

8-Fluoro-6-(methoxymethoxy)quinoline: Synthesis and Regioselective Functionalization *via* Reaction with Organolithium Compounds

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8-Fluoro-6-(methoxymethoxy)quinoline (**1**) was synthesized, and the reactivity of **1** against organolithium compounds was studied under different reaction conditions. With BuLi, directed *ortho*-metalation (DoM) was accompanied by 1,2-addition to the C=N bond. 1,2-Addition was exclusively observed with *t*-BuLi. Selective *ortho*-metalation was achieved with MeLi (*Table*). Based on these findings, a short and high-yielding synthesis of the highly functionalized quinolines **12a–c** was developed.

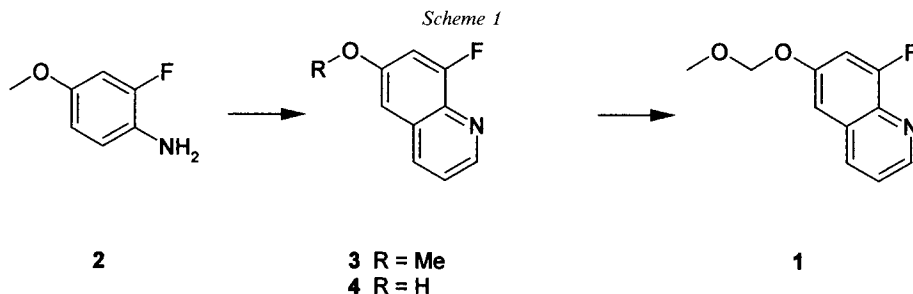
Introduction. – *Directed ortho-Metalation* (DoM) by organolithium compounds has become a widely used method for the regioselective functionalization of aromatic and heteroaromatic compounds [1][2]. However, in π -deficient aza-aromatics DoM is accompanied by 1,2-addition of RLi to the C,N multiple bond [3]. Thus, in the case of monofluorinated quinolines, 1,2-adducts were predominantly obtained upon reaction with BuLi in the presence of *N,N,N',N'*-tetramethylethylenediamine (TMEDA). However, lithiation *ortho* to the F-atom (DoM) was possible by reaction with lithium diisopropylamide (LDA) in the presence of hexamethylphosphoric triamide (HMPA) [4]. Interestingly, lithiation of 8-fluoroquinoline with LDA has not been reported and, as we have found, failed.

During our work on fluorinated quinolines, we were interested in the functionalization of 8-fluoro-6-(methoxymethoxy)quinoline (**1**). We assumed that lithiation of **1** *via* DoM should be facilitated by the presence of the additional ether group, as well as by the 1,3-arrangement of the directing groups [2]. We now report on the synthesis and selective functionalization of **1** (at C(2) and C(7)) *via* DoM and 1,2-addition, which strongly depends on the lithiating agent and the reaction conditions.

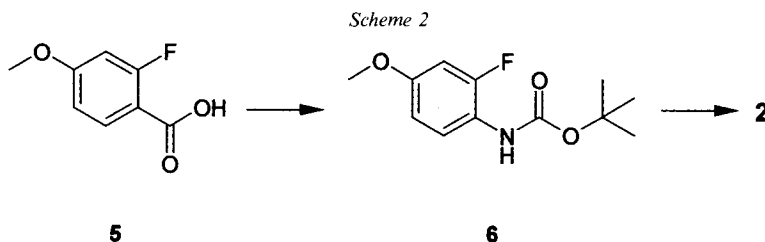
Results and Discussion. – 1. *Synthesis of 1.* Starting from 2-fluoro-4-methoxyaniline (**2**), the quinoline nucleus was elaborated by a modified *Skraup* reaction leading to 8-fluoro-6-(methoxymethoxy)quinoline (**3**) [5]. Ether cleavage with 48% aq. HBr yielded 8-fluoroquinolin-6-ol (**4**), which was finally transformed into **1** by treatment with NaH, followed by MeOCH₂Cl [6][7] (*Scheme 1*).

The known starting material **2**, which could only be obtained in low yield (> 20%) following the procedure in [8][9], was more conveniently prepared from 2-fluoro-4-methoxybenzoic acid (**5**) [10] by a modified *Curtius* degradation using diphenylphosphoryl azide (DPPA) in the presence of *t*-BuOH [11] which afforded compound **6**. This was

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easily transformed into **2** by acid-catalyzed cleavage of the Boc protecting group. Thus, starting from **5**, the aniline **2** was obtained in an excellent overall yield of 78% (Scheme 2).

