from CH_2Cl_2-*n*-hexane), mp 160-162 ˚C (lit. 163 ˚C), **24**[19]: Pale yellow oil. Yields of compounds **21a**, **b**[27] and **22a**, **b**[28] were determined by ^{1}H NMR because they could not be separate by column chromatography.

6,7-Dibromo-2-phenylquinoxaline (18): Colorless needle (from CH_2Cl_2-*n*-hexane), mp 173-176 ˚C; LRMS (EI) *m/z*: 364 ($M+2^+$, 100%), 362 (M^+, 50%), 364 ($M+4^+$, 50%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.58 (m, 3H), 8.17 (dd, 2H, J = 7.5, 1.1 Hz), 8.40 (s, 1H), 8.44 (s, 1H), 9.30 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 126.0, 126.9, 127.5, 129.2, 130.8, 133.2, 133.6, 135.9, 140.7, 141.5, 144.3, 152.6. Anal. Calcd for $C_{14}H_8Br_2N_2$: 46.19; H, 2.22; N, 7.70. Found: C, 46.32; H, 2.26; N, 7.58.

2-Phenylquinoxaline-6,7-dicarbonitrile (19): Pale yellow plate (from CH_2Cl_2-*n*-hexane), mp 275-277 ˚C; LRMS (EI) *m/z*: 256 (M^+, 100%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.65 (m, 3H), 8.25 (dd, 2H, J = 5.0, 1.3 Hz), 8.61 (s, 1H), 8.63 (s, 1H), 9.56 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 113.7, 114.9, 115.0, 128.1, 129.6, 132.1, 134.8, 137.0, 137.3, 142.0, 143.1, 147.2, 155.4. Anal. Calcd for $C_{16}H_8N_4$: C, 74.99; H, 3.15; N, 21.86. Found: C, 74.12; H, 3.09; N, 21.75.

One-pot two-step reaction for the synthesis of 2-phenyl-5,6,7,8-tetrahydroquinoxaline (25). Triphenylstibane (35.5 mg, 0.1 mmol, 10 mol%) and *trans*-1,2-diaminocyclohexane (136 mg, 1.2 mmol) were added to the solution of 2-hydroxyacetophenone (199 mg, 1 mmol) in CH_2Cl_2 (6 mL) under air, and stirred at room temperature. After TLC showed the disappearance the 2-hydroxyacetophenone, a solution

of potassium hydroxide (112 mg, 2 mmol) in MeOH (5 mL) was added. The mixture was heated under reflux for 20 h. The product was extracted by CH_2Cl_2 washed with water. The organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to column chromatography on NH-silica gel (CH_2Cl_2) to give **25** as colorless oil (94.5 mg, 0.45 mmol, 45%). The product was confirmed by comparison of NMR data, and MS spectra with that in the literature.[19]

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