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EXPERIMENTAL PAPER

Friedländer Synthesis of Novel Polycyclic Quinolines Using Solid SiO_2/H_2SO_4 Catalyst

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In the current milieu of synthetic organic chemistry, environmental, economic and industrial issues have caused practitioners to re-examine the significance and value of heterogeneous catalysts.[1,2] Among the most useful of these is silica gel/sulfuric acid (SSA),[3,4] readily prepared and easily deployed in the synthesis laboratory.[5,6] Given the widely-recognized usefulness of quinolines in drug design[7,8] and in diversity studies,[9–12] we turned our attention to the preparation of the title compounds using SSA as an improved catalyst for the Friedlander reaction. To the best of our knowledge, this is the first such report. We made and rigorously characterized novel polycyclic quinoline derivatives by the SSA catalyzed condensation of 2-aminoarylketones **1** with 1,2- or 1,3-dicarbonyl compounds. Thus **1** reacted with 1,2-cyclohexanedione or with 1,3-cyclohexanedione to give 2,3-dihydroacridin-4-ones **3** (Scheme 1) and 3,4-dihydroacridin-1-ones **5** (Scheme 2), respectively.

We optimized our reaction conditions using the preparation of compound **3a** as our model, and the results are summarized in Table 1. Using thin layer chromatography to check the reaction endpoint, we examined the model with respect to catalyst (10 mol%), solvent, temperature and time (see Experimental section). Based on yields, the best results (Table 1, entry 5) employed SSA in methanol at reflux for 2 hours and resulted in **3a** in 92% yield. The structure of **3a** was supported by elemental analysis, by FT-IR and NMR spectra, and by single-crystal X-ray diffraction studies (Figure 1).

In extending our preparations to the other compounds designated in Scheme 1, we found that yields were excellent (mean 92%). The crude products were isolated by simple filtration and were purified by recrystallization from ethyl acetate. No chromatographic procedure was required to obtain pure products.

Application of our SSA catalyzed Friedlander procedure was also made to the reactions shown in Scheme 2, in which the diketone was 1,3-cyclohexanedione. Similar results were obtained in this family of compounds. The range of yields was 90-95%. The compounds were rigorously characterized, and the single crystal X-ray structure of **5c** is shown in Figure 2.

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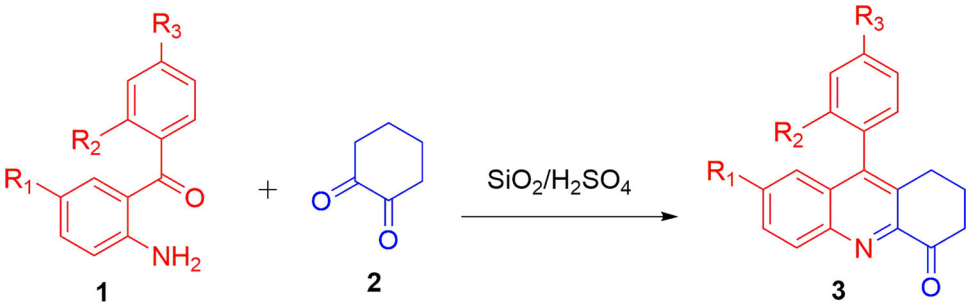
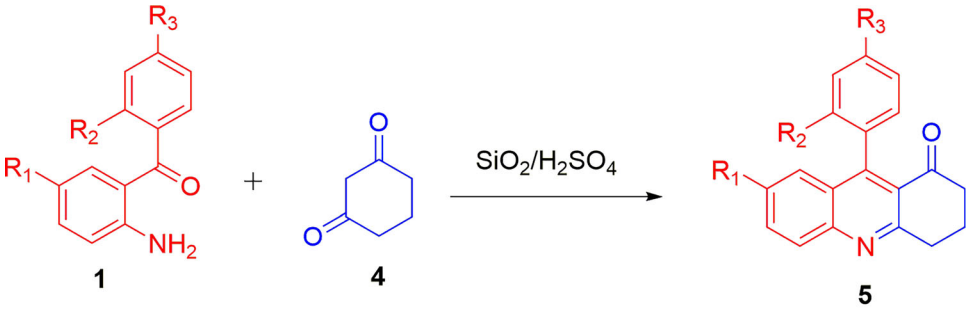
Supplemental data for this article can be accessed here.



