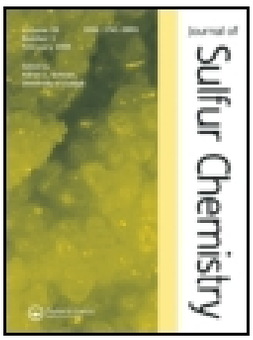

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One-pot three-component synthesis of 2*H*-thiopyrano[2,3-*b*]quinoline-2,3-dicarboxylates from 2-mercaptoquinoline-3-carbaldehydes, dialkyl acetylenedicarboxylates and Ph_3P

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ABSTRACT

An extremely concise one-pot procedure toward the synthesis of 2*H*-Thiopyrano [2,3-*b*]quinoline-2,3-dicarboxylates whose applications as medicines is predictable has been established. This approach produces three fused rings evolving from the formation of four new carbon–carbon bonds and a stereogenic center in a one-pot protocol.

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2-mercaptoquinoline-3-carbaldehyde; intramolecular wittig reaction; thiopyrano[2,3-*b*]quinolines; tricyclic framework; three-component reactions

1. Introduction

The presence of heterocyclic scaffolds in many natural and synthetic products endowed with biological properties is responsible for extensive research in recent years on the synthesis of useful polycyclic structures that include one or more heteroatoms. The quinolines and quinoline-fused heterocycles are privileged structural units presented in numerous natural products and pharmacologically active compounds [1,2].

Over recent decades, these structures have received considerable attention due to their significant biological and medicinal properties, which include antibacterial [3], antiviral [4], antimalarial [5,6], anti-inflammatory [7,8], antihistamine [9], anti-asthmatic [10], antitumor agents [11–13], activities and their potential as HIV-integrase inhibitors [14,15]. Amongst the quinoline derivatives, 2*H*-thiopyrano[2,3-*b*]quinolines are tricyclic structures which contain both a quinoline ring and a thiopyran moiety, which have been found

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Scheme 1. Synthesis of 2-chloro-3-formylquinolines and 3-formyl-2-mercaptoquinolines.

to exhibit several fascinating bioactivities. Consequently, it would seem clear that the fusion of a quinoline unit to thiopyran would generate a molecular architecture possessing interesting bioprofiles.

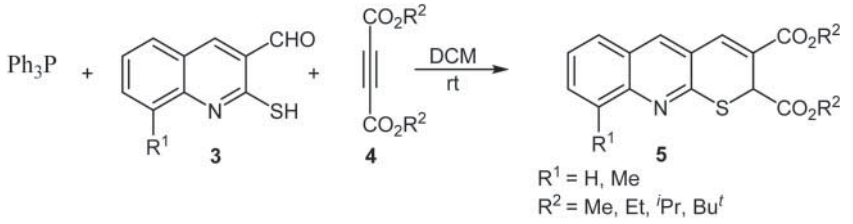
Considering the importance of the above-mentioned applications and activities, methods for the synthesis of quinoline-fused thiopyran structures are of great interest. Previously, Hekmatshoar et al. [16] developed a method to a synthesis of dialkyl 2*H*-1-benzothiopyran-2,3-dicarboxylates using triphenylphosphine, dialkyl acetylenedicarboxylate and 2-mercptobenzaldehyde. The reported approach is associated with certain disadvantages, including numerous difficult steps for the synthesis of the 2-mercaptobenzaldehyde reagent and unacceptably long reaction times. In addition, the product yields are moderate. Therefore, exploring more effective and flexible strategies and improvement in the existing synthetic procedures are highly desirable and are important strategic issues. Therefore, in this study, our goal was to develop an improved procedure, including enhancing the reaction yield of the biologically important thiopyran-fused quinoline moiety.

