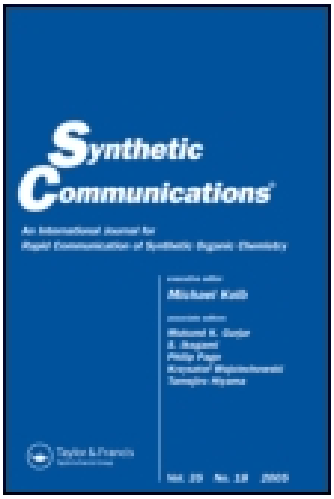

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Vilsmeier Reagent: An Efficient Reagent for the Transformation of 2-Aminobenzamides into Quinazolin-4(3H)-one Derivatives

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VILSMEIER REAGENT: AN EFFICIENT REAGENT FOR THE TRANSFORMATION OF 2-AMINOBENZAMIDES INTO QUINAZOLIN-4(3*H*)-ONE DERIVATIVES

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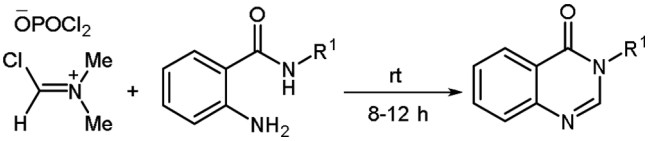
GRAPHICAL ABSTRACT



Abstract *Clean and easy preparation of quinazolin-4(3H)-one derivatives using 2-aminobenzamides and Vilsmeier reagent is described. 2-Aminobenzamides were converted into the corresponding quinazolinones under mild and efficient conditions, in good yields without undesirable by-products.*

[Supplementary materials are available for this article. Go to the publisher's online edition of Synthetic Communications® *for the following free supplemental resource(s): Full experimental and spectral details.]*

Keywords 2-Aminobenzamides; heterocycles; isotoic anhydride; 4(3*H*)-quinazolinones; Vilsmeier reagent

INTRODUCTION

The Vilsmeier reagent **3** (Scheme 1) was introduced by Anton Vilsmeier and Albrecht Haack in 1927. For the formylation of activated electron rich aromatic compounds or olefins,[1] it has been attractive for synthetic chemists to prepare different types of heterocyclic compounds.[2] This efficient reagent is easily synthesized by the reaction of *N,N*-dimethylformamide **1** (DMF) and chlorinating agents such as phosphorus oxychloride ($POCl_3$) **2**, phosphorus trichloride (PCl_3), oxalyl chloride $(COCl)_2$, and thionyl chloride ($SOCl_2$) (Scheme 1).[3]

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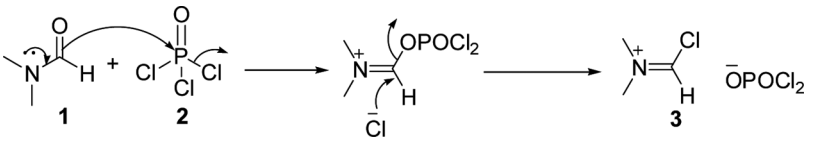
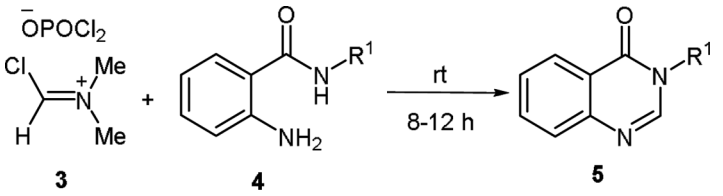


Scheme 1. Preparation of Vilsmeier reagent **3**.



Scheme 2. Synthesis of 4(3*H*)-quinazolinones **5**.

