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A novel approach toward the synthesis of azetidinones derivatives

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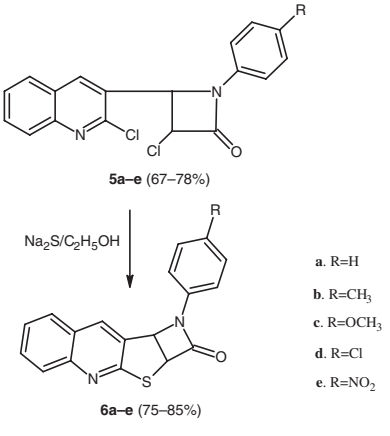
A convenient, efficient and inexpensive procedure for the synthesis of thieno[2,3-*b*]quinolines was devised. 3-Formyl-2-chloroquinoline **2a** on treating with various substituted anilines **3a–e** produced *N*-[(1*E*)-(2-chloroquinolin-3-yl)methylidene]anilines **4a–e**. The resulted compounds on further treatment with chloroacetyl chloride gave substituted azetidinones **5a–e**. These compounds are easily converted to thieno[2,3-*b*]quinolines **6a–e** by using sodium sulfide in ethanol. The structures of all the newly synthesized compounds were elucidated on the basis of elemental analysis, IR, ^{1}H NMR and mass spectral data.



Keywords: azetidinones; 3-formyl-2-chloroquinoline; Schiff base; thieno[2,3-*b*]quinolines; sulfur chemistry

1. Introduction

Quinolines and hetero-fused analogs have attracted great attention of medicinal and synthetic chemists because of their presence in natural products and physiological activities (*1*). Thieno

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derivatives are known to exhibit an array of biological activities including analgesic and anti-inflammatory activities (*2–6*). It is evident from the literature that thieno derivatives are well known for their varied biological activities such as high affinity selective 5-HT_{1A} receptor ligands (*7*), potential antihypertensive agents (*8*), molluscicidal and larvicidal activities (*9, 10*), anticonvulsant activity (*11*), antibacterial activity (*12*), and cholesterol inhibition activity (*13, 14*).

Substituted 2-azetidinones are an important class of compound for their importance in β-lactam antibiotic synthesis (*15–17*). β-Lactam drugs in heterocycles are still the most widely prescribed antibiotics used in medicine (*18*). The discovery of penicillin 2-azetidinone-based heterocycles has been one of the main classes of drugs with wide therapeutic activities viz. anticonvulsant (*19*), anti-inflammatory (*20*), antibacterial (*21*), herbicidal (*22*) and also functioning as an enzyme inhibitor (*23*), and they are effective on central nervous system disorders (*24*).