In continuation of our program directed toward the synthesis of heterocycles [17,18], the development of a simple, rapid and cost-effective method for the construction of quinoline-based sulfur compounds is of value. So we used the above strategy to the synthesis of a new class of thiopyran-based quinolines. The reaction occurred rapidly at room temperature and the final products were obtained in excellent yield. 3-Formyl-2-chloroquinolines **2a**,**b** were prepared from corresponding acetanilides **1a**,**b** via Vilsmeier–Haack reactions according to the literature [19]. The required 3-formyl-2-mercaptoquinolines **3a**,**b** were obtained by stirring respective 2-chloroquinoline-3-carbaldehydes with sodium sulfide in DMF at room temperature (Scheme 1) [20].

2. Results and discussion

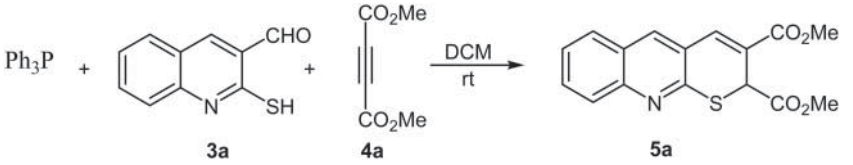
Following our research interest on the synthesis of quinoline-fused heterocyclic compounds [17,18], we herein used the same strategy reported in the literature for the synthesis of 2*H*-thiopyrano[2,3-*b*]quinoline-2,3-dicarboxylates using a one-pot three component coupling of 3-formyl-2-mercaptoquinolines, triphenylphosphine and dialkyl acetylenedicarboxylate (Scheme 2). The reaction was performed at room temperature within 15–30 min using dichloromethane (DCM) as solvent and afforded the products with excellent yields (80–90%).

Different solvents were examined to set up standard reaction conditions (Table 1). Notably, the desired reaction occurred efficiently in dichloromethane at room temperature in 15–30 min. The reaction did not proceed well in DMF and Et_2O at room temperature (entries 1 and 2).



Scheme 2. Synthesis of 2*H*-thiopyrano[2,3-*b*]quinoline- 2,3-dicarboxylates.

Table 1. Optimization of reaction conditions for the formation of **5a**.[a]



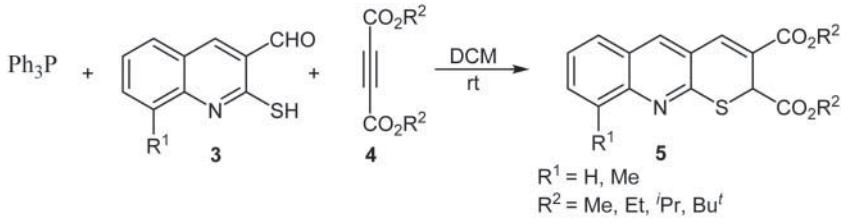
Entry	Solvent	Time (min)	Yield (%)[b]
1	DMF	20	35
2	Et_2O	30	35
3	THF	20	85
4	**CH_2Cl_2**	**15**	**90**
5	EtOAc	25	80
6	Toluene	30	80
7	CH_3CN	25	60

[a]Reaction conditions: (i) **4a** (1 mmol), **3a** (1 mmol), Triphenylphosphine (1 mmol), solvent (5 mL), rt., 15–30 min.
[b]Isolated yield.

To further validate the synthetic flexibility of this methodology, we envisioned applying it to various dialkyl acetylenedicarboxylates and also 3-formyl-2-mercaptoquinolines and the main results of these studies are collected in Table 2.