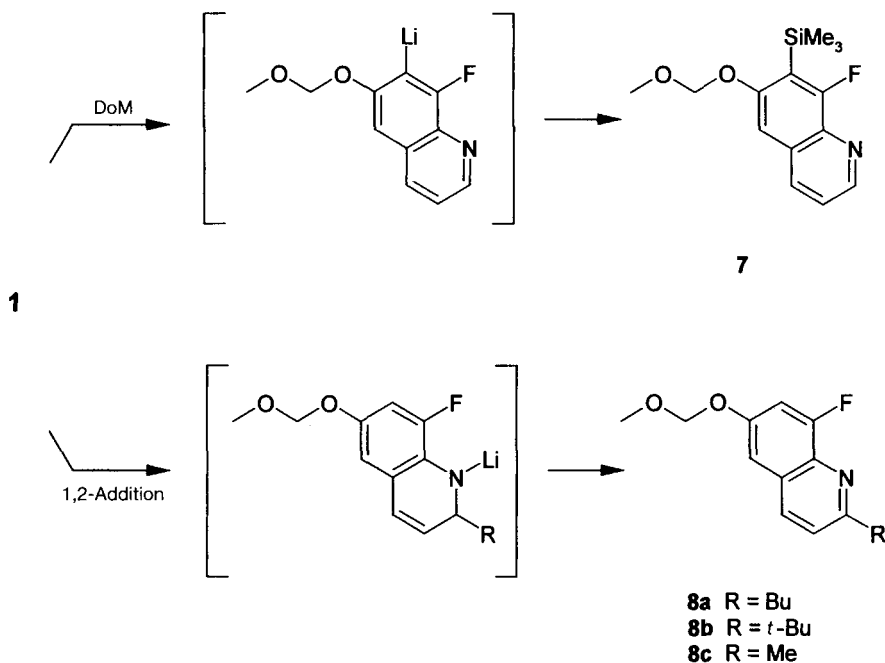


2. Reaction of 1 with RLi (DoM vs. 1,2-Addition). To study the competition between DoM and 1,2-addition, the lithiated intermediates resulting from reaction of **1** with different organolithium compounds (BuLi, *t*-BuLi, MeLi) under different reaction conditions were quenched with trimethylsilyl (trifluoromethyl)sulfonate to unambiguously determine the site and extent of lithiation, *via* the formation of the product **7**. By oxidation of the crude reaction mixture with activated MnO₂, any dihydroquinoline formed by 1,2-addition was transformed into the quinolines **8a** and **8b**. This procedure allowed us to determine the ratio of DoM vs. 1,2-addition on the basis of isolated yields of **7**, **8a**, and **8b** after chromatographic separation (Scheme 3).

When **1** was reacted with BuLi in THF at -78° , product **8a**, resulting from 1,2-addition, was predominantly formed. When the same reaction was performed in the presence of TMEDA, DoM dominated over 1,2-addition, and **7** was isolated as the major product. Reaction of **1** with *t*-BuLi in THF at -78° afforded compound **8b**, resulting from 1,2-addition, as the only isolable compound, in moderate yield. Finally, the reaction of **1** with MeLi in THF at -78° led to the exclusive formation of product **7**, resulting from DoM. However, for complete lithiation, the reaction time had to be prolonged (Table).

3. Reaction of Li-1 with Aldehyde 9, Followed by 1,2-Addition of RLi. Once we had optimized the reaction conditions for the lithiation of **1** (see Chapt. 2), we successfully developed a synthetic strategy which allowed the functionalization of **1** at C(7) (*via* DoM) as well as at C(2) (*via* 1,2-addition) in a one-pot procedure. Thus, **Li-1**, generated from **1**

