Synthesis of 4-Quinolones via Cyclocondensation of Substituted *ortho*-Amidoacetophenones: A Refit to the Camps Cyclization by Applying Trimethylsilyl Trifluoromethanesulfonate/Triethylamine

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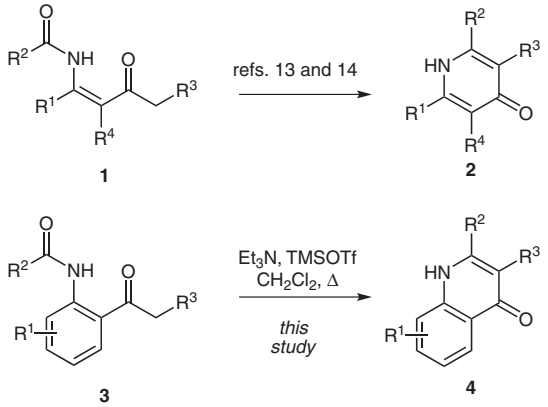
Abstract: A modification of the classical Camps cyclization is described. A series of substituted 4-quinolone derivatives is prepared via trimethylsilyl trifluoromethanesulfonate/triethylamine induced cyclocondensation of substituted *ortho*-amidoacetophenones. The process shows a broad substrate scope and allows selective preparation of 2-aryl- and 2-alkyl-substituted 4-quinolones. Enantiopure starting materials react without loss of optical purity using the modified conditions. Subsequent transformations of the products involving preparation of a 4-quinolyl nonaflate and O-selective methylation are also described.

Key words: Camps cyclization, 4-quinolones, cyclocondensation, *ortho*-amidoacetophenones, nonaflates

Substituted 4-quinolone derivatives represent an important class of nitrogen-containing heterocycles as they often exhibit specific biological activities.[1] For instance, many broad-spectrum antibiotics such as the fluoroquinolones feature a 4-quinolone core.[2] In addition to their antibacterial properties, quinolone derivatives have been identified as anti-malarial,[3] anti-cancer,[4] anti-viral[5] and anti-diabetic agents,[6] thus new methods for construction of the quinolone core are highly desirable. Despite recent approaches involving transition metal catalyzed bond-forming processes,[7] and other strategies,[8] classical synthetic methods such as the Conrad–Limpach,[9] the Gould–Jacobs[10] and the Camps cyclization[11] are still widely employed for the construction of substituted 4-quinolones. However, all these methods feature thermal cyclocondensation reactions, typically requiring harsh conditions employing high temperatures (>200 °C) and/or strong bases. This limits the scope of these methods and often results in a tedious purification. Since the required starting materials are easily available these methods are still of synthetic relevance. The Camps cyclization, which proceeds via base-induced cyclocondensation of *ortho*-amidoacetophenones, still attracts significant attention and some modified versions have been reported.[12] However, all the protocols developed so far require strong bases such as sodium hydroxide or metal alkoxides, which leads to a rather limited substrate scope. In addition, base-sensitive 2-alkyl-4-quinolones cannot be accessed selectively using these methods since the reaction conditions lead to formation of mixtures of regioisomeric 2- and 4-quinolones.[11a,12c] Herein we describe a modification of the classical Camps cyclization conditions, which features milder reaction conditions and does not require the use of a strong base.

Our group has previously developed a new approach to highly functionalized 4-pyridinone/4-hydroxypyridine derivatives **2** based on a trimethylsilyl trifluoromethanesulfonate (TMSOTf) promoted cyclocondensation reaction of β-ketoenamides **1** (Scheme 1).[13] We demonstrated that this process tolerated many functional groups and that enantiopure starting materials reacted without loss of enantiopurity.[14] In the present work, we have shown that this concept was also applicable to the cyclization of structurally related *ortho*-amidoacetophenones **3**, leading to 4-quinolones **4** which are benzannulated analogues of pyridinones **2**.