1a, **3a**, R_1= H, R_2= H, R_3= H; **1b**, **3b**, R_1= Cl, R_2= H, R_3= H;
1c, **3c**, R_1= Cl, R_2= Cl, R_3= H; **1d**, **3d**, R_1= Cl, R_2= F, R_3= H;
1e, **3e**, R_1= Br, R_2= F, R_3= H; **1f**, **3f**, R_1= H, R_2= H, R_3= Br

Scheme 1. Synthesis of 2,3-dihydroacridin-4-ones (**3**).



1a, **5a**, R_1= H, R_2= H, R_3= H; **1b**, **5b**, R_1= Cl, R_2= H, R_3= H;
1c, **5c**, R_1= Cl, R_2= Cl, R_3= H; **1d**, **5d**, R_1= NO_2, R_2= H, R_3= H;
1e, **5e**, R_1= H, R_2= H, R_3= Br; **1f**, **5f**, R_1= H, R_2= H, R_3= F

Scheme 2. Synthesis of 3,4-dihydroacridin-1-ones (**5**).

Table 1. Optimization of reaction conditions for preparation of **3a**.

Entry	Catalyst	Solvent	Temperature	Time[a]	Yield[b] (%)
1.	–	CH_3OH	Reflux	5	42
2.	SiO_2	CH_3OH	Reflux	5	52
3.	H_2SO_4	CH_3OH	Reflux	8	65
4.	SiO_2/H_2SO_4	CH_3OH	RT	12	60
5.	**SiO_2/H_2SO_4**	**CH_3OH**	**Reflux**	**2**	**92**
6.	SiO_2/H_2SO_4	CH_3CN	Reflux	6	70
7.	SiO_2/H_2SO_4	H_2O	Reflux	2	66
8.	SiO_2/H_2SO_4	CH_3COOH	Reflux	6	80
9.	SiO_2/H_2SO_4	C_2H_5OH	Reflux	4	85
10.	SiO_2/H_2SO_4	–	neat	3	75
11.	Recycled SSA	CH_3OH	Reflux	2	90

[a]Time duration of reaction in hours.
[b]Isolated pure products.

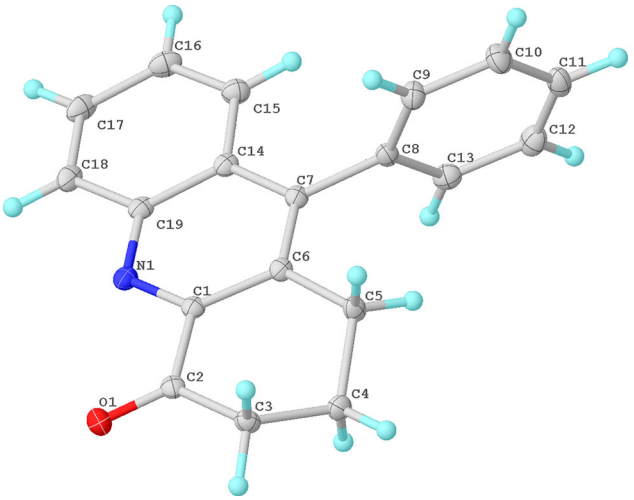


Figure 1. ORTEP crystal structure of compound **3a**.

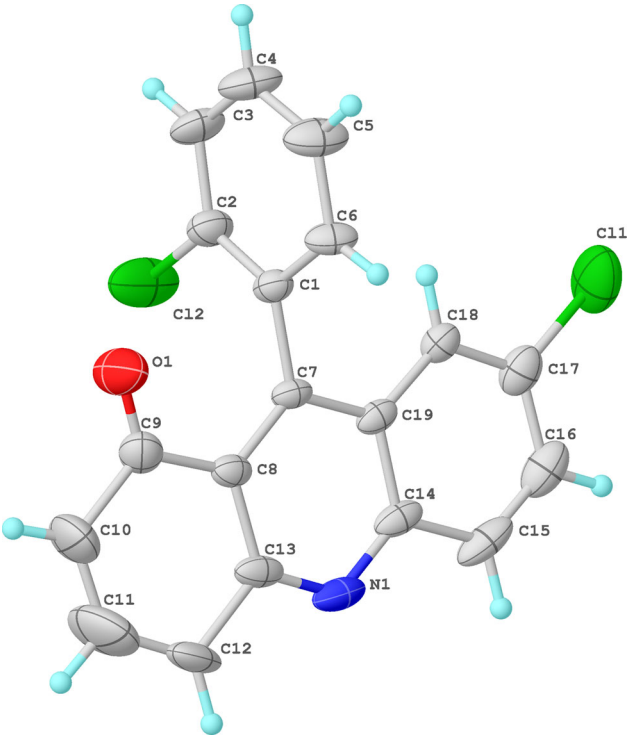


Figure 2. ORTEP crystal structure of compound **5c**.

