Synthesis of 4-alkoxy-8-hydroxyquinolines

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Contribution from the Empart group of Infotech Oulu

Experimental procedures of compounds **4b-e** and **5b-e**

pp S2-S5

IR, ¹H NMR, ¹³C NMR and HRMS for all compounds

pp S6-S58

hmbc NMR-spectra for 4-hydroxy-8-tosyloxyquinoline (3)

p S13

4-butoxy-8-tosyloxyquinoline (4b). The title compound was prepared by the procedure described in the journal (see Synthesis of **4a**) by first reacting **3** (1.00 g, 3.17 mmol) with NaH (116 mg, 4.83 mmol) and then n-butyl bromide (1.71 mL, 15.9 mmol) was added. The mixture was stirred for 24 h at room temperature. The reaction mixture was poured into water (200 mL) and allowed to stand at room temperature overnight. The precipitate was collected by filtration and washed with water. The final purification by flash chromatography (1:7 acetone/dichloromethane) afforded the title compound (924 mg, 78 %) as white crystals. Mp: 93 °C. IR (KBr): ν 2962, 2873 cm^{-1} . ^1H NMR (400 MHz, DMSO- d_6): δ 0.95 (3H, t, $J = 7.4$ Hz), 1.50 (2H, tq, $J = 7.4$ Hz), 1.81 (2H, tt, $J = 6.9$ Hz), 2.37 (3H, s), 4.21 (2H, t, $J = 6.3$ Hz), 7.03 (1H, d, $J = 5.2$ Hz), 7.40 (2H, d, $J = 8.2$ Hz), 7.46-7.54 (2H, m), 7.81 (2H, d, $J = 8.3$ Hz), 8.07 (1H, dd, $J = 8.2$ Hz), 8.63 (1H, d, $J = 5.2$ Hz). ^{13}C NMR (100 MHz, DMSO- d_6): δ 14.1, 19.2, 21.6, 30.7, 68.8, 102.8, 121.4, 122.7, 122.9, 125.6, 128.8 (2C), 130.3 (2C), 132.9, 142.3, 145.2, 145.8, 152.6, 161.1. HRMS: calcd for $\text{C}_{20}\text{H}_{22}\text{NO}_4\text{S}$ ($[\text{M}+\text{H}]^+$) 372.1270, found 372.1306.

4-octyloxy-8-tosyloxyquinoline (4c). The title compound was prepared by the procedure described in the journal (see Synthesis of **4a**) by first reacting **3** (1.00 g, 3.17 mmol) with NaH (116 mg, 4.83 mmol) and then 1-bromo-octane (2.76 mL, 15.8 mmol) was added. The mixture was stirred for 24 h at room temperature. The oily residue was extracted from water with chloroform and concentrated in vacuo. The final purification by flash chromatography (5 mL methanol/1.5 L 1:1 acetone/n-hexane) afforded the title compound (1.06 g, 78 %) as off-white crystals. Mp: 49 °C. IR (KBr): ν 3056, 2952, 2922, 2849 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 0.89 (3H, t, $J = 6.6$ Hz), 1.30-1.41 (8H, m), 1.53 (2H, tt, $J = 7.2$ Hz), 1.91 (2H, tt, $J = 7.0$ Hz), 2.39 (3H, s), 4.13 (2H, t, $J = 6.4$ Hz), 6.67 (1H, d, $J = 5.1$ Hz), 7.24 (2H, d, $J = 8.1$ Hz), 7.41 (1H, t, $J = 8.0$ Hz), 7.55 (1H, dd, $J = 7.6$ Hz), 7.86 (2H, d, $J = 8.3$ Hz), 8.12 (1H, dd, $J = 8.4$ Hz), 8.61 (1H, d, $J = 5.2$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 14.1, 21.6, 22.6, 26.1, 28.8, 29.2, 29.3, 31.8, 68.7, 101.3, 121.2, 122.7, 123.1, 124.6, 128.8 (2C), 129.4 (2C), 133.3, 142.6, 144.9, 145.3, 151.8, 161.3. HRMS: calcd for $\text{C}_{24}\text{H}_{30}\text{NO}_4\text{S}$ ($[\text{M}+\text{H}]^+$) 428.1896, found 428.1903.

