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Original scientific paper

PEG-mediated synthesis of 6-pyrazinyl-/fused pyrazinylquinazolin-4(3*H*)-ones using Castro–Stephen coupling, oxidation and cyclocondensation reactions

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Abstract: A PEG-mediated green synthesis of 6-pyrazinyl-/fused pyrazinylquinazolin-4(3*H*)-ones was developed starting from 6-iodo-3-methyl-2-phenylquinazolin-4(3*H*)-one by means of quinazolinone-based internal alkyne/1,2--diketone as intermediates using the Castro–Stephen coupling reaction/potassium permanganate mediated oxidation and cyclocondensation reactions.

Keywords: $KMnO_4$; bisazaheterocycle; 1,2-diamine; 1,2-diketone.

INTRODUCTION

Quinazolin-4(3*H*)-one either of natural or synthetic origin, is one among the most frequently encountered heterocyclic compounds in the field of medicinal chemistry, polymer chemistry and dyes. Particularly, quinazolin-4(3*H*)-one based C–C linked unsymmetrical bisheterocyclic compounds exhibit a wide range of biological activities. For example, 2-heterylquinazolin-4(3*H*)-ones exhibit antitubercular,[1] antimicrobial,[2] analgesic,[3] anti-inflammatory,[3] antiulcer,[4] anticonvulsant,[5] antihypertensive,[6] antiplatelet,[7] anticancer[8] and antifeedent activities.[9] In addition, they have a prominent application in polymer chemistry as heat stable epoxy resins, fiber reactive dyes and polymers.[10] A literature survey also revealed the importance of 6-heteryl-quinazolin-4(3*H*)-ones in pharmaceutical chemistry as antihypertensive,[11] antiviral,[12] antitumor[13] and TGFβ type 1 receptor inhibitor to treat many fibrosis.[14] Additionally, these act as size expanded nucleoside which in turn are useful in the study of steric effects of DNA–protein interactions and also served as building blocks for a large size genetic system.[15]

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On the other hand, over many years, the pyrazines and fused pyrazines, such as quinoxaline and pyridopyrazine compounds, have been a source of great interest to organic, medicinal, and material scientists due to their display of diverse biological properties,[16] such as antibacterial, antiviral, antihelmintic, anti-inflammatory and anticancer activity.

Poly(ethyleneglycol-400) (PEG-400), the most commercially important type of polyether, has drawn great attention in recent years, due to its low toxicity, unique solubility and low cost. It is widely used as a green solvent and a heterogeneous catalyst in organic reactions. PEG, considered as acyclic crown ether analogue, has been developed as green phase transfer catalyst (PTC), an alternative to quaternary ammonium salts and crown ethers, and as a solvent promoter for various reactions. A literature survey revealed the extensive use of PEG-400 as a PTC in nucleophilic substitution,[17] oxidation[18] and reduction[19] reactions. In some instances, the advantage of the use of PEG-400 as an alternative to quaternary ammonium salts resulted in higher yields of the products and shorter duration of the reaction time in phase transfer reactions.

In view of diverse biological activities of pyrazines/fused pyrazines and quinazolin-4(3*H*)-one scaffold containing compounds, interest in the synthesis of C–C linked unsymmetrical bisheterocyclic compounds, wherein, the pyrazine ring is linked to the 6-position of quinazolin-4(3*H*)-one, developed. Herein, the PEG-mediated synthesis of 6-pyrazinyl-/fused pyrazinylquinazolin-4(3*H*)-ones, starting from 6-iodo-quinazolin-4(3*H*)-one in good to high yields is reported. The key intermediates, quinazolin-4(3*H*)-one-based alkyne was generated in a Castro–Stephen coupling reaction. One-step oxidation and cyclocondensation reactions were performed in PEG-400 with improved yields.