In conclusion, we have presented our results on the SSA-catalyzed Friedlander preparation of novel polycyclic quinolines. These were formed from the reactions of *o*-aminoarylketones with 1,2- and 1,3-cyclohexanediones. Compared to previous catalysts used for these reactions,[13–16] the present methods combine the important advantages of high yields, mild reaction conditions, simplicity of operations, and the use of a readily-available and inexpensive catalyst.

Table 2. Characterization details for compounds **3** and **5**.

Entry	Product	Time (hrs)	Yield[a] (%)	Molecular Formula	M.p (°C) (Lit. M.p.)	FT-IR (KBr, cm^{-1}) ν_{max}	^{1}H NMR (400 MHz, $CDCl_3$) (ppm) δ	^{13}C NMR (100 MHz, $CDCl_3$) (ppm) δ	Elemental Analysis (HR-MS)
1.	**3a**	2	92	$C_{19}H_{15}NO$	213-215	1701, 1598	2.056-2.119 (m, 2H, C_4-CH_2), 2.796-2.872 (m, 4H, C_3-CH_2, C_5-CH_2), 7.207-7.234 (m, 2H, C_9-, C_{13}-H), 7.367-7.440 (m, 2H, C_{16}-, C_{17}- H,), 7.453-7.532 (m, 3H, C_{10}-, C_{11}-, C_{12}-H), 7.634-7.676 (m, 1H, C_{15}-H), 8.327 (d, 1H, C_{18}-H, $J = 8.0$ Hz)	22.60, 27.99, 40.22, 125.85, 128.37, 128.72, 128.84, 129.16, 129.51, 131.52, 133.46, 136.14, 147.14, 148.15, 148.41, 197.59	Calcd. : C, 83.49; H, 5.53; N, 5.12; Found: C, 83.40; H, 5.61; N, 5.06. ($C_{19}H_{15}NO$ $[M+H]^+$: Found: 274.123; Calcd.: 274.119)
2.	**3b**	2	93	$C_{19}H_{14}ClNO$	250-252 (252-254[18])	1700, 1602	2.093-2.125 (m, 2H, C_4-CH_2), 2.809-2.893 (m, 4H, C_3-CH_2, C_5-CH_2), 7.217-7.241 (m, 2H, C_9-, C_{13}-H), 7.369 (d, 1H, C_{15}-H, J_m = 2.40 Hz), 7.509-7.571 (m, 3H, C_{10}-, C_{11}-, C_{12}-H), 7.613 (dd, 1H, C_{17}-H, J_m= 2.40 Hz, J_o= 8.80 Hz), 8.285 (d, 1H, C_{18}-H, $J = 8.80$ Hz)	22.41, 28.03, 40.13, 124.61, 128.72, 129.07, 129.37, 130.72, 133.11, 134.44, 135.02, 135.40, 145.53, 147.43, 148.56, 197.22	Calcd. : C, 74.15; H, 4.59; N, 4.55; Found: C, 74.21; H, 4.63; N, 4.48.
3.	**3c**	2	93	$C_{19}H_{13}Cl_2NO$	185-187 (188-190[18])	1700, 1599	2.129-2.162 (m, 2H, C_4-CH_2), 2.690-2.820 (m, 2H, C_3-CH_2), 2.878-2.914 (m, 2H, C_5-CH_2), 7.177-7.230 (m, 2H, C_{11}-, C_{12}-H), 7.452-7.491 (m, 2H, C_{10}-, C_{13}-H),7.593-7.647 (m, 2H, C_{15}-, C_{17}-H), 8.310 (d, 1H, C_{18}-H, $J = 8.80$ Hz)	22.20, 27.38, 40.14, 123.94, 127.53, 128.91, 130.28, 130.46, 130.65, 130.93, 133.25, 133.31, 134.30, 135.06, 135.40, 144.51, 145.57, 148.63, 197.04	Calcd. : C, 66.69; H, 3.83; N, 4.09; Found : C, 66.62; H, 3.88; N, 4.02.