4-isopropoxy-8-tosyloxyquinoline (4d). The title compound was prepared by the procedure described in the journal (see Synthesis of

4a) by first reacting **3** (1.00 g, 3.17 mmol) with NaH (116 mg, 4.83 mmol) and then isopropylbromide (1.79 mL, 19.1 mmol) was added to the mixture. The resulting mixture was stirred 48 h at room temperature. The reaction mixture was poured into water (300 mL) and allowed to stand at room temperature overnight. The precipitate was collected by filtration and washed with water. The final purification by flash chromatography (10 mL methanol /1.5 L 1:1 acetone/n-hexane) afforded the title compound (836 mg, 74 %) as white crystals. Mp: 137 °C. IR (KBr): ν 2982, 2928 cm^{-1} . ^1H NMR (200 MHz, DMSO- d_6): δ 1.38 (6H, d, $J = 6.0$ Hz), 2.37 (3H, s), 4.92 (1H, sept, $J = 6.0$ Hz), 7.06 (1H, d, $J = 5.4$ Hz), 7.38-7.54 (4H, m), 7.82 (2H, d, $J = 8.3$ Hz), 8.05 (1H, dd, $J = 2.1$ Hz), 8.62 (1H, d, $J = 5.2$ Hz). ^{13}C NMR (50 MHz, DMSO- d_6): δ 21.3, 21.6, 71.2, 103.1, 121.3, 122.4, 123.1, 125.2, 128.5, 130.0, 132.7, 142.4, 144.9, 145.6, 152.3, 159.7. HRMS: calcd for $\text{C}_{19}\text{H}_{19}\text{NO}_4\text{NaS}$ ($[\text{M}+\text{Na}]^+$) 380.0932, found 380.0943.

4-benzyloxy-8-tosyloxyquinoline (4e). The title compound was prepared by the procedure described in the journal (see Synthesis of **4a**) by first reacting **3** (1.00 g, 3.17 mmol) with NaH (116 mg, 4.83 mmol) and then benzylbromide (0.4 mL, 3.36 mmol) was added to the resulting sodium salt. The mixture was stirred for 1 h at room temperature and then poured into water. The precipitate was collected from water by filtration and recrystallized from ethanol-chloroform mixture to afford the product (719 mg, 56 %) as off-white crystals. Mp: 188 °C. IR (KBr): ν 3064, 2932, 2882 cm^{-1} . ^1H NMR (400 MHz, DMSO- d_6 , 50 °C): δ 2.39 (3H, s), 5.40 (2H, s), 7.16 (1H, d, $J = 5.1$ Hz), 7.36-7.56 (9H, m), 7.83 (2H, d, $J = 8.4$ Hz), 8.13 (1H, dd, $J = 7.4$ Hz), 8.66 (1H, d, $J = 5.1$ Hz). ^{13}C NMR (100 MHz, DMSO- d_6 , 50 °C): δ 21.6, 70.7, 103.4, 121.4, 122.8, 123.0, 125.8, 128.2 (2C), 128.7, 128.8 (2C), 129.1 (2C), 130.2 (2C), 133.2, 136.4, 142.5, 145.3, 145.7, 152.4, 160.9. HRMS: calcd for $\text{C}_{23}\text{H}_{20}\text{NO}_4\text{S}$ ($[\text{M}+\text{H}]^+$) 406.1113, found 406.1114.

