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Microwave assisted synthesis of 3-substituted-4(3*H*)-quinazolinone using silica supported potassium carbonate

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ABSTRACT

A new synthetic route to 3-substituted-4(3*H*)-quinazolinone has been developed using Potassiumcarbonate-silica as a solid support under microwave irradiation which is an environmental friendly procedure. The use of basic catalyst under solvent free condition for the ring closure of this heterocyclic system is notable.

KEYWORDS

Quinazolinone;
Solid support;
Anthranilate;
Solvent free;
Potassium carbonate.

INTRODUCTION

Recent attention has been focused much on the derivatives of quinazolinones, especially in view of their potential pharmalogical and biological activities, such as anti-parasitic, antitumor[1a], antibacterial[1b], antifungal[1c,d], antiinflammatory[1e], antiviral[1f], potential anticonvu-lsants[1g], anticoccidial activity[1h] and as Tyrosine Kinase Inhibitors[1i,j]. Quinazolinone moieties are also found in several naturally occurring bioactive alkaloids such as rutaecarpine[2], anacine[3], fiscalin[4], sclerotigenin[5], circumdatin[6], benzomalvin[7], etc.

The first synthesis of 4-quinazolinones was reported by Niementowski in 1895 which involved a cyclocondensation of anthranilic acids and amides at temperatures exceeding 150°C[8]. Grimmel modified the former synthesis by heating *N*-acetylanthranilic acids with anilines in toluene or xylene in the presence of condensing agents such as phosphorus trichloride, phosphorus oxychloride or thionyl chloride[9]. However, a search for a more reliable and suitable drug is always fascinating and challenging. Therefore, a number of synthetic methods for the preparation of substituted 4-quinazolinones have been described *via* alternate pathways such as reaction of thioureas with isatoic anhydride[10], cyclodehydration of 2-benzamidobenzoic acid[11], treating 2-aminobenzonitriles with urea, hydrogen peroxide and pyrolyzing *O*-acetylaminobenzamides[12]. Numerous catalysts also have been employed for the preparation of these compounds such as using alumina supported-$CeCl_3/7H_2O$–NaI[13], silica sulfuric acid under solvent-free conditions[14], ionic liquid[15], $AlCl_3/ZnCl_2$-SiO_2[16], lanthanum(III) nitrate hexahydrate or p-toluenesulfonic acid[17], $Bi(TFA)_3$-[nbp]$FeCl_4$[18], $ZnCl_2$[19], sodium perborate[20], heteropolyacids[21], $ZrCl_4$[22] and $InCl_3$[23].

The replacement of current chemical processing techniques with more environmentally benign alternatives is an increasingly attractive subject. The increasing environmental consciousness throughout the world has put a pressing need to develop an alternate synthetic approach for biologically and synthetically important compounds. This requires a new approach which will reduce the material and energy intensity of chemical processes and products which minimize or eliminate the dis-

Full Paper

persion of harmful chemicals in the environment in a way that enhances the industrially benign approach and meets the challenges of green chemistry. The development of microwave assisted reactions have profound impact on organic synthesis which require the use of high dielectric solvents such as dimethyl sulfoxide and dimethylformamide on solid support where the organic compound is adsorbed on the surface of the inorganic oxides such as alumina, silica and clay, however, microwave assisted solution phase reaction is confined only to low pressure and the use of special vessel and sealed container. Therefore, the use of solid support solvent-free microwave assisted reaction has a better scope which provides an opportunity to work with open vessel thus avoiding the risk of high pressure formation.

EXPERIMENTAL

Microwave reactions were carried out in a CEM Discover Benchmate microwave digester. Melting points of new compounds were taken in open capillary tubes and are uncorrected. Infrared spectra were recorded on a BOMEM DA-8 FTIR instrument and the frequencies are expressed in cm^{-1}. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance II-400 spectrometer using $CDCl_3$ as the solvent. Chemical shifts are reported in ppm downfield from internal tetramethylsilane and are given on the δ scale. Mass spectral data were obtained with a JEOL D-300 (EI) mass spectrometer. Elemental analyses were carried out on a Heraeus CHN-O-Rapid analyzer. All compounds give satisfactory elemental analyses within ± 0.4% of the theoretical values. All reactions were monitored by TLC using precoated aluminum sheets (silica gel 60 F_{254} 0.2 mm thickness) and developed in an iodine chamber or under UVGL-15 mineral light 254 lamp. Column chromatographic separations were carried out using ACME silica gel (60–120 mesh).