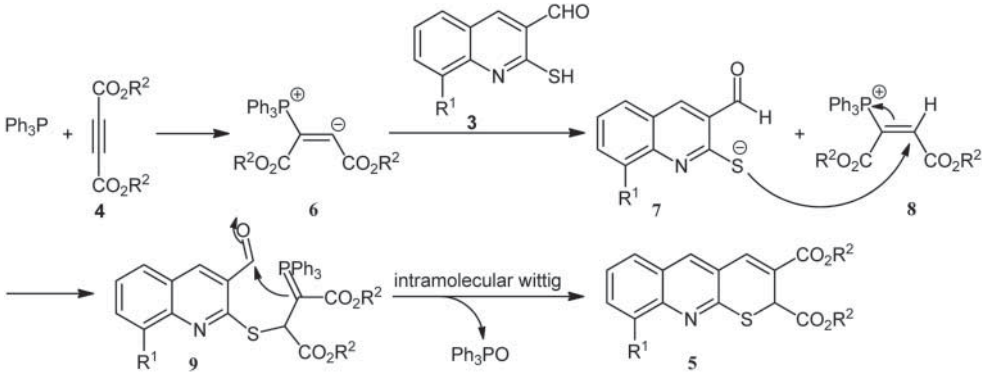
The structure of all the products was confirmed upon careful analysis of the data obtained from IR, mass, ^{1}H NMR and ^{13}C NMR spectra. The mass spectrum of **5a** displayed the molecular ion peak at the appropriate *m/z* value. In the IR spectrum of **5a**, two absorption bands at 1743 and 1703 cm^{-1}, and three absorption bands at 1650, 1613 and 1565 cm^{-1}, which are related to C=O and Ar stretching frequencies, clearly indicated the most significant functional groups of the product. The ^{1}H NMR spectrum of **5a** exhibited two signals at 3.63 and 3.80 ppm, readily recognized as two OMe of two carboxylates. A signal at 5.13 ppm belonged to the aliphatic CH of thiopyran ring; five other signals in the range of 7.55–7.94 ppm belong to the five aromatic hydrogens, which gave rise to characteristic signals in the aromatic region of the spectrum. A signal at 8.46 ppm belonged to the olefinic hydrogen of the thiopyran ring. Observation of 16 distinct signals in the ^{1}H-decoupled ^{13}C NMR spectrum of **5a** is in agreement with the proposed structure. The reaction is assumed to proceed via the formation of a reactive zwitterionic intermediate **6**, which is formed from the reaction of triphenylphosphine with dialkyl acetylenedicarboxylates. The latter is subsequently protonated by the SH proton of the substrate **3**. The resulting anion **7** then attacks the carbon–carbon double bond of intermediate **8** and furnishes the yield **9** that undergoes an intramolecular Wittig reaction with the carbonyl group of the aldehyde moiety to form the desired product **5** (Scheme 3).

Table 2. Synthesis 2*H*-thiopyrano[2,3-*b*]quinoline-2,3-dicarboxylates.[a]



Entry	R^1	R^2	Product	Yield (%)[b]
1	H	Me	**5a**	90
2	H	Et	**5b**	85
3	H	^{i}Pr	**5c**	80
4	H	^{t}Bu	**5d**	87
5	Me	Me	**5e**	87
6	Me	Et	**5f**	83
7	Me	^{t}Bu	**5g**	83

[a]Reaction conditions: (i) **4** (1 mmol), **3** (1 mmol), Triphenylphosphine (1 mmol), DCM (5 mL), rt., 15–30 min.
[b]Isolated yield.



Scheme 3. Plausible mechanism for the formation of 2*H*-thiopyrano[2,3-*b*]quinoline-2,3-dicarboxylates.

3. Conclusion

In conclusion, we have used an efficient and one-pot protocol for the synthesis of *N*,*S*-tricyclic compounds, all of which incorporate thiopyrano[2,3-*b*]quinoline unit. The outcome of the work highlights substantial improvements in the reaction rates and yields without any catalyst and base. Additionally, this protocol offers advantages over the published procedure [16], such as efficiency, simplicity, generality, high yields, short reaction time and ease of product isolation.

4. Experimental

4.1. Materials

All reagents and solvents were of commercial quality and used without further purification. Dichloromethane solvent was completely dried. Analytical thin-layer chromatography

(TLC) was performed on plates precoated with silica-gel layers. Elemental analyses for C, H and N were performed using a Heraeus CHN–O–Rapid analyzer. Mass spectra were recorded on an Aglient Technologies 5975C VL MSD mass spectrometer operating at an ionization potential of 70 eV. Nuclear magnetic resonance spectra (^{1}H, ^{13}C) were recorded on 500 or 300 MHz NMR spectrometers. IR spectra were recorded as KBr pellets on a NICOLET FT-IR 100 spectrometer; absorbencies are reported in cm^{-1}.