4-butoxy-8-hydroxyquinoline (5b). The title compound was prepared by the procedure described in the journal (see Synthesis of **5a**)

from **4b** (1.50 g, 4.04 mmol) and sodium hydroxide (1 M, 12.0 mL, 12.0 mmol). Recrystallization from a mixture of ethanol and water (10:1) gave the product **5b** (632 mg, 72 %) as off-white crystals. Mp: 114 °C. IR (KBr): ν 3273 (br), 2957, 2928, 2872 cm^{-1} . ^1H NMR (400 MHz, DMSO- d_6): δ 0.98 (3H, t, $J = 7.4$ Hz), 1.53 (2H, tq, $J = 7.4$ Hz), 1.84 (2H, tt, $J = 6.6$ Hz), 4.24 (2H, t, $J = 6.3$ Hz), 7.03-7.07 (2H, m), 7.38 (1H, t, $J = 8.0$ Hz), 7.55 (1H, dd, $J = 8.4$ Hz), 8.66 (1H, d, $J = 5.2$ Hz), 9.64 (1H, br s). ^{13}C NMR (100 MHz, DMSO- d_6): δ 14.2, 19.3, 30.8, 68.5, 102.4, 111.7, 111.9, 122.0, 126.9, 139.8, 149.6, 153.6, 161.5. HRMS: calcd for $\text{C}_{13}\text{H}_{16}\text{NO}_2$ ($[\text{M}+\text{H}]^+$) 218.1181, found 218.1163.

4-octyloxy-8-hydroxyquinoline (5c). The title compound was prepared by the procedure described in the journal (see Synthesis of **5a**) from **4c** (1.00 g, 2.34 mmol) and sodium hydroxide (1 M, 7.00 mL, 7.00 mmol). Recrystallization from methanol gave the product (500 mg, 79 %) as off-white crystals. Mp: 85 °C. IR (KBr): ν 3310 (br), 3032, 2950, 2855 cm^{-1} . ^1H NMR (400 MHz, DMSO- d_6): δ 0.92 (3H, t, $J = 6.7$ Hz), 1.33-1.46 (8H, m), 1.57 (2H, tt, $J = 7.2$ Hz), 1.96 (2H, tt, $J = 7.0$ Hz), 4.20 (2H, t, $J = 6.4$ Hz), 6.76 (1H, d, $J = 5.5$ Hz), 7.18 (1H, d, $J = 7.3$ Hz), 7.41 (1H, t, $J = 7.9$ Hz), 7.68 (1H, d, $J = 8.5$ Hz), 8.62 (1H, d, $J = 5.5$ Hz). ^{13}C NMR (100 MHz, DMSO- d_6): δ 14.1, 22.7, 26.1, 28.9, 29.2, 29.3, 31.8, 68.7, 101.3, 110.3, 112.2, 121.6, 126.4, 139.4, 149.0, 152.0, 162.1. HRMS: calcd for $\text{C}_{17}\text{H}_{24}\text{NO}_2$ ($[\text{M}+\text{H}]^+$) 274.1807, found 274.1796.

4-isopropoxy-8-hydroxyquinoline (5d). The title compound was prepared by the procedure described in the journal (see Synthesis of **5a**) from **4d** (800 mg, 2.24 mmol) and sodium hydroxide (1 M, 6.70 mL, 6.70 mmol). Recrystallization from ethanol gave the product (336 mg, 74 %) as off-white crystals. Mp: 135 °C. IR (KBr): ν 3300, 2981, 2934 cm^{-1} . ^1H NMR (200 MHz, DMSO- d_6): δ 1.39 (6H, d, $J = 6.0$ Hz), 4.92 (1H, sept, $J = 6.0$ Hz), 7.02-7.07 (2H, m), 7.35 (1H, t, $J = 7.6$ Hz), 7.53 (1H, dd, $J = 8.4$ Hz), 8.64 (1H, d, $J = 5.2$ Hz). ^{13}C NMR (50

MHz, DMSO-d₆): δ 21.7, 70.8, 102.7, 111.6, 111.7, 122.2, 126.5, 139.9, 149.3, 153.3, 160.1. HRMS: calcd for C₁₂H₁₄NO₂ ([M+H]⁺) 204.1025, found 204.1015.