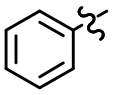
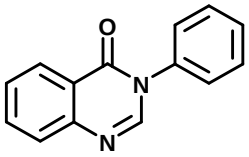
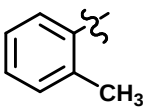
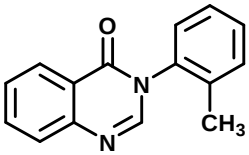
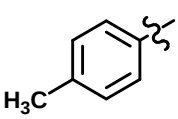
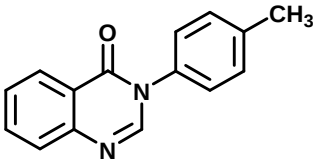
Preparation of SiO_2 supported K_2CO_3

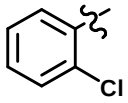
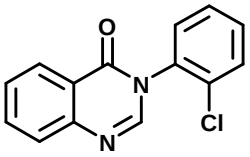
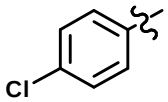
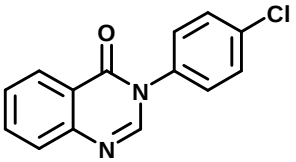
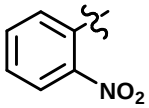
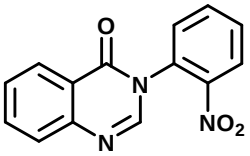
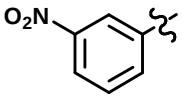
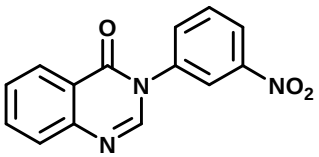
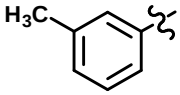
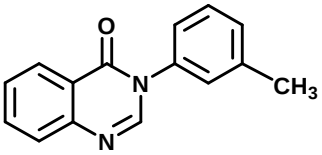
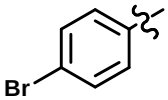
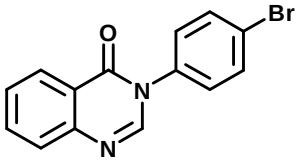
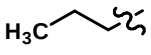
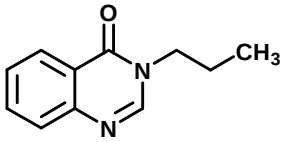
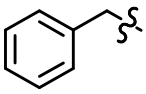
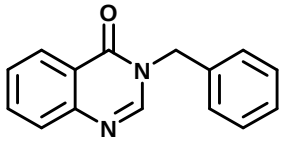
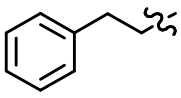
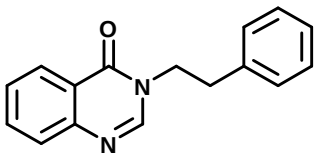
To a stirring solution of 4.14 g (0.03 mol) of K_2CO_3 in 20 ml of water, 10g of SiO_2 (column chromatographic grade, 60 Å, 200-400 mesh) was added. The mixture was stirred for 20 min and then gently heated on a hot plate, with intermittent swirling, until a free-flowing white solid was obtained. The catalyst was further dried by placing the beaker in an oven maintained at 120°C for at least 24 hours prior to use where the catalyst was also activated.

General procedure for microwave assisted synthesis of 4(3H)-quinazolinone

A mixture of the anthranilate ester (1 mmol), triethyl orthoformate (1 mmol) and amine (1 mmol) was mixed with 100 mg of $K_2CO_3.SiO_2$ and was irradiated in a microwave digester at 5–10 bar, 80–120 W, for specified time as given in the TABLE 1 without the use of solvent. After the reaction was completed (monitored by TLC) the resultant mixture was filtered, and the recovered catalyst was washed with CH_2Cl_2 (50 ml). The combined organic extracts were concentrated

TABLE 1 : Silica supported potassium carbonate synthesis of 3-substituted-4(3H)-quinazolinone under microwave irradiation.