Scheme 3

Table. Reactions of **1** with RLi

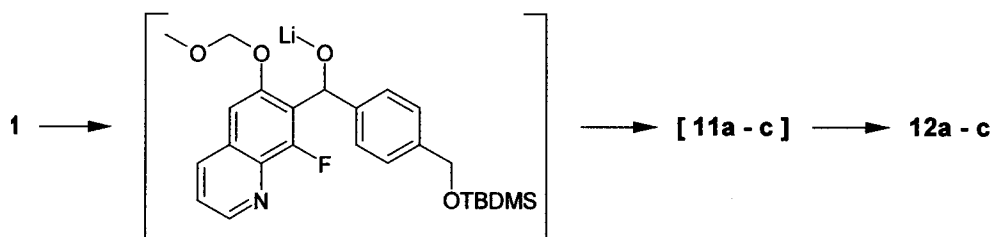
Entry	RLi	Solvent	Time [h]	Products (yield [%])
1	BuLi	THF	2	8a (47.4) 7 (18.5)
2	BuLi	THF/TMEDA	2	8a (12.2) 7 (60.1)
3	<i>t</i> -BuLi	THF	2	8b (59.0) 7 (not found)
4	MeLi	THF	6	8c (not found) 7 (82.6)

and MeLi by DoM, was reacted with 4-[[*tert*-butyl]dimethylsilyloxy]methyl}-benzaldehyde (**9**) to afford the intermediate **10**. Treatment of the solution of **10** with organolithium compounds (BuLi, *t*-BuLi, MeLi) provided the dihydroquinolines **11a–c** via subsequent 1,2-addition in a clean reaction, after hydrolytic workup. Compounds **11a–c** were directly oxidized with activated MnO₂ to afford the highly functionalized quinolines **12a–c** in good-to-excellent yields (Scheme 4). It is noteworthy that the reaction of **10** with MeLi required higher temperatures (–15°) than the corresponding reactions with either BuLi or *t*-BuLi (–78°).

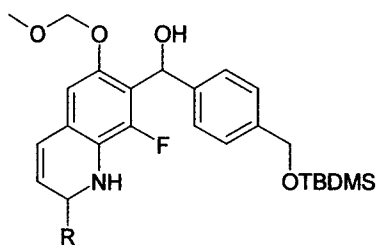
Aldehyde **9** was prepared from the known bromo compound **13** by halogen/Li exchange with BuLi and reaction of the resulting carbanion with DMF [12] (Scheme 5).

We gratefully acknowledge the help of our colleagues Dr. W. Arnold (NMR), Mr. A. Bubendorf (IR), Mr. W. Meister (MS), and Mrs. S. Ulsemer (excellent technical assistance). We also thank Profs. Sir J. Baldwin, F. Diederich, and A. Vasella for helpful discussions.

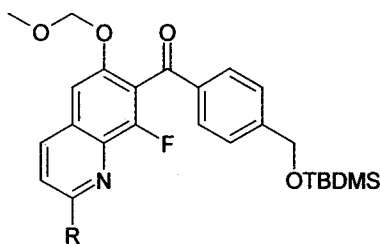
Scheme 4



10



11a - c

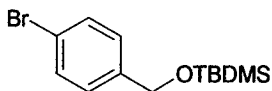


12a - c

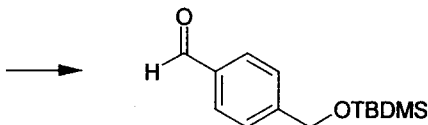
a R = Bu
b R = *t*-Bu
c R = Me

TBDMS = (*t*-Bu)Me₂Si

Scheme 5



13



9

Experimental Part

General. THF: *Fluka, puriss.*, distilled from Na/benzophenone. TMEDA: *Fluka, puriss.*, distilled from CaH. Organolithium reagents: BuLi (~ 1.6M in hexane, *Fluka*), *t*-BuLi (~ 1.5M in pentane, *Fluka*), and MeLi (~ 1.6M in Et₂O, *Fluka*). Reactions with organolithium compounds were performed under Ar in dried glassware. Reactions were monitored by TLC: silica gel 60 *F*₂₅₄ (precoated plates, *Merck*). Column chromatography (CC): silica gel 60 (230–400 mesh ASTM, *Merck*). M.p.: *Büchi 530*, uncorrected. ¹H-NMR: 250 MHz, δ in ppm against TMS as internal standard.