4-benzyloxy-8-hydroxyquinoline (5e). The title compound was prepared by the procedure described in the journal (see Synthesis of **5a**) from **4e** (1.00 g, 2.46 mmol) and sodium hydroxide (1 M, 7.40 mL, 7.40 mmol). Recrystallization from ethanol gave the product (480 mg, 79 %) as white crystals. Mp: 123 °C. IR (KBr): ν 3271 (br), 3033, 2981, 2934, 2873 cm⁻¹. ¹H NMR (200 MHz, DMSO-d₆): δ 5.42 (2H, s), 7.10 (1H, dd, J = 7.6 Hz), 7.17 (1H, d, J = 5.2 Hz), 7.37-7.51 (4H, m), 7.56-7.64 (3H, m), 8.71 (1H, d, J = 5.2 Hz), 9.70 (1H, br s). ¹³C NMR (50 MHz, DMSO-d₆): δ 70.7, 103.4, 112.1, 112.5, 122.5, 127.5, 128.6 (2C), 129.0, 129.5 (2C), 137.0, 140.3, 150.0, 154.1, 161.5. HRMS: calcd for C₁₆H₁₂NO₂ ([M⁺]) 250.0868, found 250.0848.

4,8-dihydroxyquinoline (2) in KBr

D:\datafir\mkulju\4,8diHq.0

4,8diHq

KBr

2006/05/03

4,8-dihydroxyquinoline (2) in DMSO-d₆

SpinWorks 2.0: 4,8-dihydroxyquinoline

file: C:\Documents and Settings\mkulju\Onat\1iedostat1. Synteesiantikelli\NMR\4,8-dihydroxyquinoline
transmitter freq.: 400.132471 MHz
time domain size: 65536 points
width: 6250.83 Hz = 20.620234 ppm = 0.125898 Hz/pt
number of scans: 16

freq. of 0 ppm: 400.130000 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

4,8-dihydroxyquinoline (2) in DMSO-d₆

SpinWorks 2.0: 4,8-dihydroxyquinoline

4,8-dihydroxyquinoline (2)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

79 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:		5.0	3.0	20.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
162.0567	162.0555	1.2	7.4	6.5	C9 H8 N O2
	162.0531	3.6	22.2	3.5	C7 H9 N O2 Na

4-hydroxy-8-tosyloxyquinoline (3) in KBr

D:\datafir\mkulju8-(Tsoxy)quinolin-4-ol.0

8-(tosyloxy)quinolin-4-ol

KBr

2006/01/27

S11

SpinWorks 2.0: 8-tosyloxyquinolin-4-ol

4-hydroxy-8-tosyloxyquinoline (3) in DMSO-d₆

SpinWorks 2.0: 4-hydroxy-8-tosyloxyquinoline

4-hydroxy-8-tosyloxyquinoline (3) in DMSO-d₆

SpinWorks 2.5: hmbc 4-hydroxy-8-tosyloxyquinoline

4-hydroxy-8-tosyloxyquinoline (3)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

124 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0	
Maximum:			20.0	
Mass	Calc. Mass	mDa	PPM	DBE
316.0648	316.0644	0.5	1.6	10.5
	316.0619	2.9	9.2	7.5
				Formula
				C16 H14 N O4 S
				C14 H15 N O4 Na S

4-methoxy-8-tosyloxyquinoline (4a) in KBr

D:\datafir\mkulju\4Me8Tsq.0

4-Me-8-Tsq

KBr

2006/04/06

freq. of 0 ppm: 400.130000 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

```
file: C:\Documents and Settings\mkulju\Omni tiedoston1. Synteesiartikkel\NIR\4-Me-8-Tsq\H-NMR\fid      exp: <zg30>
```

transmitter freq.: 400.132471 MHz

time domain size: 65536 points

width: 8250.83 Hz = 20.620234 ppm = 0.125898 Hz/pt

number of scans: 16

4-methoxy-8-tosyloxyquinoline (4a) in DMSO-d₆

SpinWorks 2.0: 4-methoxy-8-tosyloxyquinoline

4-methoxy-8-tosyloxyquinoline (4a)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