4.2. General procedure for the preparation of compounds 5a–g

2-Mercaptoquinoline-3-carbaldehyde (1 mmol) and triphenylphosphine (1 mmol) were taken in dichloromethane (2 mL), and dialkyl acetylenedicarboxylate (1 mmol) was added dropwise to the reaction mixture and stirred for 15–30 min. After conclusion of the reaction (monitored by TLC), the reaction mixture was evaporated and washed with diethyl ether for **5a** and the precipitate thus obtained was filtered. For other compounds **5b–g**, we used column chromatography for the purification.

4.2.1. (RS)-dimethyl 2H-thiopyrano[2,3-b]quinoline-2,3-dicarboxylate (5a)

Yellow powder; mp 185–187°C; yield 0.28 g (90%); IR (KBr) $\bar{\nu}$ cm^{-1}: 1743, 1703, 1650, 1613, 1565; ^{1}H NMR (DMSO) δ (ppm) 3.63 (s, 3 H), 3.80 (s, 3 H), 5.13 (s, 1 H), 7.55 (t, J = 7.4 Hz, 1 H), 7.76 (t, J = 8.0 Hz, 1 H), 7.83 (d, J = 8.2 Hz, 1 H), 7.89 (d, J = 7.9 Hz, 1 H), 7.94 (s, 1 H), 8.46 (s, 1 H); ^{13}C NMR (DMSO) δ (ppm) 40.52 (CHS), 52.6 (C^3–CO_2*Me*), 53.1 (C^2-CO_2*Me*), 123.2 (C^{5a}), 123.6 (C^{4a}), 126.2 (CH^7), 126.8 (CH^6), 127.5 (CH^8), 128.8 (CH^9), 131.8 (CH^5), 137.1 (CH^4), 138.1 (C^3), 147.7 (C^{9a}), 154.7 (C^{10a}), 164.9 (*C*O_2Me), 170.0 (*C*O_2Me); EI-MS: *m*/*z* (%) 315 (14, M^+), 283 (2), 258 (15), 257 (41), 256 (100), 226 (2), 213 (3), 212 (3), 199 (3), 198 (17), 197 (14), 196 (25), 185 (3), 153 (4). Anal. Calcd for $C_{16}H_{13}NO_4S$ (315.34): C, 60.94; H, 4.16; N, 4.44%. Found: C, 60.99; H, 4.10; N, 4.41%.

4.2.2. (RS)-diethyl 2H-thiopyrano[2,3-b]quinoline-2,3-dicarboxylate (5b)

Light yellow powder; mp 137–139°C; yield 0.29 g (85%); IR (KBr): $\bar{\nu}$ cm^{-1} 1738, 1703, 1653, 1613; ^{1}H NMR ($CDCl_3$): δ (ppm) 1.20 (t, J = 7.1 Hz, 3 H), 1.34 (t, J = 7.1 Hz, 3 H), 4.12 (q, J = 7.1 Hz, 2 H), 4.29 (q, J = 7.1 Hz, 2 H), 4.87 (s, 1 H), 7.41 (t, J = 7.1 Hz, 1 H); 7.64 (t, J = 7.6 Hz, 1 H), 7.68 (d, J = 8.2 Hz, 1 H), 7.83 (s, 1 H), 7.87 (d, J = 8.6 Hz, 1 H), 7.91 (s, 1 H); ^{13}C NMR ($CDCl_3$): δ (ppm) 14.1 (OCH_2CH_3), 14.3 (OCH_2CH_3), 41.4 (CHS), 61.8 (OCH_2CH_3), 62.3 (OCH_2CH_3), 123.9 (C^{5a}), 124.1 (C^{4a}), 126.6 (CH^7), 126.7 (CH^6), 128.2 (CH^8), 128.4 (CH^9), 131.5 (CH^5), 137.4 (CH^4), 137.5 (C^3), 148.6 (C^{9a}), 155.5 (C^{10a}), 165.0 (*C*O_2Et), 169.8 (*C*O_2Et); EI-MS: *m*/*z* (%) 343 (10, M^+), 273 (2), 272 (12), 271 (31), 270 (100), 256 (4), 244 (4), 243 (11), 242 (61), 198 (7), 197 (8), 196 (17), 186 (3), 154 (2). Anal. Calcd for $C_{18}H_{17}NO_4S$ (343.40): C, 62.96; H, 4.99; N, 4.08%. Found: C, 62.99; H, 4.01; N, 4.02%.