4.	**3d**	2.5	90	$C_{19}H_{13}ClFNO$	199-201 (198-200[18])	1705, 1597	1.981-2.059 (m, 2H, C_4-CH_2), 2.643-2.806 (m, 4H, C_3-CH_2, C_5-CH_2), 7.076-7.119 (m, 1H, C_{12}-H), 7.138-7.186 (m, 1H, C_{10}-H), 7.208-7.249 (m, 2H, C_{11}-, C_{13}-H), 7.390-7.447 (m, 1H, C_{15}-H), 7.511 (dd, 1H, C_{17}-H, J_m= 2.40 Hz, J_o= 8.80 Hz), 8.182 (d, 1H, C_{18}-H, $J = 8.80$ Hz)	22.24, 27.57, 40.10, 116.36, 116.57, 122.76, 124.06, 124.83, 129.26, 130.87, 133.27, 135.43, 141.27, 145.52, 148.49, 158.26, 160.72, 196.97	Calcd.: C, 70.05; H, 4.02; N, 4.30; Found : C, 70.15; H, 4.12; N, 4.19.
5.	**3e**	2	91	$C_{19}H_{13}BrFNO$	201-203	1703, 1599	2.094-2.187 (m, 2H, C_4-CH_2), 2.760-2.928 (m, 4H, C_3-CH_2, C_5-CH_2), 7.195-7.217 (m, 1H, C_{12}-H), 7.262-7.372 (m, 2H, C_{11}-, C_{13}-H), 7.514-7.571 (m, 2H, C_{10}-, C_{15}-H), 7.769 (dd, 1H, C_{17}-H, J_m= 2.40 Hz, J_o= 8.80 Hz), 8.231 (d, 1H, C_{18}-H, $J = 8.80$ Hz)	22.24, 27.57, 40.11, 116.49, 122.57, 123.97, 124.84, 127.44, 129.66, 131.21, 133.30, 133.43, 135.48, 141.19, 145.71, 148.57, 158.27, 160.73, 196.97	Calcd. : C, 61.64; H, 3.54; N, 3.78; Found C, 61.58; H, 3.59; N, 3.68. ($C_{19}H_{13}BrFNO$ $[M+H]^+$: Found: 370.023; Calcd. 370.221)
6.	**3f**	2	93	$C_{19}H_{14}FNO$	244-246	1701, 1598	2.095-2.159 (m, 2H, C_4-CH_2), 2.813-2.843 (m, 2H, C_3-CH_2), 2.872-2.905 (m, 2H, C_5-CH_2), 7.144 (d, 2H, C_9-, C_{13}-H, $J = 8.0$ Hz), 7.382 (dd, 1H, C_{17}-H, J_m= 1.60 Hz, J_o= 8.80 Hz), 7.468-7.510 (m, 1H, C_{15}-H), 7.674-7.722 (m, 3H, C_{10}-, C_{12}-, C_{16}-H), 8.362 (d, 1H, C_{18}-H, $J = 8.80$ Hz)	22.54, 27.99, 40.16, 122.76, 125.48, 128.43, 129.01, 129.68, 130.90, 131.68, 132.17, 133.34, 135.01, 146.78, 147.17, 148.41, 197.31	Calcd. : C, 78.33; H, 4.84; N, 4.81; Found C, 78.39; H, 4.76; N, 4.89. ($C_{19}H_{14}FNO$ $[M+H]^+$: Found: 352.033 Calcd. : 352.236)
7.	**5a**	2	95	$C_{19}H_{15}NO$	152-154 (151-153[19])	1693, 1558	2.193-2.258 (m, 2H, C_3-CH_2), 2.662-2.695 (m, 2H, C_4-CH_2), 3.337-3.368 (m, 2H, C_2-CH_2), 7.139-7.163 (m, 2H, C_9-, C_{13}-H), 7.351-7.392 (m, 1H, C_{17}- H), 7.424-7.488 (m, 4H, C_{10}-, C_{11}-, C_{12}-, C_{16}- H), 7.713-7.755 (m, 1H, C_{15}-H), 8.040 (d, 1H, C_{18}-H, $J = 8.4$ Hz)	21.40, 34.62, 40.66, 123.90, 126.45, 127.57, 128.05, 128.13, 128.26, 128.50, 131.76, 137.67, 148.69, 151.50, 162.27, 197.98	Calcd.: C, 83.49; H, 5.53; N, 5.12; Found C, 83.41; H, 5.61; N, 5.04. ($C_{19}H_{15}NO$ $[M+H]^+$: Found: 274.122 Calcd. : 274.119)

(*continued*)

Table 2. Continued.