Entry	R	Product	Yield (%)	Reaction time (sec)	Mp(°C)
4a			85	300	137-139[16]
4b			83	330	139-140[22]
4c			85	330	144-145[16]

Entry	R	Product	Yield (%)	Reaction time (sec)	Mp(°C)
4d			78	420	136-138[22]
4e			75	390	180-181[16]
4f			70	390	154-155[22]
4g			73	290	154-155[22]
4h			90	330	137-138[22]
4i			88	330	186
4j			86	300	82-83
4k			90	360	118–119
4l			85	360	123-124

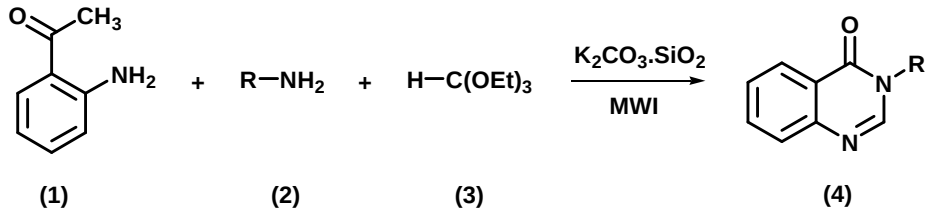
on a rotary evaporator, and the resulting residue was column-chromatographed with hexane/ethyl acetate as eluent to afford the pure compounds.

RESULTS AND DISCUSSION

According to our literature survey, the environmental friendly syntheses of quinazolinones using solid supports are not well documented. Most of the procedure for the synthesis of quinazolinone employs the derivatives of anthranilic acid and alkoyl/acyl halide where the second condensed ring is closed through the synthesis of benzoxazinone which on further reaction with amine gives quinazolinone. In order to avoid the use of toxic