Abbreviations. DIPEA: *N,N*-Diisopropylethylamine. DPPA: Diphenylphosphoryl azide. TMEDA: *N,N,N',N'*-Tetramethylethylenediamine.

tert-Butyl N-(2-Fluoro-4-methoxyphenyl)carbamate (6). A soln. of 2-fluoro-4-methoxybenzoic acid (**5**) [10] (48.95 g; 0.50 mol), DIPEA (61.6 ml; 0.60 mol), and DPPA (99.08 g; 0.60 mol) in toluene (300 ml) and *t*-BuOH (300 ml) was gently refluxed for 16 h. The solvent was removed *in vacuo*, the residue diluted with H₂O (500 ml) and extracted with cyclohexane/AcOEt 3:1 (3 \times 250 ml). The combined org. layers were washed with H₂O (250 ml), sat. NaCl soln. (250 ml), and dried (MgSO₄). The crude product was purified by FC on silica gel

(cyclohexane/AcOEt 3:1). 70.05 g (96.8%) of **6**. Colorless oil. IR (neat): 3460, 3340, 1726, 1598, 1527, 1492, 1430, 1395, 1368, 1306, 1249, 1209, 1159, 1119, 1105, 1024, 950, 836. ¹H-NMR (CDCl₃): 1.51 (s, 9 H); 3.76 (s, 3 H); 6.45 (br. s, 1 H); 6.65 (m, 1 H + 1 H); 7.87 (~ dd, $J_1 \approx J_2 \approx 9$, 1 H). EI-MS: 241 (M^+), 185 ($[M - C_4H_8]^+$), 141, 126, 57 (100).

2-Fluoro-4-methoxyaniline (2). A soln. of **6** (60.31 g; 0.25 mol) in MeOH (500 ml) was slowly saturated with gas. HCl, and stirring was continued for 1 additional h. The solvent was completely removed *in vacuo*, the residue dissolved in a minimal amount of H₂O, and pH adjusted to 8 by careful addition of 1N NaOH. After extraction with Et₂O (3 × 250 ml), the combined org. layers were dried (K₂CO₃), and the crude product was purified by sublimation (40°, 0.1 mbar): 28.44 g (80.6%) of **2**. Colorless crystals. M.p. 48°. IR (nujol): 3395, 1639, 1591, 1512, 1468, 1450, 1379, 1329, 1312, 1267, 1236, 1192, 1146, 1071, 1029, 940, 830. ¹H-NMR (CDCl₃): 3.38 (br. s, 2 H); 3.73 (s, 3 H); 6.54 (ddd, $J = 8.69, 2.73, 1.16$, 1 H); 6.62 (dd, $J = 12.46, 2.73$, 1 H); 6.72 (dd, $J = 9.99, 8.69$, 1 H). EI-MS: 141 (M^+), 126 (100, $[M - Me]^+$), 98.

8-Fluoro-6-methoxyquinoline (3). A well-stirred mixture of **2** (70.58 g, 0.50 mol), arsenic(V) oxide hydrate (97.46 g, 0.13 mol), boric acid (61.83 g, 1.00 mol), and anh. glycerol (230.26 g, 2.50 mol) was heated to 60° (internal temp.). Conc. H₂SO₄ (223.74 g, 2.28 mol) was added dropwise, the mixture heated to 120° (internal temp.) for 16 h and poured onto crushed ice (1000 g) after cooling to r.t. The pH was adjusted to 8 by careful addition of conc. NH₃, the aq. phase was saturated with NaCl and extracted with *t*-BuOMe (3 × 250 ml). The combined org. layers were washed with H₂O (250 ml), sat. NaCl soln. (250 ml), and dried (MgSO₄) in the presence of decolorizing charcoal (20 g). The residual oil was filtered through a short column of neutral alumina act. 3 (hexane/AcOEt 1:2). The product was finally purified by FC on silica gel (cyclohexane/AcOEt 7:3) and bulb-to-bulb distillation under high vacuum: 33.54 g (37.86%) of **3**. Colorless oil. B.p. 150° (0.1 mbar). IR (neat): 1633, 1595, 1580, 1502, 1472, 1452, 1427, 1378, 1339, 1264, 1198, 1156, 1136, 1088, 1054, 839, 784. ¹H-NMR (CDCl₃): 3.92 (s, 3 H); 6.88 (d, $J = 2.56$, 1 H); 7.10 (dd, $J = 11.69, 2.56$, 1 H); 7.41 (dd, $J = 8.38, 4.22$, 1 H); 8.06 (ddd, $J = 8.38, 1.51, 1.51$, 1 H); 8.80 (dd, $J = 4.22, 1.51$, 1 H). EI-MS: 177 (100, M^+), 147, 134, 107.