128 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:			20.0		
Mass	Calc. Mass	mDa	PPM	DBE	Formula
330.0799	330.0800	-0.1	-0.4	10.5	C17 H16 N O4 S
	330.0776	2.3	6.9	7.5	C15 H17 N O4 Na S

N-methyl-8-tosyloxyquinolin-4-one (4f) in KBr

D:\datafir\mkulju\NMe8Tsq.0

N-Me-8-Tsq

KBr

2006/04/06

N-methyl-8-tosyloxyquinolin-4-one (4f) in DMSO-d₆

SpinWorks 2.0: N-methyl-8-tosyloxyquinolin-4-one

file: C:\Documents and Settings\mkulju\OneDrive\1_Synthesis\mkulju\NMR\N-Me-8-TsQ[C-NMR]\d exp: <ggg30>
transmitter freq.: 100.622830 MHz
time domain size: 65536 points
width: 24038.46 Hz = 238.896696 ppm = 0.366798 Hz/pt
number of scans: 104
freq. of 0 ppm: 100.612769 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

N-methyl-8-tosyloxyquinolin-4-one (4f)

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

136 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:		10.0	3.0	20.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
352.0652	352.0644	0.9	2.5	13.5	C19 H14 N O4 S
	352.0619	3.3	9.4	10.5	C17 H15 N O4 Na S

NMe8Tsq

LCT-15090 117 (2.344) AM (Gen,4, 95.00, Ar,5000.0,556.28); Sm (Mn, 2x1.00); Sb (1,40.00) 974

4-butoxy-8-tosyloxyquinoline (4b) in KBr

D:\datafir\mkujju\4Bu8Tsq.0

4-Bu-8-Tsq

KBr

2006/04/06

S24

SpinWorks 2.0: 4-butoxy-8-tosyloxyquinoline

freq. of 0 ppm: 400.130000 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

4-butoxy-8-tosyloxyquinoline (4b) in DMSO-d₆

SpinWorks 2.0: 4-butoxy-8-tosyloxyquinoline

file: C:\Documents and Settings\mkulju\OneDrive\Documents\1. Synthesiantikelti\NMR\4-Bu-8-TsQ\NMR\fid exp1 <zag90>
transmitter freq.: 100.62280 MHz
time domain size: 32768 complex points
width: 24038.46 Hz = 238.89666 ppm = 0.366798 Hz/pt
number of scans: 384

4-butoxy-8-tosyloxyquinoline (4b)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

143 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:		5.0	3.0	20.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
372.1306	372.1270	3.6	9.7	10.5	C20 H22 N O4 S

4-octyloxy-8-tosyloxyquinoline (4c) in KBr

D:\datafir\mkulju\4Oc8Tsq.0

4-Oc-8-Tsq

KBr

2006/04/06

4-octyloxy-8-tosyloxyquinoline (4c) in CDCl₃

SpinWorks 2.0: 4-octyloxy-8-tosyloxyquinoline

file: C:\Documents and Settings\mkulju\Oneat tiedostot\1. Synteesiatitkelt\NMR\4-Oct-8-Tsq\HNMR\fid exp: <230>
transmitter freq.: 400.132471 MHz
time domain size: 65536 points
width: 8250.83 Hz = 20.620234 ppm = 0.125898 Hz/pt
number of scans: 16
freq. of 0 ppm: 400.130000 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

4-octyloxy-8-tosyloxyquinoline (4c) in CDCl₃

SpinWorks 2.0: 4-octyloxy-8-tosyloxyquinoline

file: C:\Documents and Settings\mkulju\OneDrive\1. Synthesieritkellin\NMR\4-Oct-8-Tsq\1C-NMR\fid exp: <agg30>
 transmitter freq.: 100.62280 MHz
 time domain size: 65536 points
 width: 24038.46 Hz = 238.89666 ppm = 0.366798 Hz/pt
 number of scans: 179

freq. of 0 ppm: 100.612769 MHz
 processed size: 32768 complex points
 LB: 0.000 GB: 0.0000