Successful and versatile transformations in organic chemistry using Vilsmeier reagent or its modified form,[4] such as formylation,[5] cyclohaloaddition,[2a] cyclization,[6] and ring annulations,[7] led to the formation of various heterocycles. The results stimulated us to investigate synthesis of 4(3*H*)-quinazolinones as biologically important compounds using Vilsmeier reagent and to present an efficient surrogate for the synthesis of the title compounds **5** (Scheme 2).

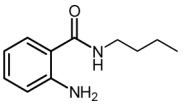
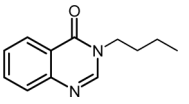
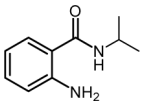
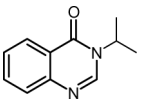
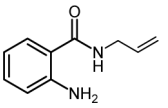
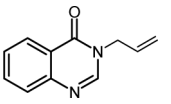
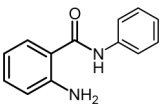
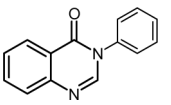
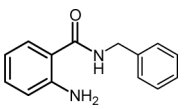
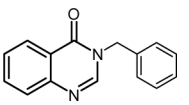
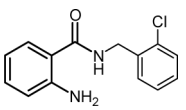
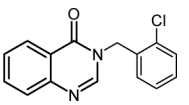
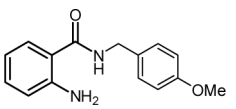
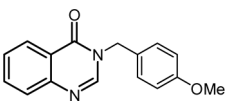
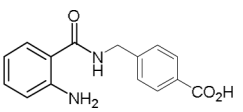
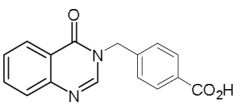
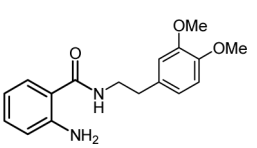
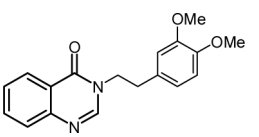
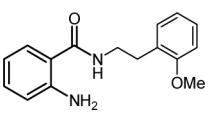
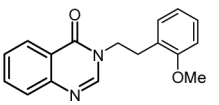
4(3*H*)-Quinazolinones are an important class of *N*-heterocycles owing to their presence in the numerous natural products and synthetic organic compounds along with proficient various pharmacological properties.[8] They have been used as antiulcer,[9] anti-inflammatory,[10] anticancer,[11] hypolipidemic,[12] and anticonvulsant agents.[13] Also quinazolin-4(3*H*)-one skeleton is found in a range of biologically important natural products including febrifugine and isofebrifugine, which possess antimalarial activity.[14] Although a wide range of routes has been reported for the synthesis of 4(3*H*)-quinazolinones,[15] biological properties of this class of heterocycles has encouraged us to design a simple and efficient approach for their preparation.

RESULTS AND DISCUSSION

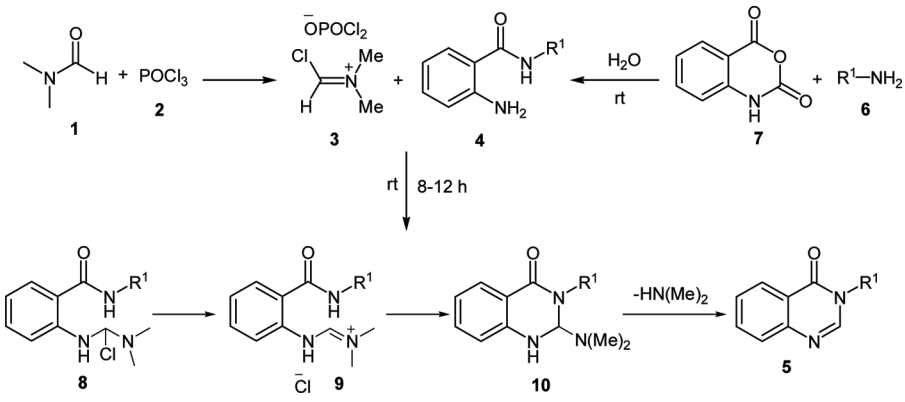
We recently reported novel synthesis of 1,4-benzodiazepine-3,5-diones under Bargellini reaction conditions,[16] benzo[*d*][1,3,2]diazaborinin-4(1*H*)-one,[17] and 3-alkyl-2-(alkylamino)quinazolin-4(3*H*)-one,[18] using 2-aminobenzamide derivatives as an proficient ambident nucleophile. In continuation of our studies on the synthesis of different kinds of heterocycles,[19] herein, we report synthesis of 3-substituted-4(3*H*)-quinazolinones **5** using 2-aminobenzamides **4** and Vilsmeier reagent **3** at ambient temperature (Scheme 2).