EXPERIMENTAL

General

IR spectra for all the compounds were recorded in solid KBr on an Infracold model 337 Perkin–Elmer instrument. Melting points were measured in open capillary tubes and are uncorrected. The ^{1}H- and ^{13}C-NMR spectra of the synthesized compounds were recorded at 300 and 75 MHz, respectively, using a Bruker Avance 400 MHz NMR spectrometer in $CDCl_3$ solvent and the chemical shifts are expressed in δ / ppm relative to TMS as internal standard and coupling constants (*J*) are in Hz. Spin multiplicities were shown as *s* (singlet), *t* (triplet), *q* (quartet) and *m* (multiplet). Mass analysis was performed on quadruple–time of flight (Q–Tof) mass spectrometer using electron spray ionization (ESI) in the positive mode. TLC was performed using aluminum sheets precoated with silica gel 60 F_{254}.

Analytical and spectral data of the compounds are given in Supplementary material to this paper.

Synthesis

*Procedure for the preparation of 3-methyl-2-phenyl-6-(phenylethynyl)quinazolin-4(3*H*)--one (**2**).* A mixture of 6-iodo-3-methyl-2-phenylquinazolin-4(3*H*)-one (1.0 g, 2.76 mmol) and copper(I) phenylacetylide (0.55 g, 3.32 mmol) was refluxed in pyridine (75 mL) under a nitro-

gen atmosphere for 12 h. Then, the reaction mixture was cooled and diluted with water. The resulting solid was filtered off and purified by column chromatography over silica gel using a mixture of ethyl acetate and pet-ether (3:7) as the eluent to give a light yellow amorphous solid 3-methyl-2-phenyl-6-(phenylethynyl)quinazolin-4(3*H*)-one (**2**). Yield: 0.69 g, 75 %; m.p.: 151–153 °C.

*Procedure for the preparation of 1-(3-methyl-4-oxo-2-phenyl-3,4-dihydroquinazolin-6-yl)-2-phenylethane-1,2-dione (**3**).* To a solution of alkyne **2** (0.080 g, 0.24 mmol) in 3 mL of a mixture of PEG, water, dichloromethane (1:1:1) was added portion wise potassium permanganate (0.12 g, 0.72 mmol). The resulting solution was stirred at 40 °C for 2.5 h to complete the reaction. The precipitated MnO_2 in solution was reduced to soluble Mn^{2+} by adding $NaNO_2$ (0.060 g) and 10 % H_2SO_4 (0.62 mL) in small portions. The combined contents were extracted with ethyl acetate, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by silica gel column chromatography using (1:1) ethyl acetate and pet-ether as the eluent to give **3** as white powder. Yield: 70 mg, 80 %; m.p.: 161–163 °C.

*Preparation of 3-methyl-2-phenyl-6-pyrazinyl/fused pyrazinylquinazolin-4(3*H*)-ones (**5**, 7, **9a**–**f** and **9a′**–**f′**)*

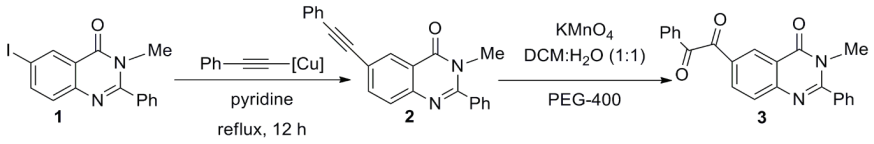
General procedure I. To a solution of 1,2-diketone **3** (0.20 g, 0.55 mmol) in acetic acid (5 mL) and water (1 mL), the appropriate 1,2-diamine (**4**, **6** and **8a–f**, 0.55 mmol) was added and the resulting solution was stirred at 80 °C. After completion of the reaction (monitored by TLC), the solution was cooled, poured into ice-cold water and extracted with ethyl acetate. The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo*. The resulting residue was purified by silica gel column chromatography using a mixture of ethyl acetate and pet-ether (4:6) as the eluent to give the corresponding bisazaheterocycles **5**, **7**, **9a**–**f** and **9a′–f′**.