Scheme 1 TMSOTf/Et_3N-promoted cyclocondensations of precursors **1** and **3** leading to 4-pyridinones **2** and 4-quinolones **4**

In a preliminary experiment we found that the reaction conditions employed for the cyclocondensation of β-ketoenamides could be transferred to the cyclization of known *ortho*-amidoacetophenone **3a** to afford the expected product, 2-phenylquinolin-4-one (**4a**) in moderate yield. To test the generality of this cyclocondensation reaction a series of substituted *ortho*-amidoacetophenones **3a–l** was prepared (Table 1). The required starting materials were synthesized following established procedures by either condensation of substituted *ortho*-aminoacetophenones with acyl chlorides (method A) or directly with carboxylic acids using bromotripyrrolidin-1-ylphos-

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phonium hexafluorophosphate (PyBroP)[15] as the coupling reagent (method B). Alternatively, precursors **3** could be prepared by copper-catalyzed amidation of 2′-bromo-substituted acetophenone derivatives following a procedure developed by Buchwald and co-workers (method C).[12c] It should be mentioned that no attempts were made to optimize the syntheses of the starting materials; in some cases the yields could certainly be improved.

Table 1 Preparation of the Precursor *ortho*-Amidoacetophenones **3a–l**

3

Entry	R^1	R^3	R^2	Product	Yield (%)
1	H	H	Ph	**3a**	99[a]
2	H	H	2-pyridyl	**3b**	90[b]
3	H	H	2-thienyl	**3c**	68[b]
4	H	H	pentyl	**3d**	99[a]
5	H	H	*i*-Bu	**3e**	99[a]
6	H	H	(*R*)-CH(Et)Ph	**3f**	86[b]
7	5-F	H	pentyl	**3g**	33[c]
8	4-CO_2Me	H	pentyl	**3h**	79[a]
9	4-OMe	Me	Ph	**3i**	58[c]
10	H	H	(*R*)-CH(OTBS)Ph	**3j**	82[a]
11	H	H	$(CH_2)_4$COMe	**3k**	47[b]
12	H	H	CH=CHMe	**3l**[d]	99[b]

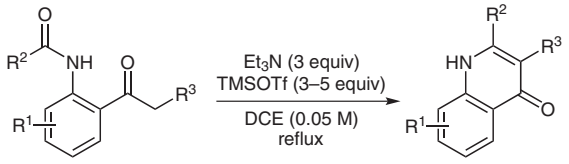
[a] Method A: X = NH_2, Y = Cl, py (1.1 equiv), CH_2Cl_2 (0.1–0.2 M), r.t., 16 h.
[b] Method B: X = NH_2, Y = OH, PyBroP (1.1 equiv), Et_3N (1.1 equiv), CH_2Cl_2 (0.1 M), r.t., 16 h.
[c] Method C: X = Br, Y = NH_2, CuI (0.1 equiv), $MeHNCH_2CH_2NHMe$ (0.1 equiv), K_2CO_3 (2.0 equiv), toluene (0.3 M), 100 °C, 16 h.
[d] Product **3l** was formed as a 2:3 mixture of the conjugated and non-conjugated alkenes.

Before examining the scope of the cyclocondensation reaction in detail we sought to further optimize the process. Employing *ortho*-amidoacetophenone **3a** as a model substrate, we varied the solvent, the reaction temperature and equivalents of the reagents (Et_3N and TMSOTf). However, none of these changes enhanced the efficacy of the cyclization. In all the experiments, full conversion of the starting material was achieved within 48 hours but the yield of 4-quinolone **4a** was low (~40%). A major problem was the separation of the 4-quinolone from the salt triethylammonium trifluoromethanesulfonate ([Et_3NH][OTf]) which was formed during the reaction. By a simple modification of the work-up procedure, which involved a basic wash of the organic layer with sodium hydroxide solution (2 M), the undesired salt by-product could be removed completely resulting in a simplified purification and a significantly improved yield of 4-quinolone **4a** (83%).