Our investigations were initiated by preparing 2-aminobenzamide derivatives **4** and Vilsmeier reagent **3**. According to our previous works,[16–18] all 2-aminobenzamides **4** were synthesized by a simple and green procedure. Equivalent amounts of

Table 1. Synthesis of 4(3*H*)-quinazolinones **5**

Entry	2-Aminobenzamides **4**	Product **5**		Mp (°C) Observed	Mp (°C) Reported	Yield (%)[a]
1			**5a**	72–74	70–72[15a]	80
2			**5b**	85–87	87–88[15f]	80
3			**5c**	68–70	65[15e]	75
4			**5d**	145–147	141–142[15a]	65
5			**5e**	155–156	154–155[15h]	82
6			**5f**	117–119	116[15c]	75
7			**5g**	137–138	134–135[15a]	80
8			**5h**	269–270	—	75
9			**5i**	109–110	107–109[15g]	80
10			**5j**	120–122	—	77

[a]Isolated yields.



Scheme 3. Proposed sequences for the synthesis of 4(3*H*)-quinazolinones **5**.

amines **6** and isatoic anhydride **7** were reacted in water at room temperature for 2 h. After this time the precipitated products were filtered and dried. The obtained 2-aminobenzamides **4** were pure and used for further reactions.

Vilsmeier reagent **3** was simply prepared by stirring a mixture of dimethylformamide DMF **1** (4.6 mL, 60 mmol) and phosphorus oxychloride **2** (5.6 mL, 60 mmol) at room temperature for 30 min. Then 2-amino-*N*-benzylbenzamide **4e** (2 mmol) was added to reaction mixture and the reaction continued at room temperature for 8 h. After completion of reaction, the corresponding product, **5e**, was obtained in 82% yield. We found that in situ preparation of reagent is sufficient to achieve the condensation reaction and no by-product is obtained. Any salt which formed during the reaction was removed by simple aqueous workup.

Good results with simple and clean procedure encouraged us to expand our method to utilize different 2-aminobenzamide derivatives **4** in these reactions (Scheme 2) and synthesize various 4(3*H*)-quinazolinones **5** (Table 1). The structures of all products were confirmed by ^{1}H NMR and ^{13}C NMR spectroscopy and compared with those reported in the literature.[15]

A proposed sequence is depicted schematically in Scheme 3. The initial step involves nucleophilic attack by an amino group in 2-aminobenzamide derivatives **4** at the electrophilic carbon of Vilsmeier reagent **3** to form **8** and then iminium salt **9**. Intramolecular cyclization (**10**) followed by the loss of $NH(Me)_2$ group leads to the formation of product **5**.

CONCLUSION

In conclusion, we have implemented an efficient, clean, and user-friendly approach for the synthesis of diversely 3-substituted-4(3*H*)-quinazolinone derivatives. This approach involves the condensation reaction of 2-aminobenzamides, derived from isatoic anhydride and various amines; and Vilsmeier reagent at room temperature without any by-products. Although previously reported procedures gave the title compounds in good yields, our method offered them from available starting material without utilizing expensive catalysts or microwave irradiation.