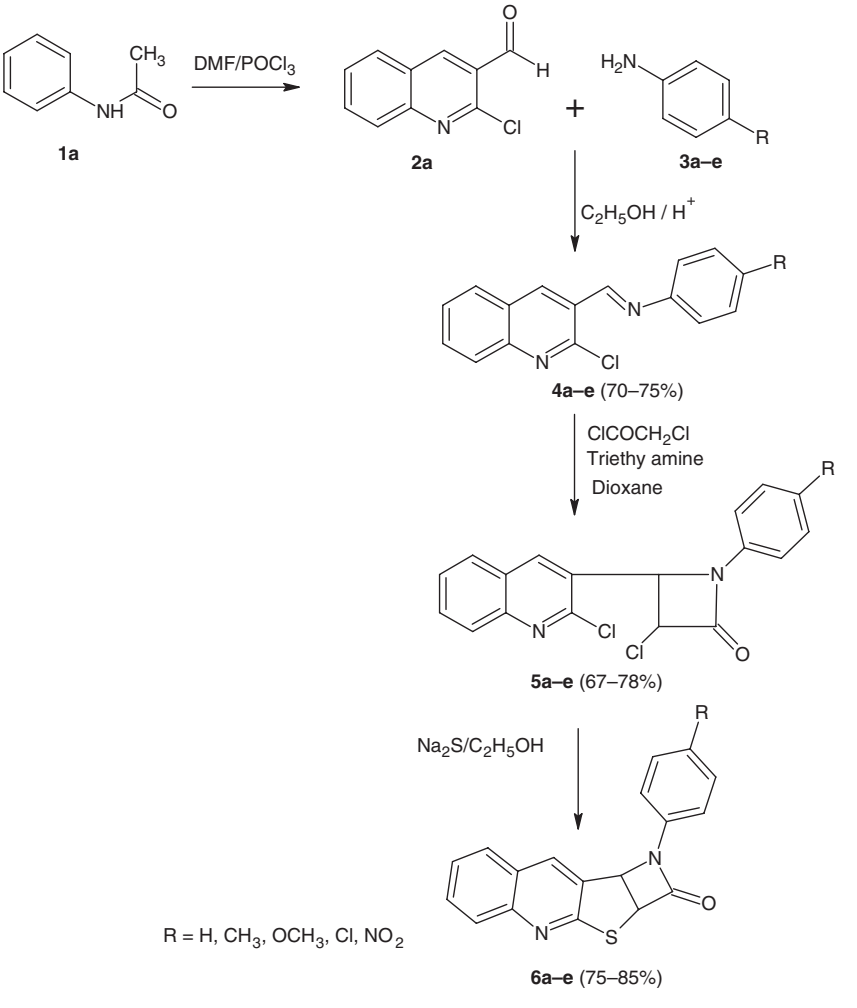
The conversion of aryl quinolines into thieno[2,3-*b*]quinolines is of vital importance in synthetic organic chemistry. Thieno[2,3-*b*]quinolines are prepared via their corresponding halogenated derivatives. A survey of the literature reveals that there are numerous methods (*25–28*) for the construction of thieno quinolines. Such procedures are obviously accompanied by poor yields, undesired side products, vigorous reaction conditions, long reaction times, expensive or toxic reagents and tedious work-up. Therefore, there is still a need to introduce new methods and inexpensive reagents for such functional group transformations. In this communication, we wish to report an efficient and inexpensive method for the synthesis of thieno[2,3-*b*]quinolines starting from an easily accessible dichloro substrate by a reaction with sodium sulfide in ethanol media with a quantitative yield under remarkably soft conditions. We also determined that the best results were achieved using sodium sulfide. The constructions of the thieno ring by using Na_2S in ethanol media were proven to be a useful procedure. In continuation of our program directed toward studies on sulfur chemistry (*29–33*) and synthesis of new potentially bioactive molecules, we wish to examine the feasibility and efficiency of this approach to synthesize some new thieno[2,3-*b*]quinolines. This new method fulfills the need for a medicinal, bioorganic, industrial cost-effective and commercial method for the synthesis of quinoline-based sulfur compounds.

2. Results and discussions

In this contribution, we focus our attention on the fast and efficient synthesis of thieno quinoline derivatives. Initially, the key intermediate 3-formyl-2-chloroquinoline **2a** was prepared from easily available acetanilide by the reported method (Scheme 1) (*34*).

The Schiff bases **4a–e** were prepared from compound **2a** by reaction with various aromatic aldehydes **3a–e** in the presence of a catalytic amount of acetic acid in absolute ethanol. The IR spectrum of **4a** exhibits an absorption band at 1623.50 cm^{-1} and the ^1H NMR exhibits multiplets at δ 7.10–8.50 ppm for 10 protons and as imine protons at δ 9.01 ppm confirming its structure.

At the same time, we focussed our attention on the synthesis of azetidinones **5a–e**. The compounds **4a–e** were made to react with chloro acetyl chloride and triethylamine in dioxane. The structural analysis of the newly synthesized molecule **5a** includes IR, ^1H NMR and mass spectral investigations. The IR spectra of the compound revealed sharp strong absorption bands at 1735.81 cm^{-1} for the azetidinone ring. The ^1H NMR spectrum exhibited multiplets in the region between 7.26 and 8.50 ppm for 10 aromatic protons. The two protons in the azetidinone ring, *i.e.* −N−CH−C− and −C−CH−Cl are found to resonate as doublets at δ 4.91 and δ 5.71 ppm, respectively. The structure was further confirmed by mass spectral studies. It gave a molecular ion peak at m/z 343 (M^+) which corresponds to the molecular formula $C_{18}H_{12}Cl_2N_2O$ (Scheme 1). The spectral details for all the synthesized compounds are given in Section 3 and are in agreement with the assigned structures.