8-Fluoroquinolin-6-ol (4). A stirred soln. of **3** (17.2 g; 0.1 mol) in 48% aq. HBr (200 ml) was gently refluxed for 8 h. The reaction mixture was cooled to 0°, and pH was adjusted to 8 by careful addition of conc. NH₃. The precipitated crude product was filtered, washed with several small portions of ice cold H₂O and recrystallized from EtOH: 14.52 g (89.0%) of **4**. Colorless crystals. M.p. 240° (dec.). IR (KBr): 3455, 1634, 1595, 1519, 1468, 1457, 1405, 1376, 1345, 1267, 1189, 1161, 1132, 1083, 987, 852, 835, 773. ¹H-NMR (CDCl₃): 7.02 (d, $J = 2.48$, 1 H); 7.15 (dd, $J = 12.41, 2.50$, 1 H); 7.49 (dd, $J = 8.43, 4.12$, 1 H); 8.21 (ddd, $J = 8.43, 1.52, 1.52$, 1 H); 8.70 (dd, $J = 4.12, 1.52$, 1 H); 10.38 (br. s, 1 H). EI-MS: 163 (100, M^+), 135, 107.

8-Fluoro-6-(methoxymethoxy)quinoline (1). Oil-free NaH, prepared from 55% dispersion in oil (2.16 g; 50.0 mmol) by washing with hexane, was suspended in dry THF (50 ml). A soln. of **4** (7.34 g; 45.0 mmol) in dry DMF (35 ml) was added dropwise to the well-stirred suspension. Stirring was continued for 30 min before dropwise addition of a soln. of MeOCH₂Cl (4.35 g; 55.0 mmol) in dry THF (25 ml). During addition, the temp. was kept below 30° by intermittent cooling with ice/H₂O. After 1 h, half-sat. NaHCO₃ soln. (10 ml) was added, the mixture was concentrated *in vacuo*, the residue was diluted with H₂O (250 ml), and extracted with AcOEt (3 × 100 ml). The combined org. extracts were washed with sat. NaCl soln. (100 ml) and dried (MgSO₄). The crude product was purified by FC on silica gel (hexane/AcOEt 1:1) and crystallized from Et₂O/hexane at -78°: 8.85 g (94.9%) of **1**. Colorless crystals. M.p. 46°. IR (KBr): 1633, 1595, 1504, 1476, 1440, 1377, 1335, 1261, 1150, 1125, 1080, 1038, 1003, 940, 916, 867, 847, 777. ¹H-NMR (CDCl₃): 3.53 (s, 3 H); 5.29 (s, 2 H); 7.17 (br. s, 1 H); 7.19 (dd, $J = 11.9, 2.51$, 1 H); 7.42 (dd, $J = 8.38, 4.18$, 1 H); 8.07 (ddd, $J = 8.38, 1.58, 1.58$, 1 H); 8.83 (dd, $J = 4.18, 1.58$, 1 H). EI-MS: 207 (M^+), 134, 45 (100).

Reaction of 1 with RLi: General Procedure. A soln. of the appropriate organolithium compound (5.1 mmol) was slowly added *via* syringe to a soln. of **1** (1.04 g; 5.0 mmol) in dry THF (25 ml), or TMEDA (2.90 g; 25.0 mmol) and dry THF (25 ml) at -78°. The mixture was stirred for the time indicated in the *Table* before addition of TMS-triflate (0.9 ml; *ca.* 5.2 mmol). The temp. was allowed to rise to 0°, sat. NH₄Cl soln. (5 ml) was added and stirring continued for another 15 min at r.t. The org. layer was separated and concentrated *in vacuo*. The aq. layer was extracted with AcOEt (3 × 5 ml), all org. phases were combined, washed with sat. NaCl soln. (5 ml), dried (MgSO₄), and concentrated *in vacuo*. The residue was dissolved in CH₂Cl₂ (25 ml) and, after addition of activated MnO₂ (2.5 g), the mixture was stirred for 4 h. MnO₂ was removed by suction filtration and washed with several small portions of CH₂Cl₂. The filtrate was concentrated *in vacuo*, and the products formed were separated by CC on silica gel (hexane/*t*-BuOMe 8:2) (for product distribution, see the *Table*).

2-Butyl-8-fluoro-6-(methoxymethoxy)quinoline (8a). Colorless oil. IR (neat): 1643, 1603, 1570, 1499, 1430, 1379, 1344, 1258, 1155, 1134, 1077, 1011, 947, 922, 855. ¹H-NMR (CDCl₃): 0.96 (t, $J = 7.33$, 3 H); 1.43 (m, 2 H); 1.78 (m, 2 H); 2.98 (≈ dd, $J = 7.79, 7.79$, 2 H); 3.52 (s, 2 H); 5.27 (s, 2 H); 7.13 (s, 1 H); 7.16 (dd, $J = 12.7, 2.57$,

1 H); 7.31 (*d*, *J* = 8.53, 1 H); 7.97 (*dd*, *J* = 8.53, 1.51, 1 H). EI-MS: 263 (M^+), 234 ($[M - Et]^+$), 221 ($[M - C_3H_6]^+$), 45 (100).