4-octyloxy-8-tosyloxyquinoline (4c)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

129 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:		5.0	3.0	20.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
428.1903	428.1896	0.7	1.7	10.5	C24 H30 N O4 S

WA-octyl

LCT-14977 57 (1.144) AM (Cen,4, 95.00, Ar,5000.0,556.28); Sm (Mn, 2x1.00); Sb (1,40.00) 3.77e3

4-isopropoxy-8-tosyloxyquinoline (4d) in KBr

D:\datafir\jahe\4-ipro-8-tq.0

4-ipro-8-tq

KBr

2006/10/06

4-isopropoxy-8-tosyloxyquinoline (4d) in DMSO-d₆

SpinWorks 2.5:

file: C:\Documents and Settings\Juhale\OneDrive\NMR\2\1d exp: <2g30>
transmitter freq: 200.131236 MHz
time domain size: 24750 points
width: 4125.41 Hz = 20.613537 ppm = 0.166663 Hz/pt
number of scans: 16
freq: 0 ppm: 200.129999 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.000

4-isopropoxy-8-tosyloxyquinoline (4d) in DMSO-d₆

SpinWorks 2.5: J2, 4-ipro-8-tq, DMSO

file: C:\Documents and Settings\j\OneDrive\OneDrive\OneDrive\11fid exp: <ggg30>

transmitter freq.: 50.327740 MHz
processed size: 32768 complex points
LB: 0.300 GB: 0.000time domain size: 65536 points
width: 12019.23 Hz = 238.819105 ppm = 0.183399 Hz/pt
number of scans: 12001

4-isopropoxy-8-tosyloxyquinoline (4d)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -0.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

162 formula(e) evaluated with 4 results within limits (up to 50 closest results for each mass)

Minimum:	-0.5				
Maximum:	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	Formula
380.0943	380.0932	1.1	2.8	10.5	C19 H19 N O4 Na S
	380.0957	-1.3	-3.5	13.5	C21 H18 N O4 S
	380.0923	2.0	5.4	18.5	C24 H14 N O4
	380.0899	4.5	11.7	15.5	C22 H15 N O4 Na

4-benzoyloxy-8-tosyloxyquinoline (4e) in KBr

D:\datafir\mkulju\4Bz8Tsq.0

4-Bz-8-Tsq

KBr

2006/04/06

4-benzoyloxy-8-tosyloxyquinoline (4e) in DMSO-d₆ at 50 °C

SpinWorks 2.0: 4-benzoyloxy-8-tosyloxyquinoline

file: C:\Documents and Settings\mkulju\OneDrive\Documents\1. Synthesiantikku\NMR\4-Bz-8-Tsq\1fid exp: <zg0>
transmitter freq.: 400.132471 MHz
time domain size: 65536 points
width: 6250.83 Hz = 20.620234 ppm = 0.125998 Hz/pt
number of scans: 16
freq. of 0 ppm: 400.130000 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

4-benzoyloxy-8-tosyloxyquinoline (4e) in DMSO-d₆ at 50 °C

SpinWorks 2.0: 4-benzoyloxy-8-tosyloxyquinoline

file: C:\Documents and Settings\mkulju\Ornat\liedostok1. Synthesiantikell\NMR\4-Bz-8-Ts\q2fid exp: <zpg30>

transmitter freq.: 100.62280 MHz

time domain size: 65536 points

width: 24038.46 Hz = 238.89666 ppm = 0.366798 Hz/pt

number of scans: 253

freq. of 0 ppm: 100.612769 MHz

processed size: 32768 complex points

LB: 1.000 GB: 0.0000

4-benzoyloxy-8-tosyloxyquinoline (4e)