General procedure II. To a solution of 1,2-diketone **3** (0.20 g, 0.55 mmol) in PEG-400:water (3:1 volume ratio), the appropriate 1,2-diamine (**4, 6** and **8a**–**f**, 0.060 g, 0.55 mmol) was added and the resulting solution stirred at 80 °C. After completion of the reaction, the solution was cooled and the solid that settled was filtered, and the solid purified by silica gel column chromatography using a mixture of ethyl acetate and pet-ether (4:6) as the eluent to give the corresponding bis*aza*-heterocycles **5**, **7**, **9a**–**f** and **9a′**–**f′**.

The recovered PEG-400/water solution was reused for a further four cycles for the synthesis of compound **7**.

RESULTS AND DISCUSSION

6-Iodo-3-methyl-2-phenylquinazolin-4(3*H*)-one (**1**), the key material for quinazolin-4(3*H*)-one based internal alkynes, was prepared using a reported procedure[20] (Scheme 1). At the outset, only a few reports were available for the preparation of quinazolin-4(3*H*)-one based internal alkynes.[21–23]



Scheme 1. Preparation of 1-(3-methyl-4-oxo-2-phenyl-3,4-dihydroquinazolin-6-yl)-2-phenylethane-1,2-dione (**3**).

In particular, previously, the synthesis of 2-benzoyl-6-(phenylethynyl) quinazolin-4(3*H*)-one[24] was achieved employing a Sonogashira coupling reaction. Alternatively, the utilization of the Castro–Stephens coupling reaction was considered for the synthesis of 3-methyl-2-phenyl-6-(phenylethynyl) quinazolin-4(3*H*)-one (**2**) due to it being straightforward and efficient. Remarkably, this process avoids the use of palladium salts. To this end, compound **1** was treated with copper(I) phenylacetylide in pyridine under reflux for 12 h to give alkyne **2**.

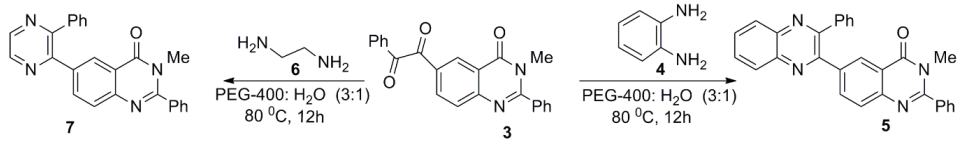
To the best of our knowledge, alkyne **2** is a new compound. It was characterized by its IR, HRMS, ^{1}H- and ^{13}C-NMR spectral data. The peaks at ν_{max} 2200 cm^{-1} in the IR and at δ 90.9 and 88.4 ppm in ^{13}C-NMR spectra of **2** indicate the presence of an acetylenic group. Furthermore, in the ^{1}H-NMR spectrum, upfield shifts of the signals were observed for C-5H and C-7H of quinazolin-4(3*H*)-one (δ 8.49 and 7.85 ppm) in comparison with the corresponding signals in **1** (δ 8.65 and 8.01 ppm). This upfield shift is rationalized based on the diamagnetic anisotropic effect of acetylene moiety. 2-Phenyl-3-methyl 6-iodoquinazolin-4(3*H*)-one showed the $[M+H]^+$ ion peak at *m/z* 337.1327 in the high-resolution mass spectrum, corresponding to the molecular chemical formula of $C_{23}H_{16}N_2O$.

Various oxidizing agents were reported to oxidize the internal alkyne to 1,2-diketone. Among them, potassium permanganate at neutral pH conditions was used under different conditions[25–30] as an efficient oxidizing agent due to its low cost, easy availability and simple work-up procedure. Initially, $KMnO_4$ oxidation in the presence of $NaHCO_3$/$MgSO_4$ in acetone–water solvent mixture was chosen to realize the transformation of the internal alkyne **2** to 1,2-diketone **3** at room temperature for 4 h. The desired product **3** was obtained in 60 % yield. In order to increase the yield of **3**, it was intended to use $KMnO_4$ oxidation in PEG-400 owing to its crown ether and PTC properties.