4.2.3. (RS)-diisopropyl 2H-thiopyrano[2,3-b]quinoline-2,3-dicarboxylate (5c)

Yellow powder; mp 115–117°C; yield: 0.29 g (80%); IR (KBr): $\bar{\nu}$ cm^{-1} 1729, 1643; ^{1}H NMR ($CDCl_3$): δ (ppm) 1.17 (d, J = 6.5 Hz, 3 H), 1.19 (d, J = 6.5 Hz, 3 H), 1.29 (d, J = 6.5 Hz, 3 H), 1.32 (d, J = 6.5 Hz, 3 H), 4.81 (s, 1 H), 4.92 (sept, J = 6.5 Hz, 1 H), 5.14 (sept, J = 6.5 Hz, 1 H), 7.41 (t, J = 7.0 Hz, 1 H), 7.63 (t, J = 7.0 Hz, 1 H), 7.69 (d, J = 7.5 Hz, 1 H), 7.80 (s, 1 H), 7.87 (d, J = 8.5 Hz, 1 H), 7.91 (s, 1 H); ^{13}C NMR ($CDCl_3$): δ (ppm)

21.4 (OCH*Me*$_2$), 21.5 (OCH*Me*$_2$), 21.76 (OCH*Me*$_2$), 21.84 (OCH*Me*$_2$), 41.7 (CH–S), 69.3 (OCHMe$_2$), 69.8 (OCHMe$_2$), 124.0 (C^{5a}), 124.3 (C^{4a}), 126.5 (CH^7), 126.5 (CH^6), 128.0 (CH^8), 128.2 (CH^9), 131.2 (CH^5), 137.0 (CH^4), 137.1 (C^3), 148.4 (C^{9a}), 155.5 (C^{10a}), 164.3 (*C*O$_2{}^i$Pr), 169.2 (*C*O$_2{}^i$Pr); EI-MS: *m*/*z* (%) 372 (M$^+$ + 1, 28), 371 (M$^+$, 38), 370 (5), 329 (2), 312 (2), 300 (2), 287 (5), 286 (24), 285 (54), 284 (95), 283 (26), 270 (10), 269 (19), 258 (3), 256 (5), 244 (24), 243 (52), 242 (100), 241 (24), 214 (7), 199 (5), 198 (22), 197 (38), 196 (53), 186 (9), 185 (4), 170 (3), 154 (7), 153 (12), 152 (9), 140 (4), 128 (6), 127 (5), 126 (4), 115 (3), 101 (2), 85 (2), 69 (5). Anal. Calcd for $C_{20}H_{21}NO_4S$ (371.45): C, 64.67; H, 5.70; N, 3.77%. Found: C, 64.61; H, 5.79; N, 3.71%.