The results shown in Table 2 revealed that the scope of the cyclization was very broad. Aromatic and heteroaromatic substituents such as phenyl and pyridyl were tolerated leading to the 2-aryl- and 2-heteroaryl-substituted 4-quinolone derivatives **4a** and **4b** in good yields (Table 2, entries 1 and 2). In contrast, the 2-thienyl-substituted 4-quinolone **4c** was obtained in a moderate 39% yield (Table 2, entry 3). It can be speculated that this significant drop in efficiency might be attributed to the electron-donating properties of the thienyl group which decreases the electrophilicity of the amide group. When alkyl *ortho*-amidoacetophenone **3d** was subjected to the cyclization conditions the corresponding 2-alkyl-substituted 4-quinolone **4d** was obtained in excellent yield (Table 2, entry 4). In contrast to the classic conditions, where a 1:2 mixture of 2- and 4-quinolones was formed in a total yield of 72%,[12c] the regioisomeric 2-quinolone arising from deprotonation at the amide α-carbon atom was not detected by ^{1}H NMR analysis of the crude product. Encouraged by this finding we decided to further investigate the scope of the cyclization reaction with respect to 2-alkyl-substituted 4-quinolones, which were not accessible selectively via the classical Camps cyclization. Substrates with β-branched alkyl groups were tolerated as was demonstrated by the valeric acid derived 4-quinolone **4e** formed in moderate yield (Table 2, entry 5). Surprisingly, we found that *ortho*-amidoacetophenone **3f** with a branched alkyl group at the amide α-carbon atom did not undergo cyclization into the desired 4-quinolone **4f** (Table 2, entry 6). In several experiments no significant conversion could be detected even after several days. The starting material was recovered quantitatively and the optical purity of **3f** was found to be unchanged (98% ee as determined by HPLC on a chiral stationary phase). This rules out a competing deprotonation at the amide α-carbon atom as a reason for the failure of this cyclization. As yet, we cannot provide a definitive explanation and can only speculate that the cyclization process is rather sensitive toward the steric demand of the amide substituent.

To test the influence of different aryl substituents on the amidoacetophenone on the cyclization efficiency, compounds **3g–i** were prepared and converted into the respective 4-quinolones **4g–i**. The electronic properties of the ring substituents did not have a significant influence on the reactivity and the expected quinolone derivatives were obtained in yields ranging from 44–63% (Table 2, entries 7–9). Nevertheless, entries 8 and 9 (Table 2) demonstrated that methoxycarbonyl and methoxy groups were tolerated under the reaction conditions. Entry 9 (Table 2) shows that not only acetophenone derivatives are suitable reaction partners but propiophenone derivatives also provide the respective 3-alkyl-substituted quinolone **4i**.

Table 2 Cyclocondensation of *ortho*-Amidoacetophenones **3** into 4-Quinolones **4**