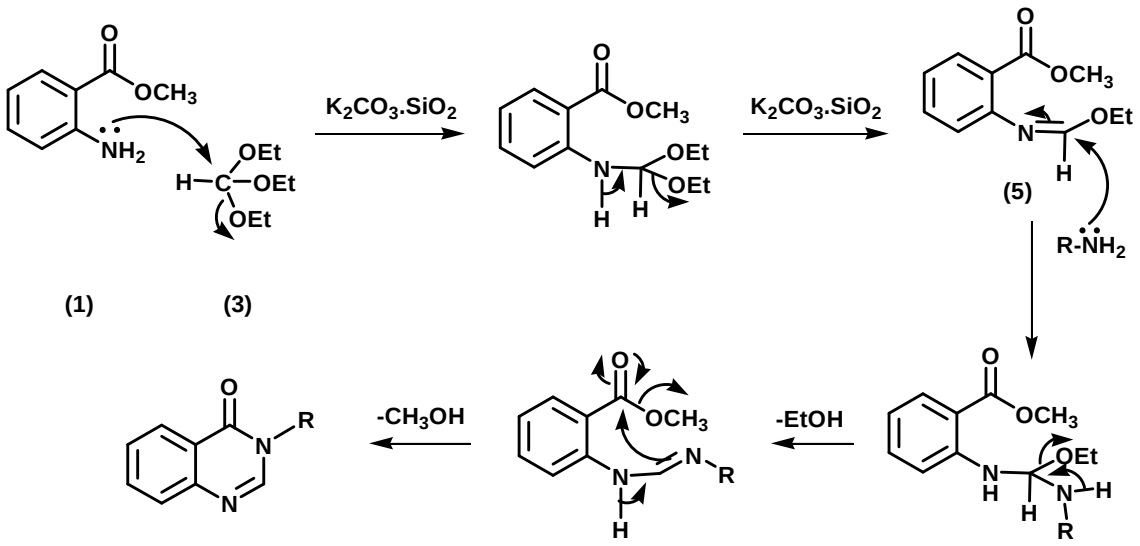
Full Paper

reagent we have replaced the anthranilic acid with alkyl anthranilate and acyl chloride with orthoester. We have developed a convenient route for the synthesis of 3-substituted-quinazolin-4(3*H*)-one (Scheme 1) starting from methyl anthranilate **(1)**, orthoester **(3)** and substituted amines **(2)** (both aromatic and aliphatic amine). The one pot synthesis of the titled compound is achieved by cyclocondensation, which may involve the intermediate imidic ester **(5)** (Scheme 2). The imidic ester is very prone to react with an amine, thus leading to the amidine intermediate 7 which is generated in *situ* by the condensation of amino group from methyl anthranilate with ortho ester in presence of silica supported potassium carbonate, followed by the cyclisation with aliphatic or aromatic amine to give the products in good to excellent yields (TABLE 1). In some cases the crude product was contaminated with some starting materials and some side reaction, which could easily be removed by recrystallisation or by column chromatography. In conjunction with our ongoing synthesis of heterocyclic systems[24] from readily available non-toxic starting materials, we herein report the synthesis of the derivatives of quinazolinones using potassium carbonate-silica as solid support. Potassium carbonate-solid supported reactions have been reported in many organic transformations and heterocyclic syntheses[25]. Therefore, in order to broaden the scope of quinazolinone chemistry we have applied the same principle for the synthesis of 3-substituted-quinazolin-4(3*H*)-one which gives satisfactory results.



(1) (2) (3) (4)

Scheme 1



(1) (3) (5) (6) (7) (4)

Scheme 2 : Proposed mechanism for the formation of the title compound.

SPECTROSCOPIC AND ANALYTICAL DATA

3-phenyl-4(3*H*)-quinazolinone (4a)

IR(KBr) ν_{max}/cm^{-1}: 1689(C=O), 1633, 1454; ^{1}H-NMR (TMS) δ/ppm: 8.35 (s, 1H), 8.07 (s, 1H), 7.73-7.61 (m, 3H), 7.49-7.33 (m, 4H) and 7.21 (t, 1H); ^{13}C-NMR (TMS) δ/ppm: 161.0, 148.3, 147.1, 137.2, 133.7, 131.3, 129.4, 128.7, 127.3, 126.8, 126.5 and 122.1; MS (ESI): m/z: 222 (M^+, 100%), 223.2 (M^++1, 15%). Anal. Calcd. for $C_{14}H_{12}N_2O$ are: 75.66; H, 4.54; N, 12.60; Found: 75.47; H, 4.63; N, 12.47.

3-(2-methylphenyl)-4(3*H*)-quinazolinone (4b)

IR(KBr) ν_{max}/cm^{-1}: 1690(C=O), 1597, 1462; ^{1}H-NMR (TMS) δ /ppm : 8.37 (s, 1H), 8.12 (d, 1H), 7.71-7.63 (m, 3H), 7.11-7.37 (m, 4H) and 2.45 (s, 3H); ^{13}C-NMR (TMS) δ/ppm: 160.3, 147.7, 146.6, 135.2, 134.4, 133.6, 130.7, 129.4, 128.2, 127.1, 126.2, 126.7, 125.2, 122.7 and 18.7; MS (ESI): m/z: 236.2 (M^+, 100%), 237.2 (M^++1,17%). Anal. Calcd. for $C_{15}H_{12}N_2O$ are: C, 76.25; H, 5.12; N, 11.86; Found: C, 76.35; H, 5.32; N, 11.75.

3-(4-methylphenyl)-4(3*H*)-quinazolinone (4c)

IR(KBr) ν_{max}/cm^{-1}: 2923, 1690(C=O), 1600, 1471; ^{1}H-NMR (TMS) δ/ppm: 8.29 (s, 1H), 8.17 (d, 1H), 7.69-7.76 (m, 3H), 7.19-7.50 (m,4H) and 2.34 (s, 3H); ^{13}C-NMR (TMS) δ/ppm: 159.6, 147.1, 146.2, 136.3, 133.4, 134.7, 133.0, 129.5, 128.1, 126.1, 125.7, 121.3 and 20.1; MS (ESI): m/z: 236.2 (M^+, 100%), 237.2 (M^++1,15%). Anal. Calcd. for $C_{15}H_{12}N_2O$ (*M*r = 236.2) are: C, 76.25; H, 5.12; N, 11.86; Found: C, 76.32; H, 5.18; N, 11.70.