Scheme 1. General synthetic procedure for the synthesis of azetidinones derivatives.

Very interestingly, the reactions of **5a**–**e** with sodium sulfide in ethanol underwent ring cyclization to give thieno quinolines **6a**–**e**. As we expected, the ^{1}H NMR spectrum of **5a** exhibited two peaks at δ 4.96 ppm and δ 5.89 ppm for the two protons present in the azetidinone ring (−N−CH−C− and −S−CH−). The aromatic protons resonate as multiplets at δ 7.35–8.60 ppm. The structure was further confirmed by recording its mass spectra. Also, Beilstein's test confirms the absence of chlorine.

3. Experimental

IR spectra were taken on a Perkin Elmer 157 Infrared spectrophotometer. The ^{1}H NMR spectra (300 MHz) were recorded on a Bruker supercon FT NMR instrument using TMS as the internal standard and mass spectra on a Jeol JMS-D 300 mass spectrometer operating at 70 eV. Melting points were determined in open capillary tubes and are uncorrected. Purity of the compounds was checked by TLC on silica gel and they were purified by column chromatography.

3.1. *Preparation of 3-formyl-2-chloroquinoline (4a)*

The Vilsmeier reagent was prepared by adding 24 ml (0.031 mol) of *N*,*N*-dimethylformamide to a round-bottom flask in an ice-cold condition (0–5°C) under constant stirring. To this flask, 36 ml (0.38 mol) of phosphorus oxychloride was added dropwise over a period of half-an-hour and the resultant mixture was stirred for a further hour. The appropriate acetanilide **1a** (1 g) was then added to the Vilsmeier reagent and stirred for another half-an-hour and the reaction mixture was refluxed on a water bath for 4–6 h. After the reaction was completed (TLC monitoring), the reaction mixture was poured onto 500 g of crushed ice under constant manual stirring. After neutralization with sodium hydroxide solution, the precipitate (80%, 1.52 g) obtained was filtered, washed well with water, dried and recrystallized using ethyl acetate.

3.2. *Preparation of Schiff base (4a)*

3-Formyl-2-chloroquinoline **2a** (0.01 mol), aniline (0.01 mol) **3a** and a catalytic amount of acetic acid were taken in ethanol (20 ml) and heated to reflux for 3–4 h. After the completion of the reaction (TLC), the reaction mixture was poured onto crushed ice; the solid mass thus separated out was filtered, washed with water and dried to give the desired compound **4a**. Similarly, the compounds **4b–e** were prepared in 70–75% yields.

3.2.1. *N-[(1E)-(2-Chloroquinolin-3-yl)methylidene]aniline (**4a**)*

Solid, 73%; mp 186°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.10–8.50 (10H, m, Ar-H), 9.01 (1H, s, $-$N = CH); IR (KBr) ν (cm^{-1}): 1623.50. [M$^+$], 266. Calcd. (%) for $C_{16}H_{11}ClN_2$: C, 72.05; H, 4.16; N, 10.50. Found: C, 72.35; H, 4.27; N, 10.37.

3.2.2. *N-[(1E)-(2-Chloroquinolin-3-yl)methylidene]-4-methylaniline (**4b**)*

Solid, 73%; mp 194°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.60 (3H, s, CH_3), 7.12–8.60 (9H, m, Ar-H); 9.03 (1H, s, $-$N = CH), IR (KBr) ν (cm^{-1}): 1622.60. [M$^+$], 280. Calcd. (%) for $C_{17}H_{13}ClN_2$: C, 72.73; H, 4.67; N, 9.98. Found: C, 72.54; H, 4.43; N, 9.77.

3.2.3. *N-[(1E)-(2-Chloroquinolin-3-yl)methylidene]-4-methoxyaniline (**4c**)*

Solid, 75%; mp 175°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 3.94 (3H, s, $-OCH_3$), 7.13–8.45 (9H, m, Ar-H); 9.02 (1H, s, $-$N = CH), IR (KBr) ν (cm^{-1}): 1623.50. [M$^+$], 296. Calcd. (%) for $C_{17}H_{13}ClN_2O$: C, 68.81; H, 4.42; N, 9.44. Found: C, 68.58; H, 4.37; N, 9.18.