4.2.4. (RS)-di-tert-butyl 2H-thiopyrano[2,3-b]quinoline-2,3-dicarboxylate (5d)

Cream powder; mp 157–159°C; yield 0.35 g (87%); IR (KBr) $\bar{\nu}$ cm^{-1}: 1718, 1644, 1613, 1556; ^{1}H NMR (CDCl$_3$) δ (ppm) 1.37 (s, 9 H), 1.54 (s, 9 H), 4.73 (s, 1 H), 7.40 (t, J = 7.3 Hz, 1 H), 7.62 (dt, J = 8.1 Hz, J = 1.0 Hz, 1 H), 7.68 (d, J = 8.2 Hz, 1 H), 7.72 (s, 1 H), 7.87 (d, J = 8.0 Hz, 1 H), 7.87 (s, 1 H); ^{13}C NMR (CDCl$_3$) δ (ppm) 27.9 (OC*Me*$_3$), 28.2 (OC*Me*$_3$), 42.9 (CHS), 82.2 (O*C*Me$_3$), 82.5 (O*C*Me$_3$), 124.3 (C^{5a}), 125.8 (C^{4a}), 126.5 (CH^7), 126.7 (CH^6), 128.2 (CH^8), 128.3 (CH^9), 131.2 (CH^5), 136.4 (CH^4), 136.9 (C^3), 148.5 (C^{9a}), 155.9 (C^{10a}), 164.0 (*C*O$_2{}^t$Bu), 168.9 (*C*O$_2{}^t$Bu); EI-MS: *m*/*z* (%) 300 (4), 299 (12), 298 (49), 271 (2), 270 (9), 245 (2), 244 (13), 243 (35), 242 (100), 198 (6), 197 (6), 196 (11), 186 (2), 58 (5), 57 (88), 41 (7). Anal. Calcd for $C_{22}H_{25}NO_4S$ (399.50): C, 66.14; H, 6.31; N, 3.51%. Found: C, 66.10; H, 6.35; N, 3.56%.

4.2.5. (RS)-dimethyl 9-methyl-2H-thiopyrano[2,3-b]quinoline-2,3-dicarboxylate (5e)

Yellow powder; mp 164–166°C; yield 0.29 g (87%); IR (KBr) $\bar{\nu}$ cm^{-1}: 1741, 1705, 1646, 1610, 1573; ^{1}H NMR (CDCl$_3$) δ (ppm) 2.70 (s, 3 H), 3.71 (CO$_2$*Me*), 3.87 (CO$_2$*Me*), 4.91 (s, 1 H), 7.33 (t, J = 7.6 Hz, 1 H), 7.51 (d, J = 7.0 Hz, 1 H), 7.55 (d, J = 8.0 Hz, 1 H), 7.85 (s, 1 H), 7.89 (s, 1 H); ^{13}C NMR (CDCl$_3$) δ (ppm) 17.9 (Me), 41.3 (CHS), 52.8 (CO$_2$*Me*), 53.3 (CO$_2$*Me*), 123.1 (C^{5a}), 123.6 (C^{4a}), 126.4 (CH^8), 126.5 (CH^7), 126.6 (CH^6), 131.7 (CH^5), 136.5 (C^9), 137.96 (CH^4), 138.01 (C^3), 147.8 (C^{9a}), 154.0 (C^{10a}), 165.6 (*C*O$_2$Me), 170.4 (*C*O$_2$Me); EI-MS: *m*/*z* (%) 330 (3, M$^+$ + 1), 329 (17, M$^+$), 297 (2), 273 (2), 272 (15), 271 (39), 270 (100), 227 (3), 213 (2), 212 (13), 211 (10), 210 (21), 199 (2), 179 (3), 178 (2), 166 (3). Anal. Calcd for $C_{17}H_{15}NO_4S$ (329.37): C, 61.99; H, 4.59; N, 4.25%. Found: C, 61.90; H, 4.55; N, 4.29%.