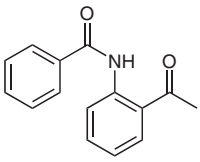
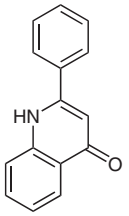
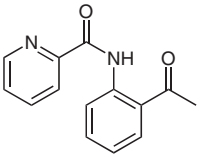
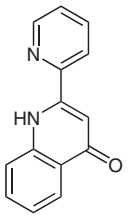
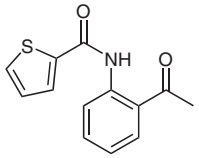
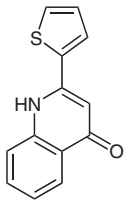
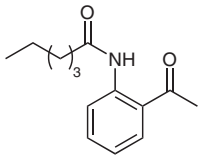
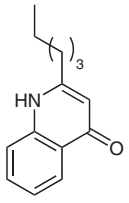
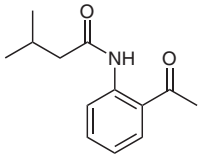
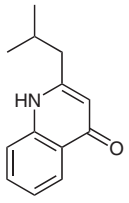
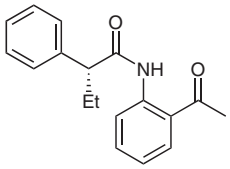
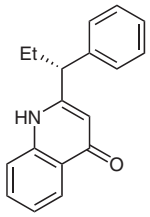
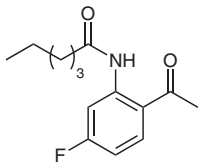
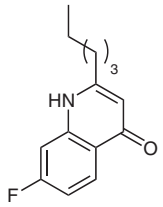
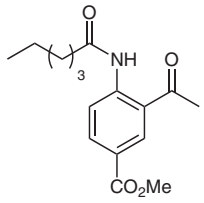
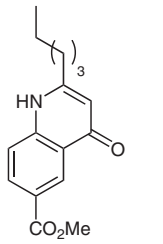
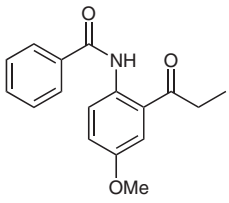
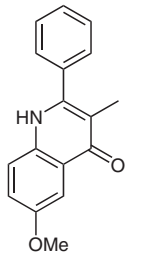
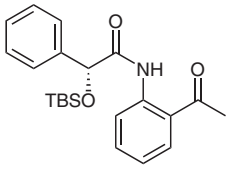
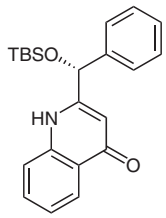
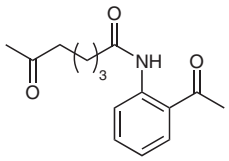
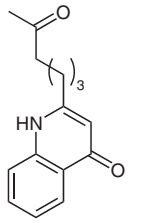
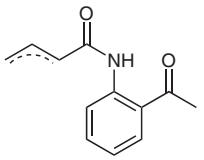
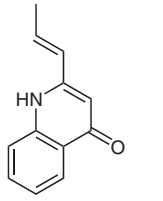
Entry	*ortho*-Amidoacetophenone	4-Quinolone	Yield (%)
1	 **3a**	 **4a**[a]	83
2	 **3b**	 **4b**[a]	85
3	 **3c**	 **4c**[a]	39
4	 **3d**	 **4d**[a]	92
5	 **3e**	 **4e**	31 (48%)[b]
6	 **3f** (ee = 98%)[c]	 **4f**	0

Table 2 Cyclocondensation of *ortho*-Amidoacetophenones **3** into 4-Quinolones **4** (continued)

7	 **3g**	 **4g**	63 (93%)[b]
8	 **3h**	 **4h**	51
9	 **3i**	 **4i**	44 (62%)[b]
10	 **3j**	 **4j** (ee >95%)[d]	81
11	 **3k**	 **4k**	26
12	 **3l**[e]	 **4l**	33

[a] Compounds **4a**–**d** are known.[12c]
[b] Yield in parentheses based on recovered starting material.
[c] Determined by chiral HPLC analysis.
[d] Determined by derivatization with chiral carboxylic acids.
[e] Compound **3l** was used as a 2:3 mixture of the conjugated and unconjugated alkenes.