3.2.4. *4-Chloro-N-[(1E)-(2-chloroquinolin-3-yl)methylidene]aniline (**4d**)*

Solid, 71%; mp 204°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.25–8.65 (9H, m, Ar-H); 9.03 (1H, s, $-$N = CH), IR (KBr) ν (cm^{-1}): 1622.70. [M$^+$], 301. Calcd. (%) for $C_{16}H_{10}Cl_2N_2$: C, 63.81; H, 3.35; N, 9.30. Found: C, 63.52; H, 3.49; N, 9.17.

3.2.5. *N-[(1E)-(2-Chloroquinolin-3-yl)methylidene]-4-nitroaniline (**4e**)*

Solid, 70%; mp 215°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.26–8.48 (9H, m, Ar-H); 9.04 (1H, s, $-$N = CH), IR (KBr) ν (cm^{-1}): 1622.90. [M$^+$], 311. Calcd. (%) for $C_{16}H_{10}ClN_3O_2$: C, 61.65; H, 3.23; N, 13.48. Found: C, 61.45; H, 3.34; N, 13.36.

3.3. *Preparation of azetidinones (5a)*

In a typical example, triethyl amine (0.01 mol) in 5 ml of dioxane was added to chloroacetyl chloride (0.01 mol) in 5 ml of dioxane cooled to 0°C. To this mixture, Schiff's base **4a** (0.01 mol) in 5 ml of dioxane was added and refluxed for 6 h. After the completion (monitored by TLC), the reaction mixture was poured onto ice-cold water to give a solid, which was filtered and dried. This crude product was purified by column chromatography on silica gel (eluent: ethyl acetate:petroleum ether). Similarly, the compounds **5b–e** were prepared in 67–78% yields.

3.3.1. *3-Chloro-4-(2-chloroquinolin-3-yl)-1-phenylazetidin-2-one (**5a**)*

Solid, 70%; mp 219°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.91 (1H, s, −N−CH−C), 5.71 (1H, s, Cl−CH−C = O), 7.26–8.50 (10H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1735.81 (C = O azetidinone), [M$^+$], 343. Calcd. (%) for $C_{18}H_{12}Cl_2N_2O$: C, 62.99; H, 3.52; N, 8.16. Found: C, 62.88; H, 3.64; N, 8.41.

3.3.2. *3-Chloro-4-(2-chloroquinolin-3-yl)-1-(4-methylphenyl)azetidin-2-one (**5b**)*

Solid, 73%; mp 210°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.60 (3H, s, −CH_3), 4.87 (1H, s, −N−CH−C), 5.89 (1H, s, Cl−CH−C = O), 7.20–8.49 (9H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1735.61 (C = O azetidinone), [M$^+$], 357. Calcd. (%) for $C_{19}H_{14}Cl_2N_2O$: C, 63.88; H, 3.95; N, 7.84. Found: C, 63.78; H, 3.85; N, 7.62.

3.3.3. *3-Chloro-4-(2-chloroquinolin-3-yl)-1-(4-methoxyphenyl)azetidin-2-one (**5c**)*

Solid, 78%; mp 229°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 3.90 (3H, s, −OCH_3), 4.92 (1H, s, −N−CH−C), 5.79 (1H, s, Cl−CH−C = O), 7.23–8.42 (9H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1738.61 (C = O azetidinone), [M$^+$], 377. Calcd. (%) for $C_{19}H_{14}Cl_2N_2O_2$: C, 61.14; H, 3.78; N, 7.51. Found: C, 61.24; H, 3.49; N, 7.39.

3.3.4. *3-Chloro-1-(4-chlorophenyl)-4-(2-chloroquinolin-3-yl)-1-(4-methoxyphenyl)azetidin-2-one (**5d**)*

Solid, 67%; mp 235°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.79 (1H, s, −N−CH−C), 5.70 (1H, s, Cl−CH−C = O), 7.29–8.55 (9H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1738.41 (C = O azetidinone), [M$^+$], 373. Calcd. (%) for $C_{18}H_{11}Cl_3N_2O$: C, 57.25; H, 2.94; N, 7.42. Found: C, 57.39; H, 2.83; N, 7.29.