4.2.6. (RS)-diethyl 9-methyl-2H-thiopyrano[2,3-b]quinoline-2,3-dicarboxylate (5f)

Yellow powder; mp 165–167°C; yield 0.29 g (83%); IR (KBr): $\bar{\nu}$ cm^{-1} 1730, 1692, 1578; ^{1}H NMR (CDCl$_3$): δ (ppm) 1.24 (t, J = 6.5 Hz, 3 H), 1.37 (t, J = 7.5 Hz, 3 H), 2.72 (s, 3 H), 4.15 (m, 2 H), 4.32 (m, 2 H), 4.89 (s, 1 H), 7.35 (t, J = 7.5 Hz, 1 H); 7.52 (d, J = 7.0 Hz, 1 H), 7.58 (d, J = 8.5 Hz, 1 H), 7.87 (s, 1 H), 7.93 (s, 1 H); ^{13}C NMR (CDCl$_3$): δ (ppm) 13.9 (OCH$_2$*C*H$_3$), 14.2 (OCH$_2$*C*H$_3$), 17.7 (Me), 41.5 (CHS), 61.6 (OCH$_2$CH$_3$), 62.1 (OCH$_2$CH$_3$), 123.5 (C^{5a}), 123.6 (C^{4a}), 126.2 (CH^8), 126.3 (CH^7), 126.5 (CH^6), 131.5 (CH^5), 136.4 (C^9), 137.5 (CH^4), 137.6 (C^3), 147.7 (C^{9a}), 154.1 (C^{10a}), 165.0 (*C*O$_2$Et), 169.9 (*C*O$_2$Et); EI-MS: *m*/*z* (%) 357 (8, M$^+$), 356 (2), 312 (6), 286 (7), 285 (20), 284 (100), 270 (2), 258 (4), 257 (10), 256 (57), 255 (2), 228 (3), 212 (6), 211 (8) 210 (17), 209 (4), 200 (2), 179 (2), 178 (4), 167 (3), 166 (4), 140 (3), 139 (3), 69 (6), 57 (17), 55 (3). Anal. Calcd for $C_{19}H_{19}NO_4S$ (357.42): C, 63.85; H, 5.36; N, 3.92%. Found: C, 63.80; H, 5.32; N, 3.99%.

4.2.7. (RS)-di-tert-butyl 9-methyl-2H-thiopyrano[2,3-b]quinoline-2,3-dicarboxylate (5g)

Yellow powder; mp 158–160°C; yield 0.34 g (83%); IR (KBr) $\bar{\nu}$ cm^{-1}: 1726, 1693, 1575, 1472; ^1H NMR ($CDCl_3$) δ (ppm) 1.41 (s, 9 H), 1.57 (s, 9 H), 2.73 (s, 3 H), 4.77 (s, 1 H), 7.34 (t, J = 7.0 Hz, 1 H), 7.51 (d, J = 7.0 Hz, 1 H), 7.56 (d, J = 8.0 Hz, 1 H), 7.75 (s, 1 H), 7.88 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ (ppm) 17.9 (Me), 27.8 (OC*Me*$_3$), 28.1 (OC*Me*$_3$), 43.0 (CHS), 82.1 (OCMe$_3$), 82.3 (OCMe$_3$), 123.8 (C^{5a}), 125.4 (C^{4a}), 126.16 (CH^8), 126.18 (CH^7), 126.5 (CH^6), 131.4 (CH^5), 136.1 (C^9), 136.4 (CH^4), 137.3 (C^3), 147.3 (C^{9a}), 154.6 (C^{10a}), 164.0 ($CO_2{}^t$Bu), 169.0 ($CO_2{}^t$Bu); EI-MS: m/z (%) 413 (6, M^+), 314 (27), 313 (68), 312 (100), 284 (34), 259 (11), 258 (65), 257 (84), 256 (79), 255 (72), 212 (24), 211 (54), 210 (78), 209 (16), 178 (14), 166 (15), 139 (9), 58 (15), 57 (98). Anal. Calcd for $C_{23}H_{27}NO_4S$ (413.53): C, 66.80; H, 6.58; N, 3.39%. Found: C, 66.85; H, 6.52; N, 3.32%.

Disclosure statement

No potential conflict of interest was reported by the authors.

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