Although, potassium permanganate oxidation of alkyne to α-diketone under various reaction conditions including PTC condition have been reported, its use in combination with PEG-400 has not hitherto been explored. These observations prompted us to use PEG-400 as a phase transfer catalyst in the potassium permanganate oxidation of quinazolin-4(3*H*)-one based alkyne **2** to the 1,2-diketone **3**. The reaction of alkyne **2** with $KMnO_4$ in aqueous dichloromethane (DCM:H_2O, 1:1) using PEG-400 as phase transfer catalyst at pH 7–7.5 for 2.5 h at 40 °C led to the desired compound 1,2-diketone **3** in 80 % isolated yield (Scheme 2). To the best of our knowledge, compound **3** is a new compound and hence, its structure was established from its IR, HRMS, ^{1}H- and ^{13}C-NMR spectral data. The appearance of three peaks (1677, 1709 and 1774 cm^{-1}): in its IR and three in the ^{13}C-NMR spectra (δ 193.6, 192.7 and 161.8 ppm) designate the presence of three carbonyl groups in **3**. In the ^{1}H-NMR spectrum, the downfield shift of C-5H and C-7H in quinazolinone moiety (δ 8.84 ppm, 1H, *d*, J = 1.18 Hz; δ 8.40 ppm, 1H, *dd*, J = 1.98 Hz & 8.50 Hz,) and *ortho* protons of the phenyl ring (δ 8.01 ppm,

2H, *dd*, *J* = 1.06 and 8.24 Hz) is due to paramagnetic anisotropic effect of the C=O groups. The *N*-methyl protons resonate at δ 3.51 ppm as a singlet and the remaining nine aromatic protons are accounted for in the range δ 7.85–7.52 ppm. The high-resolution mass spectrum showed the $[M+H]^+$ ion peak at *m*/*z* 369.1235 indicates the molecular weight of **3** as 368.1163. This oxidation method provides a very simple procedure that results the 1,2-diketone in good yield and high purity. The advantage of this method over the other $KMnO_4$ oxidation reactions are: *i*) PEG-400 acts as PTC as well as reaction medium, *ii*) shorter duration of time and *iii*) avoiding the use of salts such as $MgSO_4$ and $NaHCO_3$. With 1,2--diketone **3** in hand, the preparation of 6-pyrazinyl/fused pyrazinylquinazolin--4(3*H*)-ones was undertaken.

Previously, the synthesis of pyrazines and fused pyrazines was reported from the condensation of 1,2-dicarbonyl compound and 1,2-diamine in acetic acid or aqueous acetic acid. The condensation of diketone **3** independently with symmetrical 1,2-diamines **4** and **6** in aqueous acetic acid for 12 h at 80 °C. The expected cyclo-condensed and oxidative cyclo-condensed products, 6-pyrazinyl/ /fused pyrazinylqunazol-4(3*H*)-ones **5** (68 % yield) and **7** (70 % yield) were obtained respectively. It was intended to use a neutral solvent that could be expect to perform the function of aqueous acetic acid in the condensation of a 1,2--dicarbonyl compound and symmetrical 1,2-diamines **4** and **6**. Alternatively, the same reaction was carried out in PEG-400/water (3:1) at 80 °C for 12 h. After work-up, the desired cyclo-condensed and oxidative cyclo-condensed[31] products **5** (75 % yield) and **7** (78 % yield) were isolated, respectively. These two compounds were characterized by IR, HRMS, ^{1}H-NMR and ^{13}C-NMR spectral data.

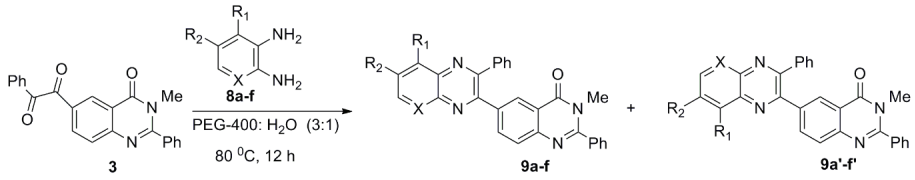


Scheme 2. Synthesis of 3-methyl-2-phenyl-6-pyrazinyl / fused pyrazinylquinazolin-4(3*H*)--ones (**5** and **7**).