Entry	Product	Time (hrs)	Yield[a] (%)	Molecular Formula	M.p (°C) (Lit. M.p.)	FT-IR (KBr, cm^{-1}) ν_{max}	^{1}H NMR (400 MHz, $CDCl_3$) (ppm) δ	^{13}C NMR (100 MHz, $CDCl_3$) (ppm) δ	Elemental Analysis (HR-MS)
8.	**5b**	2	92	$C_{19}H_{14}ClNO$	185-187 (185-186[20])	1686, 1556	2.189-2.254 (m, 2H, C_3-CH_2), 2.661-2.694 (m, 2H, C_4-CH_2), 3.313-3.344 (m, 2H, C_2-CH_2), 7.117-7.142 (m, 2H, C_9-, C_{13}-H), 7.387 (d, 1H, C_{15}-H, J_m = 2.40 Hz), 7.463-7.514 (m, 3H, C_{10}-, C_{11}-, C_{12}-H), 7.659 (dd, 1H, C_{17}-H, J_m= 2.40 Hz, J_o= 8.80 Hz), 7.970 (d, 1H, C_{18}-H, J = 8.80 Hz)	21.28, 34.55, 40.60, 124.48, 126.75, 127.92, 128.01, 128.31, 128.34, 130.22, 132.44, 132.59, 136.87, 147.09, 150.53, 162.55, 197.70.	Calcd.: C, 74.15; H, 4.59; N, 4.55; Found C, 74.08; H, 4.51; N, 4.62.
9.	**5c**	2	93	$C_{19}H_{13}Cl_2NO$	194-196 (192-194[20])	1692; 1556	2.197-2.267 (m, 2H, C_3-CH_2), 2.620-2.773 (m, 2H, C_4-CH_2), 3.333-3.370 (m, 2H, C_2-CH_2), 7.078 (dd, 1H, J_m= 2.00 Hz, J_o= 7.20 Hz, C_{11}-H), 7.273 (d, 1H, J = 2.40 Hz, C_{15}-H), 7.371-7.452 (m, 2H, C_{10}, C_{13}-H), 7.525 (dd, 1H, J_m= 2.00 Hz, J_o= 7.20 Hz, C_{12}-H), 7.682 (dd, 1H, J_m= 2.40 Hz, J_o= 8.80 Hz, C_{17}-H), 8.002 (d, 1H, J = 8.80 Hz, C_{18}-H)	21.26, 34.41, 40.13, 124.55, 125.92, 126.91, 127.47, 129.37, 129.46, 129.62, 130.40, 132.12, 132.86, 135.97, 147.09, 162.51, 197.36	Calcd.: C, 66.68; H, 3.83; N, 4.09. Found: C, 66.63; H, 3.89; N, 4.15.
10.	**5d**	2.5	90	$C_{19}H_{14}N_2O_3$	188-190 (186-188[20])	1694; 1552	2.227-2.292 (m, 2H, C_3-CH_2), 2.704-2.737 (m, 2H, C_4-CH_2), 3.374-3.406 (m, 2H, C_2-CH_2), 7.147- 7.174 (m, 2H, C_{10}, C_{12}-H), 7.509-7.535 (m, 3H, C_9, C_{11}, C_{13}-H), 8.151 (d, 1H, J = 9.20 Hz, C_{18}-H), 8.390 (d, 1H, J = 2.40 Hz, C_{15}-H), 8.475 (dd, 1H, J_m= 2.40 Hz, J_o= 9.20 Hz, C_{17}-H)	21.03, 34.85, 40.47, 124.91, 125.05, 125.33, 126.81, 128.05, 128.57, 130.59, 135.81, 145.58, 150.48, 153.25, 166.14, 197.10.	Calcd.: C, 71.69; H, 4.43; N, 8.80. Found: C, 71.66; H, 4.47; N, 8.84.
11.	**5e**	2	92	$C_{19}H_{14}BrNO$	224-226	1682, 1560	2.386-2.451 (m, 2H, C_3-CH_2), 2.861-2.894 (m, 2H, C_4-CH_2), 3.565-3.597 (m, 2H, C_2-CH_2), 7.220 (d, 2H, C_{10}-, C_{12}-H, J = 8.40 Hz), 7.600 (d, 2H, C_{16}-, C_{17}-H, J = 4.00 Hz), 7.793 (d, 2H, C_9-, C_{13}-H, J = 8.40 Hz), 7.934-7.976 (m, 1H, C_{15}-H), 8.315 (d, 1H, C_{18}-H, J = 8.80 Hz)	21.03, 33.69, 40.49, 121.89, 123.88, 126.92, 127.29, 127.83, 129.72, 131.40, 132.29, 136.38, 148.01, 150.78, 162.33, 197.84	Calcd.: C, 64.79; H, 4.01; N, 3.98; Found C, 64.71; H, 4.09; N, 3.88. ($C_{19}H_{14}BrNO$ $[M+H]^+$: Found: 352.033 Calcd. : 352.231)
12.	**5f**	2	93	$C_{19}H_{14}FNO$	166-168	1680, 1563	2.253-2.318 (m, 2H, C_3-CH_2), 2.727-2.760 (m, 2H, C_4-CH_2), 3.430-3.462 (m, 2H, C_2-CH_2), 7.164-7.252 (m, 4H, C_9-, C_{10}-, C_{12}-, C_{13}-H), 7.446-7.516 (m, 2H, C_{16}-, C_{17}-H), 7.792-7.837 (m, 1H, C_{15}-H), 8.180 (d, 1H, C_{18}-H, J = 8.40 Hz)	21.09, 33.85, 40.56, 115.28, 124.14, 126.79, 127.94, 127.97, 129.78, 129.86, 132.12, 148.11, 150.93, 161.16, 162.33, 163.61, 197.88	Calcd.: C, 78.33; H, 4.84; N, 4.81; Found C, 78.41; H, 4.78; N, 4.89. ($C_{19}H_{14}FNO$ $[M+H]^+$: Found: 292.113 Calcd. : 292.109)