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

61 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:		10.0	3.0	20.0	
		mDa	PPM	DBE	
Mass	Calc. Mass				Formula
406.1114	406.1113	0.1	0.3	14.5	C23 H20 N O4 S
	406.1089	2.5	6.2	11.5	C21 H21 N O4 Na S

btg

lct-14758 282 (5.645) AM (Cen, 4, 95.00, Ar, 5000.0, 556.28); Sm (Mn, 2x1.00); Sb (1, 40.00) 2.33e3

4-methoxy-8-hydroxyquinoline (5a) in KBr

D:\datafir\mkulju\4Me8Hq.0

4-Me-8-Hq

KBr

2006/04/06

4-methoxy-8-hydroxyquinoline (5a) in DMSO-d₆

SpinWorks 2.0: 4-methoxyquinolin-8-ol

4-methoxy-8-hydroxyquinoline (5a) in DMSO-d₆

SpinWorks 2.0: 4-methoxyquinolin-8-ol

file: C:\Documents and Settings\mkulju\OneDrive\Documents\1. Synthesiarbeitskette\NMR\4-Me-8-HQ\1vid exp1 <gpg3d>
 transmitter freq.: 50.327761 MHz
 time domain size: 65536 points
 width: 12019.23 Hz = 238.819105 ppm = 0.183399 Hz/pt
 number of scans: 201

freq. of 0 ppm: 50.322709 MHz
 processed size: 32768 complex points
 LB: 0.000 GB: 0.0000

4-methoxy-8-hydroxyquinoline (5a)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

50 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:			20.0		
Mass	Calc. Mass	mDa	PPM	DBE	Formula
176.0690	176.0712	-2.1	-12.0	6.5	C10 H10 N O2

4-butoxy-8-hydroxyquinoline (5b) in KBr

D:\datafir\mkulju\4Bu8Hq.0

4Bu8Hq

KBr

2006/05/03

4-butoxy-8-hydroxyquinoline (5b) in DMSO-d₆

SpinWorks 2.0: 4-butoxy-8-hydroxyquinoline

file: C:\Documents and Settings\mkulju\OneDrive\1. Synthesier\mkulju\NMR\Hyd5\1\fid exp: -zg30>

transmitter freq.: 400.132471 MHz

time domain size: 65536 points

width: 6250.83 Hz = 20.620234 ppm = 0.125898 Hz/pt

number of scans: 16

freq. of 0 ppm: 400.130000 MHz

processed size: 32768 complex points

LB: 0.000 GB: 0.0000

4-butoxy-8-hydroxyquinoline (5b) in DMSO-d₆

SpinWorks 2.0: 4-butoxy-8-hydroxyquinoline

file: C:\Documents and Settings\mkulju\OneDrive\1. Synthesiarbeitskette\NMR\Hyd52\fid exp1 -zpgg30>

transmitter freq.: 100.622830 MHz

time domain size: 65536 points

width: 24038.46 Hz = 238.89666 ppm = 0.366798 Hz/pt

number of scans: 186

freq. of 0 ppm: 100.612769 MHz

processed size: 32768 complex points

LB: 0.000 GB: 0.0000

4-butoxy-8-hydroxyquinoline (5b)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -0.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

55 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Minimum:			-0.5		
Maximum:		5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Formula
218.1163	218.1157	0.6	2.8	3.5	C11 H17 N O2 Na
218.1181	218.1181	-1.8	-8.3	6.5	C13 H16 N O2

4-octyloxy-8-hydroxyquinoline (5c) in KBr

D:\datafir\mkulju4Oc8Hq.D

4Oc8Hq

KBr

2006/05/03

4-octyloxy-8-hydroxyquinoline (5c) in CDCl₃

SpinWorks 2.0: 4-octyloxyquinolin-8-ol

file: C:\Documents and Settings\mkulju\OneDrive\Documents\1. Synteesiartikkelit\NMR\4-Oct-8-Hq\1fid expir <zg30>

freq. of 0 ppm: 400.130000 MHz

processed size: 32768 complex points

LB: 0.000 GB: 0.0000

transmitter freq.: 400.132471 MHz

time domain size: 65536 points

width: 8250.83 Hz = 20.620234 ppm = 0.125898 Hz/pt

number of scans: 16

4-octyloxy-8-hydroxyquinoline (5c) in CDCl₃

SpinWorks 2.0: 4-octyloxy-8-hydroxyquinoline

4-octyloxy-8-hydroxyquinoline (5c)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -0.5, max = 50.0