3-(2-chlorophenyl)-4(3*H*)-quinazolinone (4d)

IR(KBr) ν_{max}/cm^{-1}: 1693(C=O), 1607, 1467; ^{1}H-NMR (TMS) δ/ppm: 8.33 (s, 1H), 8.02 (d, 1H), 7.59-7.75 (m, 5H), 7.33(t, 1H) and 7.15 (t, 1H); ^{13}C-NMR (TMS) δ/ppm: 160.7, 148.5, 147.3, 135.8, 134.3, 132.4, 131.8, 131.0, 130.6, 129.5, 127.1, 126.3, 125.1 and 121.9; MS (ESI): m/z: 256 (M^+, 100%), 257 (M^++1, 15%). Anal. Calcd. for $C_{14}H_9ClN_2O$ are: C, 65.51; H, 3.53; N, 10.91; Found: C, 65.32; H, 3.43; N, 10.80.

3-(4-chlorophenyl)-4(3*H*)-quinazolinone (4e)

IR(KBr) ν_{max}/cm^{-1}: 1693(C=O), 1607, 1447; ^{1}H-NMR (TMS) δ /ppm: 8.39 (s, 1H), 8.08 (d, 1H), 7.64-7.72 (m, 3H), 7.42(d, 2H) and 7.38 (d, 2H); ^{13}C-NMR (TMS) δ/ppm: 160.9, 147.8, 147.1, 135.7, 133.8, 133.0, 130.8, 129.2, 127.5, 126.9, 126.2 and 120.7; MS (ESI): m/z: 256 (M^+, 100%), 257 (M^++1, 25%). Anal. Calcd for $C_{14}H_9ClN_2O$ are: C, 65.51; H, 3.53; N, 10.91; Found: C, 65.42; H, 3.62; N, 10.84.

3-(2-nitrophenyl)-4(3*H*)-quinazolinone (4f)

IR(KBr) ν_{max}/cm^{-1}: 1676(C=O), 1601, 1487; ^{1}H-NMR (TMS) δ/ppm: 8.45 (s, 1H), 8.20 (d, 1H), 8.09 (d, 1H), 8.01 (d, 1H) and 7.63-7.79 (m, 5H); ^{13}C-NMR (TMS) δ/ppm : 160.7, 148.2, 147.3, 142.6, 136.6, 134.3, 133.3, 127.8, 127.1, 126.6, 125.9, 125.3, 122.4 and 120.7; MS (ESI): m/z: 267 (M^+, 100%), 268.01 (M^++1, 18%). Anal. Calcd. for $C_{14}H_9N_3O_3$ are: C, 62.92; H, 3.39; N, 15.72; Found: C, 62.83; H, 3.42; N, 15.81.

3-(3-nitrophenyl)-4(3*H*)-quinazolinone (4g)

IR(KBr) ν_{max}/cm^{-1}: 1688(C=O), 1605, 1493; ^{1}H-NMR (TMS) δ/ppm: 8.77 (s, 1H), 8.12 (d, 1H), 8.00 (d, 1H), 7.65-7.82 (m, 5H) and 7.43 (s, 1H); ^{13}C-NMR (TMS) δ/ppm: 160.4, 148.7, 147.8, 147.1, 134.6, 133.5, 132.9, 129.6, 127.2, 126.8, 126.3, 121.4, 120.8 and 120.0; MS (ESI): m/z : 267 (M^+, 100%), 268 (M^++1, 23%). Anal. Calcd. for $C_{14}H_9N_3O_3$ are: C, 62.92; H, 3.39; N, 15.72; Found: C, 62.87; H, 3.28; N, 15.79.