EXPERIMENTAL

Melting points were taken on a Kofler hot stage apparatus and are uncorrected. ^{1}H-and ^{13}C(NMR) spectra were recorded on Bruker FT-500 or 400, using tetramethylsilane (TMS) as an internal standard. The infrared (IR) spectra were obtained on a Nicolet Magna FTIR 550 spectrophotometer (in KBr). All reagents and solvents were obtained from Merck and Aldrich and used without any purification. Thin-layer chromatography (TLC) was performed using silica gel 60/Kieselguhr F254 precoated on aluminum sheets (thickness 0.2 mm), commercially available from Merck. Visualization of spots on TLC plate was accomplished with ultraviolet (UV) light.

Synthesis of 4(3*H*)-Quinazolinone Derivatives 5: General Procedure

A mixture of dimethyl formamide DMF **1** (60 mmol, 4.6 mL) and phosphorus oxychloride $POCl_3$ **2** (60 mmol, 5.6 mL) was stirred at room temperature for 35 min. Then, 2-aminobenzamide derivatives **4** (2 mmol) were added to the reaction mixture and the stirring was continued for 8–12 h. Upon completion of reaction (checked by TLC), the solution was added to water and extracted with ethyl acetate (3×50 mL). The organic layer was separated, dried (Na_2SO_4), and concentrated in vacuo to yield the crude product, which was purified by recrystallization from ethyl acetate.

3-Butylquinazolin-4(3*H*)-one (5a)

Yield: 80%, yellow crystals. Mp 72–74 °C (70–72 °C)[15a]; ^{1}H NMR (DMSO-d_6, 500 MHz): $\delta_H = 8.42$ (s, 1H, H_2), 8.16 (dd, $J = 8.0$, 1.5 Hz, 1H, H_5), 7.81 (dt, $J = 8.0$, 1.5 Hz, 1H, H_7), 7.68 (d, $J = 8.0$ Hz, 1H, H_8), 7.54 (t, $J = 8.0$ Hz, 1H, H_6), 3.98 (t, $J = 7.4$ Hz, $N\underline{CH_2}CH_2CH_2CH_3$), 1.68 (quintet, $J = 7.4$ Hz, $NCH_2\underline{CH_2}CH_2CH_3$), 1.31 (sextet, $J = 7.4$ Hz, $NCH_2CH_2\underline{CH_2}CH_3$), 0.91 (t, $J = 7.4$ Hz, NCH_2CH_2-$CH_2\underline{CH_3}$). Anal. calcd. for $C_{12}H_{14}N_2O$: C, 71.26; H, 6.98; N, 13.85. Found: C, 71.51; H, 7.14; N, 13.98.

3-(4-Methoxybenzyl)quinazolin-4(3*H*)-one (5g)

Yield: 80%, yellow crystals. Mp 137–138 °C (134–135 °C)[15a]; ^{1}H NMR (DMSO-d_6, 500 MHz): $\delta_H = 8.59$ (s, 1H, H_2), 8.16 (d, $J = 7.7$ Hz, 1H, H_5), 7.84 (t, $J = 7.7$ Hz, 1H, H_7), 7.70 (d, $J = 7.7$ Hz, 1H, H_8), 7.56 (t, $J = 7.7$ Hz, 1H, H_6), 7.36 (d, $J = 8.0$ Hz, 2H, $H_{2'}$, $H_{6'}$), 6.91 (d, $J = 8.0$ Hz, 2H, $H_{3'}$, $H_{5'}$), 5.13 (s, 2H, NCH_2), 3.72 (s, 3H, OCH_3). Anal. calcd, for $C_{16}H_{14}N_2O_2$: C, 72.16; H, 5.30; N, 10.52. Found: C, 71.97; H, 5.61; N, 10.32.

SUPPORTING INFORMATION

Full experimental details and ^{1}H and ^{13}C NMR spectra are available. This material can be found via the Supplementary Content section of this article's Web page.

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