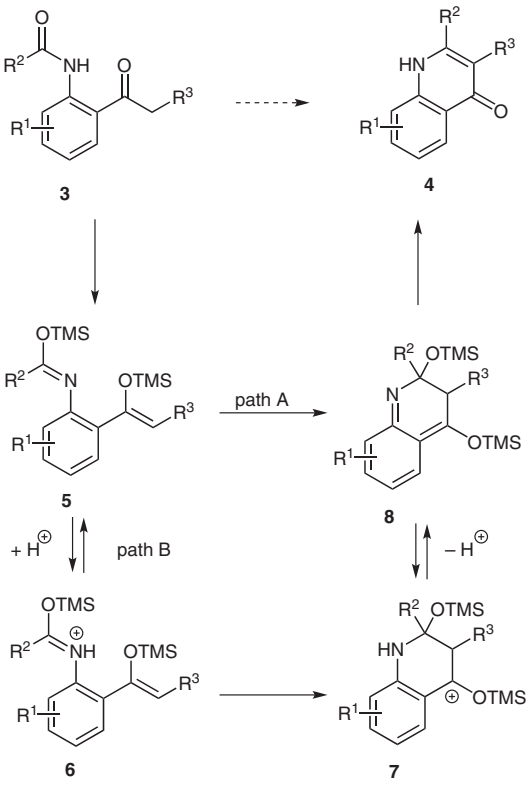
The need for strong bases in the classical Camps cyclization does not allow the use of enantiopure starting materials that might undergo racemization (e.g., *ortho*-amidoacetophenones with stereogenic centers at the amide α-carbon atom). In order to examine the racemization-free synthesis of chiral quinolone derivatives starting from enantiopure substrates using our cyclization conditions, mandelic acid derived *ortho*-amidoacetophenone derivative **3j** was prepared and cyclized to provide the quinolone **4j** (Table 2, entry 10). The desired product was obtained in high yield and excellent optical purity (ee >95%, see Supporting Information). In analogy to the respective pyridine derivatives, enantiopure hydroxymethyl-substituted quinolones obtained from **4j** might be efficient catalysts or ligands for asymmetric transformations.[16]

The striking differences in the reactivity of **3f** and **3j** might be caused by different conformations of the amido side chain. An internal hydrogen bond between the N–H proton and the *tert*-butyldimethylsilyloxy (OTBS) group in **3j** hampers free rotation around the C1–C2 bond and fixes the molecule in an eclipsed conformation. This would make the C1 atom more accessible to nucleophilic attack and therefore facilitate the reaction. However, as the mechanism of the cyclization is not yet fully understood and various pathways are conceivable (see below) we cannot provide a precise explanation for the reaction outcome and other factors cannot be excluded.

Other functional groups including ketones and olefins were also tolerated in this cyclization reaction, however the yields of the respective 4-quinolones **4k** and **4l** were rather low (Table 2, entries 11 and 12). Presumably, with substrates **3k** and **3l**, side reactions occur under the Lewis acidic reaction conditions limiting the efficiency of the cyclization process.

Typically the reaction times for the cyclocondensation reactions ranged between 16–48 hours, however, in some cases, only partial conversion was detected even after three days. In these examples the reactions were stopped and the yields based on recovered starting material are given in parentheses (Table 2). Attempts were made to shorten the reaction times by increasing the amount of TMSOTf or varying the equivalents of base, but without success. Also higher boiling chlorinated solvents were investigated (e.g. 1,2-dichlorobenzene) and the cyclization was conducted in a microwave oven. Unfortunately, these modifications did not lead to significant improvements in the reaction rates.

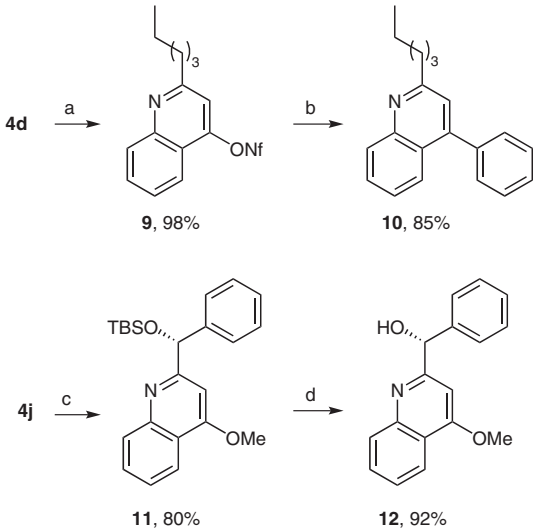
A proposed mechanism for the cyclization is depicted in Scheme 2. In analogy to the proposed mechanism for the cyclization of β-ketoenamides, the disilylated species **5** is likely to be formed in the first step. From here two different pathways are possible (A and B). Intermediate **5** can either cyclize directly via a formal 6π-electrocyclization to give the dihydroquinoline intermediate **8**, or after N-protonation leading to **6**, a similar cyclization, which can also be regarded as an intramolecular Mannich-type reaction, would afford the cationic intermediate **7**. Subsequent deprotonation would then lead to **8** which after elimination of trimethylsilanol followed by proto-desilylation of the 4-hydroxy group gives the 4-quinolone derivative **4**.