3.3.5. *3-Chloro-4-(2-chloroquinolin-3-yl)-1-(4-nitrophenyl)azetidin-2-one (**5e**)*

Solid, 78%; mp 245°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.70 (1H, s, −N−CH−C), 5.68 (1H, s, −Cl−CH−C = O), 7.34–8.60 (9H, m, Ar-H), IR (KBr) ν (cm^{-1}): 1738.47 (C = O azetidinone), [M$^+$], 388. Calcd. (%) for $C_{18}H_{11}Cl_2N_3O_3$: C, 55.69; H, 2.86; N, 10.82. Found: C, 55.58; H, 2.68; N, 10.69.

3.4. *Preparation of thieno quinolines (6a)*

A mixture of azetidinones **4a** (0.01 mol) and sodium sulfide (0.01 mol) was refluxed for 10 min on a water bath in ethanol (50 ml). The compound **5a** precipitated out as a yellow crystalline solid.

The mixture was poured onto cold water (500 ml); the resulting solid product **5a** was collected by filtration, washed with ethanol, dried and recrystallized from ethyl acetate. Similarly, the compounds **4b–e** were prepared in 75–85% yields.

3.4.1. *1-Phenyl-2a, 7b-dihydroazeto[2′,3′:4,5]thieno[2,3-b]quinoline (**6a**)*

Solid, 77%; mp 238°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.96 (1H, s, −N−CH−C), 5.89 (1H, s, −S−CH−), 7.35–8.60 (9H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1735.78 (C = O azetidinone), [M$^+$], 304. Calcd. (%) for $C_{18}H_{12}N_2OS$: C, 71.03; H, 3.97; N, 9.20; S, 10.54. Found: C, 71.23; H, 3.83; N, 9.40; S, 10.72.

3.4.2. *1-(4-Methylphenyl)-2a,7b-dihydroazeto[2′,3′:4,5]thieno[2,3-b]quinoline (**6b**)*

Solid, 80%; mp 242°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.60 (3H, s, −CH$_3$), 4.85 (1H, s, −N−CH−C), 5.78 (1H, s, −S−CH−), 7.37–8.50 (9H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1735.66 (C = O azetidinone), [M$^+$], 318. Calcd. (%) for $C_{19}H_{14}N_2OS$: C, 71.67; H, 4.43; N, 8.80; S, 10.07. Found: C, 71.49; H, 3.27; N, 8.69; S, 10.19.

3.4.3. *1-(4-Methoxyphenyl)-2a,7b-dihydroazeto[2′,3′:4,5]thieno[2,3-b]quinoline (**6c**)*

Solid, 85%; mp 269°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 3.90 (3H, s, −OCH$_3$), 4.85 (1H, s, −N−CH−C), 5.68 (1H, s, −S−CH−), 7.29–8.45 (9H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1738.64 (C = O azetidinone), [M$^+$], 334. Calcd. (%) for $C_{19}H_{14}N_2O_2S$: C, 68.24; H, 4.22; N, 8.38; S, 9.59. Found: 68.12, H, 4.39; N, 8.26; S, 9.44.

3.4.4. *1-(4-Chlorophenyl)-2a, 7b-dihydroazeto[2′,3′:4,5]thieno[2,3-b]quinoline (**6d**)*

Solid, 78%; mp 275°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.80 (1H, s, −N−CH−C), 5.73 (1H, s, −S−CH−), 7.34–8.55 (9H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1738.61 (C = O azetidinone), [M$^+$], 338. Calcd. (%) for $C_{18}H_{11}ClN_2OS$: C, 63.81; H, 3.27; N, 8.27; S, 9.46. Found: C, 63.69; H, 3.12; N, 8.15; S, 9.34.

3.4.5. *1-(4-Nitrophenyl)-2a,7b-dihydroazeto[2′,3′:4,5]thieno[2,3-b]quinoline (**6e**)*

Solid, (75%); mp. 285°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.68 (1H, s, −N−CH−C), 5.70 (1H, s, −S−CH−), 7.30–8.60 (9H, m, Ar-H). IR (KBr) ν (cm^{-1}): 1738.64 (C = O azetidinone), [M$^+$], 349. Calcd. (%) for $C_{18}H_{11}N_3O_3S$: C, 61.88; H, 3.17; N, 12.03; S, 9.18. Found: C, 61.74; H, 3.34; N, 12.24; S, 9.36.

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