3-(4-methylphenyl)-4(3*H*)-quinazolinone (4h)

IR(KBr) ν_{max}/cm^{-1}: 1698(C=O), 1584, 1465; ^{1}H-NMR (TMS) δ/ppm: 8.10 (d, 1H), 7.64-7.72 (m, 3H), 7.50.(s, 1H), 7.02-7.21 (m, 3H), 6.78 (s, 1H) and 2.41 (s, 3H); ^{13}C-NMR (TMS) δ/ppm: 159.9, 148.5, 147.2, 138.6, 134.3, 133.1, 129.5, 128.6, 127.3, 126.8, 126.2, 125.1, 124.7, 120.3 and 22.7; MS (ESI): m/z: 236 (M^+, 100%), 237 (M^++1, 19%). Anal for $C_{15}H_{12}N_2O$ (*M*r = 236) are: C, 76.25; H, 5.12; N, 11.86; Found: C, 76.32; H, 5.27; N, 11.96.

3-(4-bromophenyl)-4(3*H*)-quinazolinone (4i)

IR(KBr) ν_{max}/cm^{-1}: 1698(C=O), 1603, 1444; ^{1}H-NMR (TMS) δ /ppm: 8.73 (d, 2H), 8.05 (d, 1H), 7.58-7.70 (m, 5H) and 7.45(s, 1H); ^{13}C-NMR (TMS) δ/ppm: 160.3, 148.3, 147.5, 136.8, 133.6, 130.7, 128.7, 127.3, 126.7, 125.9, 122.6 and 120.3; MS (ESI): m/z: 300 (M^+, 100%), 301 (M^++1, 32%). Anal. Calcd. for $C_{14}H_9BrN_2O$ are: C, 55.84; H, 3.01; N, 9.30; Found: C, 55.92; H, 3.20; N, 9.17.

3-propyl-4(3*H*)-quinazolinone (4j)

IR(KBr) ν_{max}/cm^{-1}: 1674(C=O), 1612, 1453; ^{1}H-NMR (TMS) δ/ppm: 8.34 (s, 1H), 7.98 (d, 1H), 7.64-7.73 (m, 3H), 4.23 (t, 2H), 1.63-1.69 (m, 2H) and 1.03(t, 3H); ^{13}C-NMR (TMS) δ/ppm: 161.3, 148.5, 147.7, 133.6, 128.7, 127.1, 126.7, 121.1, 50.3, 21.6 and 15.3; MS (ESI): m/z: 188 (M^+, 100%), 189 (M^++1, 12%). Anal. Calcd. for $C_{14}H_9BrN_2O$ are: C, 70.19; H, 6.43; N, 14.88; Found: C, 70.32; H, 6.32; N, 14.75.

Full Paper

3-benzyl-4(3*H*)-quinazolinone (4k)

IR(KBr) ν_{max}/cm^{-1}: 1693(C=O), 1608, 1462; ^{1}H-NMR (TMS) δ/ppm : 8.26 (s, 1H), 8.08 (d, 1H), 7.69-7.77 (m, 3H), 7.24-7.38 (m, 5H), and 5.47(s, 2H); ^{13}C-NMR (TMS) δ/ppm: 161.7, 148.7, 147.9, 136.6, 133.5, 128.9, 127.7, 127.1, 126.7, 126.0, 125.6, 120.3 and 52.5; MS (ESI): m/z: 236 (M^+, 100%), 237 (M^++1, 19%). Anal. Calcd. for $C_{14}H_9BrN_2O$ are: C, 76.25; H, 5.12; N, 11.86; Found: C, 76.35; H, 5.22; N, 11.75.

3-phenethyl-4(3*H*)-quinazolinone (4l)

IR(KBr) ν_{max}/cm^{-1}: 1686(C=O), 1611, 1455; ^{1}H-NMR (TMS) δ /ppm: 8.30 (s, 1H), 8.12 (d, 1H), 7.61-7.74 (m, 3H), 7.30-7.43 (m, 5H), 3.41 (t, 2H) and 2.49 (t, 2H); ^{13}C-NMR (TMS) δ/ppm: 161.5, 148.3,147.6, 139.4, 133.7, 128.6, 127.8, 127.3, 126.9, 126.3, 125.8, 121.1, 51.6 and 41.3; MS (ESI): m/z: 250 (M^+, 100%), 251 (M^++1, 17%). Anal. Calcd. for $C_{14}H_9BrN_2O$ are: C, 76.78; H, 5.64; N, 11.19; Found: C, 76.69; H, 5.56; N, 11.27.

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