Scheme 2 Proposed mechanism for the TMSOTf-induced cyclization of *ortho*-amidoacetophenones **3** to 4-quinolones **4**

To further increase the flexibility of the substitution pattern of the prepared quinolone derivatives, compounds **4d** and **4j** were subjected to our previously reported protocols for O-selective nonaflation [using perfluoro-1-butanesulfonyl fluoride (NfF)] or O-selective methylation (Scheme 3). We have already reported various applications for pyrid-4-yl nonaflates in palladium-catalyzed cross-couplings such as Suzuki, Sonogashira and Heck reactions.[13e,17] Under standard conditions nonaflate **9** furnished Suzuki coupling product **10** in very good yield. After selective O-methylation, product **11** was converted into the enantiopure secondary alcohol **12** in excellent yield.

In summary, we have shown that Et_3N in combination with TMSOTf can successfully replace metal alkoxides in the base-promoted cyclocondensation of *ortho*-amidoacetophenones leading to 4-quinolone derivatives. The described procedure is fairly tolerant of many functional groups and allows for the selective preparation of 2-alkyl-substituted 4-quinolones. Moreover, enantiomerically pure starting materials can be employed to afford, without racemization, enantiopure 4-quinolone derivatives with chiral side chains. In one example it was demonstrated that a quinolin-4-yl nonaflate was an excellent partner in



Scheme 3 O-Selective nonaflation and O-selective methylation of 4-quinolones **4d** and **4j**, and subsequent cross-coupling to give **10** or deprotection to afford **12**. *Reagents and conditions*: (a) NaH, NfF, pyridine–THF–DMF (1:10:1), r.t., 3.5 h; (b) $PhB(OH)_2$, $Pd(PPh_3)_4$, K_2CO_3, DMF, 90 °C, 6 h; (c) MeI, K_2CO_3, DMF, r.t., 24 h; (d) TBAF (1 M in THF), THF, r.t., 2 h.

a palladium-catalyzed cross-coupling reaction. Hence, libraries of quinoline derivatives should be easily accessible via this method.

Reactions were generally performed under argon in flame-dried flasks. Solvents and reagents were added via syringe. Solvents were purified using the MB SPS-800-dry solvent system. Et_3N was distilled from CaH_2 and stored over KOH under an atmosphere of argon. Pyridine was stored over KOH under an atmosphere of argon. Other reagents were purchased and used as received unless otherwise stated. The products were purified by flash chromatography on SiO_2 (230–400 mesh, Merck or Fluka). Unless otherwise stated, yields refer to analytically pure samples. Melting points were measured with a Reichert apparatus (Thermovar) and are uncorrected. Optical rotations were determined with a Perkin–Elmer 241 polarimeter at the temperatures given. IR spectra were obtained using a Nicolet 5 SXC FT-IR spectrometer or with a Nexus FT-IR spectrometer fitted with a Nicolet Smart DuraSample IR ATR. NMR spectra were recorded using Bruker AC 250, AC 500, AV-III 700 and JOEL ECX 400, Eclipse 500 instruments. Chemical shifts are reported relative to residual solvent signals or TMS [^{1}H: δ = 0.00 (TMS), δ = 7.26 ($CDCl_3$); ^{13}C: δ = 77.0 ($CDCl_3$); ^{19}F: δ = –162 (C_6F_6)]. Integrals are in accordance with assignments and coupling constants (*J*) are given in Hz. All ^{13}C NMR spectra are proton-decoupled. For detailed signal assignments 2D spectra were recorded (COSY, HMQC, HMBC). The ^{13}C NMR signals of the $CF_3(CF_2)_3$ group are not given as unambiguous assignments were not possible due to strong splitting through coupling with the ^{19}F nuclei. HRMS analyses were performed with an Agilent 6210 ESI–TOF instrument. Elemental analyses were carried out with a CHN-Analyzer 2400 (Perkin–Elmer), a Vario EL or a Vario EL III.