To generalize the scope of the reaction, experiments were performed with various unsymmetrical 1,2-diamines **8a**–**f**, which would afford inseparable mixtures of regioisomeric products **9a**–**f** and **9a′**–**f′**. The isomers **9** were not separable by column chromatography using different solvent mixtures (Scheme 3).

The hydroxyl groups of PEG-400 *via* hydrogen bonding with oxygen atoms of carbonyl groups might increase the electrophilic character of the dicarbonyl carbons, thereby accelerating the rate of addition of 1,2-diamines on carbonyl groups. These factors might be responsible for the acceleration of the cyclocondensation reaction for quinoxaline and pyrazine ring formation. The solvent PEG

was recycled and reused in cyclocondensation of **3** with compound **6** to obtain **7** and the corresponding data are presented in Table I. The solvent was reused for 4 cycles without loss of activity and the observed yields of the product were 78––61 % from 1st to 4th use of PEG-400.



Compound	X	R_1	R_2
9a/9a'	CH	H	Br
9b/9b'	CH	H	Cl
9c/9c'	CH	NO_2	H
9d/9d'	CH	H	NO_2
9e/9e'	N	H	H
9f/9f'	N	H	Br

Scheme 4. Synthesis of 3-methyl-2-phenyl-6-fused pyrazinylquinazolin-4(3*H*)-one regioisomers (**9a**–**f** and **9a′**–**f′**).

TABLE I. Recycling of PEG-400:H_2O solvent in the cyclocondensation of **3** with **7**

Product 10	Fresh	2nd run	3rd run	4th run
Yield, %	78	75	70	61

An array of 6-quinaoxlinyl or 6-pyridiopyrazinylquinazolinones **5**, **7** and **9** were obtained in high yields (70–78 %) from PEG-mediated cyclo-condensation of **3** with various 1,2-diamines **4**, **6** and **8a**–**g**.

CONCLUSIONS

In conclusion, a novel PEG-mediated protocol for the synthesis of 6-heteryl-quinazolin-4(3*H*)-ones was achieved. The Castro–Stephen coupling reaction and PEG-mediated $KMnO_4$ oxidation provided the key intermediates internal alkyne **2** and 1,2-diketone **3** in high yields. The PEG-mediated $KMnO_4$ oxidation of **3** has been reported for the first time to obtain 1,2-diketone **3**. Interestingly, the $KMnO_4$ oxidation in PEG-400 medium is an efficient, simple, and more economical with amplified reaction rate, when compared to those found using previous protocols. Furthermore, the novel 6-pyzinyl/fused pyzinylquinazolin-4(3*H*)-ones were synthesized in high yields in PEG-mediated cyclocondensation reaction of **3** with 1,2-diamines **4**, **6** and **8a**–**g** that appears to be a good substitute to aqueous acetic acid medium. Therefore, the overall procedure is an efficient and high yielding synthesis to obtain 6-heterylquinazolin-4(3*H*)-ones. Employing the same strategy, work is under progress for the synthesis of new heterocyclic compounds.

SUPPLEMENTARY METERIAL

Analytical and spectral data of the compounds are available at http://www.shd.org.rs/ /JSCS/, or from corresponding author on request.

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ИЗВОД

СИНТЕЗА 6-ПИРАЗИНИЛ-/КОНДЕНЗОВАНИХ ПИРАЗИНИЛКИНАЗОЛИН-4(3*H*)-ОНА ПРИМЕНОМ CASTRO–STEPHEN КУПЛОВАЊА, РЕАКЦИЈАМА ОКСИДАЦИЈЕ И ЦИКЛОКОНДЕНЗАЦИЈЕ УЗ PEG КАО РЕАКЦИОНИ МЕДИЈУМ

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Развијен је еколошки поступак за синтезу 6-пиразинил-/кондензованих пиразинил-киназолин-4(3*H*)-она полазећи од 6-јод-3-метил-2-фенилкиназолин-4(3*H*)-она у присуству киназолин алкин/1,2-дикетона као интермедијера, применом Castro–Stephen куповања и оксидацијом помоћу калијум-перманганата и реакције циклокондензације, уз PEG као реакциони медијум.

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