8-Fluoro-6-(methoxymethoxy)-7-(trimethylsilyl)quinoline (7). Colorless oil. IR (neat): 1617, 1592, 1559, 1477, 1442, 1438, 1359, 1248, 1223, 1178, 1154, 1092, 1043, 958, 902, 845, 765. ¹H-NMR (CDCl₃): 0.45 (*m*, 9 H); 3.52 (*s*, 3 H); 5.30 (*s*, 2 H); 7.11 (*s*, 1 H); 7.39 (*dd*, *J* = 8.32, 4.19, 1 H); 8.03 (*ddd*, *J* = 8.32, 1.51, 1.51, 1 H); 8.80 (*dd*, *J* = 4.19, 1.51, 1 H). EI-MS: 279 (M^+), 45 (100).

2-(tert-Butyl)-8-fluoro-6-(methoxymethoxy)quinoline (8b). Colorless oil. IR (neat): 1634, 1602, 1570, 1497, 1464, 1343, 1258, 1156, 1127, 1080, 1012, 948, 922, 855. ¹H-NMR (CDCl₃): 1.46 (*s*, 9 H); 3.52 (*s*, 3 H); 5.27 (*s*, 2 H); 7.11 (*br. s*, 1 H); 7.14 (*dd*, *J* = 11.7, 2.56, 1 H); 7.53 (*d*, *J* = 8.77, 1 H); 7.97 (*dd*, *J* = 8.77, 1.59, 1 H). EI-MS: 263 (M^+), 248 ($[M - Me]^+$), 45 (100).

One-Pot 7,2-Functionalization of 1: General Procedure. MeLi (3.3 ml of 1.6M soln. in Et₂O; 5.2 mmol) was slowly added *via* syringe to a stirred soln. of **1** (1.04 g; 5.0 mmol) in dry THF (12 ml) at -78° . After 6 h, a soln. of **9** (1.30 g; 5.2 mmol) in dry THF (5 ml) was added *via* syringe, and the reaction mixture was stirred for 30 min at -78° before addition of a soln. of the appropriate organolithium compound (5.2 mmol). Stirring was continued for 15 min at -78° and for another 30 min at -15° (ice/MeOH). The mixture was quenched with sat. NH₄Cl soln. (25 ml), the org. layer was separated and concentrated *in vacuo*. The aq. layer was extracted with AcOEt (3 × 10 ml), all org. phases were combined, washed with sat. NaCl soln. (10 ml), dried (MgSO₄), and concentrated *in vacuo*. The residue was dissolved in CH₂Cl₂ (50 ml), and, after addition of activated MnO₂ (5.0 g), the mixture was stirred for 16 h at r.t. MnO₂ was removed by suction filtration and washed with several small portions of CH₂Cl₂. The filtrate was concentrated *in vacuo* and the crude product purified by CC on silica gel.

(4-{[(tert-Butyl)dimethylsilyloxy]methyl}phenyl)-[2-butyl-8-fluoro-6-(methoxymethoxy)quinolin-7-yl]-methanone (12a): CC (hexane/*t*-BuOMe 8:2). Yield: 1.97 g (77.0%). Colorless oil. IR (neat): 1677, 1633, 1606, 1496, 1461, 1413, 1377, 1344, 1319, 1264, 1225, 1155, 1113, 1095, 1059, 1017, 937, 843, 778. ¹H-NMR (CDCl₃): 0.10 (*s*, 6 H); 0.94 (*s*, 9 H); 0.96 (*t*, *J* = 7.32, 3 H); 1.43 (*m*, 2 H); 1.80 (*m*, 2 H); 2.99 (*≈ dd*, *J*₁ \sim *J*₂ \sim 7.79, 2 H); 3.43 (*s*, 3 H); 4.81 (*s*, 2 H); 5.19 (*s*, 2 H); 7.26 (*d*, *J* = 1.91, 1 H); 7.38 (*d*, *J* = 8.55, 1 H); 7.41 (*d*, *J* = 8.27, 2 H); 7.88 (*d*, *J* = 8.27, 2 H); 8.02 (*dd*, *J* = 8.55, 1.95, 1 H). EI-MS: 511 (M^+), 469 ($[M - C_3H_6]^+$), 454 ($[M - C_4H_9]^+$), 209, 45 (100).