2-Phenylquinolin-4(1*H*)-one (4a); Typical Procedure

To a soln of *ortho*-amidoacetophenone **3a** (200 mg, 0.86 mmol) in DCE (8.5 mL) was added Et_3N (0.36 mL, 2.6 mmol) followed by TMSOTf (1.12 g, 5.14 mmol). The resulting soln was heated to 95 °C under argon for 3 d. After cooling to r.t., the remaining TMSOTf was quenched by slow addition of MeOH (5 mL). All volatile components were then removed under reduced pressure before the residual material was partitioned between EtOAc (50 mL) and NaOH (2 M, 30 mL). The organic layer was separated and washed with NaOH (2 M, 2 × 30 mL) to completely remove $[HNEt_3][OTf]$ (the extraction process can be monitored by TLC, R_f $[HNEt_3][OTf]$ ~0.3 (5% MeOH in CH_2Cl_2, $KMnO_4$ stain). The aq layer was back-extracted with EtOAc (2 × 30 mL), and the combined organic layer dried over Na_2SO_4, filtered and the solvent removed under reduced pressure. The crude residue was purified by flash column chromatography (SiO_2, 5% MeOH in CH_2Cl_2) to afford **4a**. The analytical data were in agreement with those reported in the literature.[12c]

Yield: 158 mg (83%); yellow solid; mp >230 °C.

^{1}H NMR (400 MHz, $CDCl_3$–CD_3OD, 1:1): δ = 6.54 (s, 1 H, 3-H), 7.31–7.39 (m, 1 H, ArH), 7.47–7.55 (m, 3 H, ArH), 7.59–7.66 (m, 1 H, ArH), 7.71 (m_c, 3 H, ArH), 8.27 (m_c, 1 H, ArH).

^{13}C NMR (101 MHz, $CDCl_3$–CD_3OD): δ = 107.5 (d, C-3), 118.2 (s, Ar), 123.8, 124.4, 124.8, 128.7, 130.0, 132.0, 133.9 (7 d, Ar), 140.3, 151.4, 151.6 (3 s, Ar and C-2), 179.1 (s, C-4).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{15}H_{12}NO$: 222.0919; found: 222.0907.

Anal. Calcd for $C_{15}H_{11}NO$: C, 81.43; H, 5.01; N, 6.33. Found: C, 81.41; H, 5.19; N, 6.31.

2-Pentylquinolin-4-yl Nonaflate (9)

To a soln of **4d** (100 mg, 0.46 mmol) in a mixture of THF (2 mL), py (0.2 mL) and DMF (0.2 mL) was added NaH (60% wt in mineral oil, 56 mg, 1.39 mmol). The resulting soln was stirred at r.t. for 30 min and then treated with NfF (0.25 mL, 1.4 mmol). Stirring at r.t. was continued for 3.5 h before the reaction was quenched by addition of H_2O. The organic layer was separated and the aq layer extracted with EtOAc (3 × 15 mL). The combined organic layers were dried over Na_2SO_4, filtered and the solvent removed under reduced pressure. The crude residue was purified by flash column chromatography (SiO_2, hexane–EtOAc, 9:1) to afford the title compound **9**.

Yield: 225 mg (98%); colorless oil.