Reaction conditions: 2-aminobenzophenone **1a** (1 mmol) and 1,2-cyclohexanedione **2**/1,3-cyclohexanedione **3** (1.2 mmol, 1.2 equiv.) in the presence of SiO_2/H_2SO_4 (10 mol%) as a catalyst.
[a]Recrystallized pure products.

Experimental section

All the reagents and chemicals were purchased from Sigma Aldrich and AKSci. When known compounds had to be prepared according to literature procedures, pertinent references are given. The purity of the products was tested by TLC on silica gel 60 F254 25 aluminum backed sheets, 20 X 20 cm (Merck), using petroleum ether and ethyl acetate in the ratio of 75:25 as developing solvents. FT-IR spectra were obtained on a BRUKER brand, model VECTOR 22, instrument. NMR spectra were taken on a BRUKER AVANCE III HD-400 [400 MHz (^{1}H) and 100 MHz (^{13}C)] instrument. Melting points were determined on a Kofler Thermogerate apparatus and are uncorrected. Elemental analyses were obtained on a Vario EL III model CHNS (Germany) analyzer at the Department of Chemistry, Bharathiar University.

Preparation of SSA[17]

To prepare SSA, 0.5 g of silica and 0.4 mL of conc. sulfuric acid were stirred thoroughly for 30 min. The mixture was heated in a hot air oven at 85°C for 1h, cooled to room temperature, and then it was used for reactions.

General procedure for the synthesis of 9-aryl-2,3-dihydro-1H-acridin-4-ones 3 and 3,4-dihydroacridin-1-ones 5

The appropriate 2-amino-arylketone (**1,** Sigma-Aldrich, 1 mmol) and cyclic dione (**2** or **4**, 1.2 mmol) were dissolved in methanol (5 mL) and refluxed with freshly prepared SiO_2/H_2SO_4 (0.1 mmol) for 2 hrs. The completion of the reaction was monitored by TLC. The product was isolated from the reaction mixture by simple filtration, then evaporation of methanol. The obtained product was purified through recrystallization using ethyl acetate to yield the corresponding products **3** and **5**, as indicated. Complete characterization data are provided in Table 2.

Acknowledgment

Dr. Rajendran Satheeshkumar is thankful for the financial support of FONDECYT for a postdoctoral fellowship (Project No. 3190292), which is gratefully acknowledged.

Supplementary data

CIF files for compounds **3a** and **5c** have been deposited with the Cambridge Crystallographic Data Centre as CCDC number 1962353 and 1989190. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK, Fax: +44 (0) 1223 336033, or email: deposit@ccdc.cam.ac.uk.

Supplementary information

Copies of FT-NMR and HR-MS spectral data of the synthesized compounds and a table of crystal data are available as supporting information in the online version of this article or from the corresponding author upon request.

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