(4-{[(tert-Butyl)dimethylsilyloxy]methyl}phenyl)-[2-(tert-butyl)-8-fluoro-6-(methoxymethoxy)quinolin-7-yl]methanone (12b): CC (hexane/*t*-BuOMe 8:2). Yield: 2.08 g (81.3%). Colorless oil. IR (neat): 1677, 1632, 1605, 1498, 1456, 1364, 1343, 1316, 1256, 1210, 1155, 1115, 1093, 1062, 1004, 938, 843, 778. ¹H-NMR (CDCl₃): 0.10 (*s*, 6 H); 0.94 (*s*, 9 H); 1.46 (*s*, 9 H); 3.34 (*s*, 3 H); 4.80 (*s*, 2 H); 5.19 (*s*, 2 H); 7.24 (*d*, *J* = 1.91, 1 H); 7.41 (*d*, *J* = 8.07, 2 H); 7.60 (*d*, *J* = 8.87, 1 H); 7.88 (*d*, *J* = 8.07, 2 H); 8.03 (*dd*, *J* = 8.78, 1.91, 1 H). EI-MS: 511 (M^+), 469 ($[M - Me]^+$), 454 ($[M - C_4H_9]^+$), 209, 45 (100).

(4-{[(tert-Butyl)dimethylsilyloxy]methyl}phenyl)-[8-fluoro-6-(methoxymethoxy)-2-methylquinolin-7-yl]methanone (12c): CC (cyclohexane/*t*-BuOMe 6:4). Crystallization (*t*-BuOMe/hexane). Yield: 1.65 g (70.3%). Colorless crystals. *m.p.* 119°. IR (KBr): 1677, 1633, 1606, 1498, 1464, 1417, 1376, 1345, 1317, 1265, 1226, 1153, 1110, 1090, 1066, 1031, 940, 925, 913, 849, 838, 778. ¹H-NMR (CDCl₃): 0.10 (*s*, 6 H); 0.94 (*s*, 9 H); 2.77 (*s*, 3 H); 3.35 (*s*, 3 H); 4.81 (*s*, 2 H); 5.19 (*s*, 2 H); 7.27 (*d*, *J* = 1.95, 1 H); 7.37 (*d*, *J* = 8.52, 1 H); 7.41 (*d*, *J* = 8.26, 2 H); 7.88 (*d*, *J* = 8.26, 2 H); 8.01 (*dd*, *J* = 8.52, 1.95, 1 H). EI-MS: 469 (M^+), 412 ($[M - C_4H_9]^+$), 209, 45 (100).

4-{[(tert-Butyl)dimethylsilyloxy]methyl}benzaldehyde (9). To a soln. of **13** [12] (7.53 g; 25.0 mmol) in dry THF (125 ml), BuLi (17.0 ml of 1.6M soln. in hexane; 27.5 mmol) was added *via* syringe at -78° . After 2 h, dry DMF (9.6 ml; 125.0 mmol) was added *via* syringe, and stirring was continued for another 30 min. at -78° . The temp. was allowed to rise to 0°, and the mixture was quenched with sat. NH₄Cl soln. (50 ml). The org. layer was separated and concentrated *in vacuo*, the aq. layer was extracted with Et₂O (3 × 50 ml). All org. phases were combined, washed with sat. NaCl soln. (50 ml), and dried (MgSO₄). The crude product was purified by FC on silica gel (hexane/*t*-BuOMe 4:1): 5.78 g (92.3%) of **9**. Colorless oil. IR (neat): 2732, 1706, 1609, 1579, 1472, 1463, 1425, 1389, 1375, 1361, 1302, 1258, 1209, 1164, 1118, 1092, 1006, 847, 814, 778. ¹H-NMR (CDCl₃): 0.11 (*s*, 6 H); 0.96 (*s*, 9 H); 4.82 (*s*, 2 H); 7.49 (*d*, *J* = 8.08, 2 H); 7.85 (*d*, *J* = 8.08, 2 H); 10.00 (*s*, 1 H). EI-MS: 235 ($[M - Me]^+$), 193 (100, $[M - C_4H_9]^+$), 163, 135, 119, 91.

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