Monoisotopic Mass, Odd and Even Electron Ions

153 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)					
Minimum:					-0.5
Maximum:		5.0	5.0	DBE	50.0
Mass	Calc. Mass	mDa	PPM		Formula
274.1796	274.1807	-1.1	-4.0	6.5	C17 H24 N O2
	274.1783	1.3	4.7	3.5	C15 H25 N O2 Na
	274.1780	1.6	5.7	2.0	C14 H26 O5

4-isopropoxy-8-hydroxyquinoline (5d) in KBr

D:\datafir\jahe\4-ipro-8-hq.0

4-ipro-8-hq

KBr

2006/10/06

4-isopropoxy-8-hydroxyquinoline (5d) in DMSO-d₆

SpinWorks 2.5:

file: C:\Documents and Settings\Juhue\OneDrive\NMR\Juhue\carbon11fid exp: <zggg30>

freq. of 0 ppm: 50.327740 MHz
processed size: 32768 complex points
LB: 1.000 GB: 0.0000transmitter freq.: 50.327761 MHz
time domain size: 65536 points
width: 12019.23 Hz = 238.819105 ppm = 0.183399 Hz/pt
number of scans: 11769

4-isopropoxy-8-hydroxyquinoline (5d)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -0.5, max = 50.0

Monoisotopic Mass, Even Electron Ions

46 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Minimum:			-0.5		
Maximum:	5.0	4.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Formula
204.1015	204.1025	-0.9	-4.5	6.5	C12 H14 N O2
	204.1000	1.5	7.3	3.5	C10 H15 N O2 Na

J4

LCT-16068 146 (2.924) AM (Top,4, Ar,5000.0,413.27); Sm (Mn, 2x1.00); Sb (1,40.00); Sb (1,42.02e3

4-benzoyloxy-8-hydroxyquinoline (5e) in KBr

D:\datafir\mkulju4-(Bzoxyl)quinolin-8-ol.0

4-(Bzoxyl)quinolin-8-ol

KBr

2006/01/27

4-benzoyloxy-8-hydroxyquinoline (5e) in DMSO-d₆

SpinWorks 2.5: 4-benzoyloxy-8-hydroxyquinoline

file: C:\Documents and Settings\mkulji\Oneat\iedosol\NMR 2006\4.2 hydrolysis\BzLQ11fid exp: <g30>
transmitter freq: 200.131236 MHz
time domain size: 24750 points
width: 4125.41 Hz = 20.613337 ppm = 0.166693 Hz/pt
number of scans: 16

freq: 0 ppm: 200.130000 MHz
processed size: 32768 complex points
LB: 0.300 GB 0.0000

4-benzylloxy-8-hydroxyquinoline (5e) in DMSO-d₆

SpinWorks 2.0: 4-benzylloxyquinolin-8-ol

file: C:\Documents and Settings\mkulju\Ornat tiedot\st1. Synthesiantikellin\NMR\4-Bz-8-Hq\3fid - expt <ggg30>
transmitter freq.: 50.32709 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

time domain size: 32768 points
width: 6250.83 Hz = 163.936879 ppm = 0.251795 Hz/pt
number of scans: 12288

4-benzylloxy-8-hydroxyquinoline (5e)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -5.0, max = 20.0

Monoisotopic Mass, Even Electron Ions

57 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:			-5.0		
Maximum:		5.0	3.0	20.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
250.0848	250.0868	-2.0	-8.0	11.5	C16 H12 N O2

4-Bz-8-Hq

LCT-14590 39 (0.782) AM (Cen,4, 95.00, Ar,5000.0,554.26); Sm (Mn, 2x1.00); Sb (1,40.00); Si 3.22e3