IR (ATR): 2980–2860 (=C–H), 1655–1595 (C=C) cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.91 (t, *J* = 7.0 Hz, 3 H, 5′-H), 1.31–1.49 (m, 4 H, 4′-H, 3′-H), 1.81–1.87 (m, 2 H, 2′-H), 3.02 (t, *J* = 7.9 Hz, 2 H, 1′-H), 7.31 (s, 1 H, 3-H), 7.60–7.68 (m, 1 H, ArH), 7.79–7.82 (m, 1 H, ArH), 8.03 (m_c, 1 H, ArH), 8.12 (m_c, 1 H, ArH).

^{13}C NMR (101 MHz, $CDCl_3$): δ = 13.9 (q, C-5′), 22.5 (t, C-4′), 29.3 (t, C-3′), 31.5 (t, C-2′), 39.3 (t, C-1′), 111.8 (d, C-3), 119.7 (s, Ar), 120.5, 127.4, 129.0, 131.0 (4 d, Ar), 149.9 (s, Ar), 153.2 (s, C-2), 164.1 (s, C-4).

^{19}F NMR (376 MHz, $CDCl_3$): δ = –127.7, –122.6, –110.6, –82.5.

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{18}H_{17}F_9NO_3S$: 498.0780; found: 498.0807.

(*R*)-2-[(*tert*-Butyldimethylsiloxy)(phenyl)methyl]-4-methoxyquinoline (11)

To a soln of quinolone **4j** (248 mg, 0.68 mmol) in DMF (3.5 mL) was added K_2CO_3 (282 mg, 2.04 mmol) and MeI (115 mg, 0.81 mmol). The resulting suspension was stirred for 24 h at r.t. before H_2O (15 mL) and EtOAc (15 mL) were added. The organic layer was separated and the aq layer extracted with EtOAc (2 × 20 mL). The combined organic layers were washed with brine (1 × 20 mL), dried over Na_2SO_4, filtered and the solvent removed under reduced pressure. The crude residue was purified by flash column chromatography (SiO_2, hexane–EtOAc, 9:1) to afford quinoline **11**.

Yield: 206 mg (80%); colorless oil; $[\alpha]_D^{22}$ +171 (*c* 0.5, $CHCl_3$).

IR (ATR): 3065–2850 (=C–H, C–H), 1620–1510 (C=C) cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = –0.01 (s, 3 H, $SiCH_3$), 0.10 (s, 3 H, $SiCH_3$), 0.97 [s, 9 H, $C(CH_3)_3$], 3.99 (s, 3 H, OCH_3), 6.03 (s, 1 H, C*H*Ph), 7.03 (s, 1 H, 3-H), 7.18–7.22 (m, 1 H, ArH), 7.27–7.31 (m, 2 H, ArH), 7.45 (m_c, 1 H, ArH), 7.55–7.60 (m, 2 H, ArH), 7.66 (m_c, 1 H, ArH), 8.01 (m_c, 1 H, ArH), 8.13 (m_c, 1 H, ArH).

^{13}C NMR (101 MHz, $CDCl_3$): δ = –4.9 (q, $SiCH_3$), –4.8 (q, $SiCH_3$), 18.3 [s, $C(CH_3)_3$], 25.8 [q, $C(CH_3)_3$], 55.5 (q, OCH_3), 78.3 (d, *C*HPh), 97.0 (d, C-3), 120.8, 121.6, 125.2, 125.8, 127.4, 128.1, 128.6 (7 d, Ar), 129.6 (s, Ar), 143.4 (s, Ar), 148.1 (s, Ar), 162.7 (s, C-2), 165.7 (s, C-4).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{23}H_{30}NO_2Si$: 380.2040; found: 380.2048.

Anal. Calcd for $C_{23}H_{29}NO_2Si$: C, 72.78; H, 7.70; N, 3.69. Found: C, 72.89; H, 7.93; N, 3.74.

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synthesis.

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