Supporting Information

A modified Procedure for the Synthesis of 2-chloroquinoline-3-carbaldehydes using phosphorus pentachloride

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I.- General Experimental Information

The acetanilides and phosphorus pentachloride were commercially available agents and used without further purification. All solvents were purified according to *Purification of Laboratory Chemicals (4th edition)*. Silica gel plate GF254 were used for thin layer chromatography (TLC). Melting points were recorded with a micro melting point apparatus und uncorrected. ^1H and ^{13}C NMR spectra were recorded at JEOL Eclipse Plus 400 and JEOL Eclipse Plus 270. Proton chemical shifts were given in relative to tetramethylsilane (δ 0.00 ppm) in CDCl_3 . Multiplicity was indicated as follows: s (singlet), d (doublet), t (triple), m (multiplet), dd (doublet of doublets), br s (broad singlet), etc, and coupling constant were given in Hz. Carbon chemical shifts were internally referenced to the deuterated solvent signals in CDCl_3 (δ 77.00 ppm). IR spectra were recorded on NICOLET Magna IR 760 using KBr tablets. Elemental analysed the synthesized compounds were performed on a Perkin Elmer 2400 CHN analyser: results fell in the range of 0,4% of the required theoretical values.

II.- Synthesis procedures and spectroscopic data

(1) General procedure for the synthesis of 2-chloroquinoline-3-carbaldehydes 5a-i:

Dimethylformamide (12 mmol, 3 equiv.) was cooled at 0 °C in a round flask equipped with a drying tube and phosphorus pentachloride (18 mmol, 4.5 equiv.) was added slowly and the mixture was stirred for 15 minutes keeping the temperature below 0 °C. To this solution was added in a portion the corresponding acetanilide (4 mmol, 1 equiv.) and the reaction mixture was heated under reflux and stirring for the appropriate time depending of the acetanilide. The resulted mixture was cooled to 0°C and the solution was poured slowly into ice-water and stirring for ten minutes, obtaining a yellow solid which was filtered, washed several time with cold water and dried under vacuum. The 2-chloroquinoline-3-carbaldehydes were recrystalized according to the literature.^[4]

2-chloroquinoline-3-carbaldehyde (5a): Recrystalization eluent: Ethyl acetate; Yield 552 mg (72%); yellow pale solid, mp 145-146 °C. IR (KBr): 3043 (st. C-H), 2872 (st. C-H), 1688 (st. C=O), 1615-1553 (st. C=C) cm^{-1} . $^1\text{H-NMR}$ (270 MHz, J Hz, CDCl_3) δ : 10,55 (s, 1H); 8,76 (s, 1H); 8,07 (d, $J=8,6$; 1H); 7,98 (d, $J=7,9$; 1H); 7,89 (dd, $J_1=8,3$; $J_2=7,7$; 1H); 7,64 (dd, $J_1=8,3$; $J_2=7,9$; 1H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ : 189,4 (d); 150,3 (s); 140,5 (d); 133,7 (d); 130,6 (s); 129,6 (s); 129,1 (d); 127,7(d); 126,6 (d); 121,2. Anal. Calcd for $\text{C}_{10}\text{H}_6\text{ClNO}$: C, 62,48; H, 3,11; N, 7,23 %. Found: C, 62,68; H, 3,16; N, 7,31 %.

2-chloro-6-methylquinoline-3-carbaldehyde (5b): Recrystallization eluent: Ethyl acetate; Yield 525 mg (64%); yellow pale solid; mp 121-123 °C. IR (KBr): 3073 (st. C-H), 2876 (st. C-H), 1690 (st. C=O), 1623-1581-1500 (st. C=C) cm^{-1} . $^1\text{H-NMR}$ (400 MHz, J Hz, CDCl_3) δ : 10,53 (s, 1H); 8,65 (s, 1H); 7,95 (d, $J=8,8$; 1H); 7,70 (dd, $J_1=9,3$; $J_2=1,8$; 1H); 7,26 (d, $J=2,0$; 1H); 2,55 (s, 3H, CH_3). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ : 189,5 (d); 149,4 (d); 148,3 (d); 139,7 (d); 138,5 (d); 136,1 (d); 128,4 (d); 128,4 (d); 126,7 (s); 126,3 (d); 21,7 (c). Anal. Calcd for $\text{C}_{11}\text{H}_8\text{ClNO}$: C, 64,09; H, 3,83; N, 6,61 %. Found: C, 64,25; H, 3,92; N, 6,81 %.

2-chloro-7-methylquinoline-3-carbaldehyde (5c): Recrystallization eluent: Ethyl acetate; Yield 583 mg (71%); yellow pale solid; mp 142-144 °C. IR (KBr): 3056 (st. C-H), 1685 (st. C=O), 1628-1577-1520 (st. C=C) cm^{-1} . $^1\text{H-NMR}$ (400 MHz, J Hz, CDCl_3) δ : 10,51 (s, 1H); 8,68 (s, 1H); 7,85- 7,81 (m, 2H); 7,46 (d, $J=8,1$; 1H); 2,58 (s, 3H, CH_3). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ : 189,4 (d); 150,2 (d), 145,2 (d), 140,0 (d); 131,3 (s); 130,0 (s); 128,6 (s); 126,9 (s); 125,7 (d); 124,7 (s); 22,4 (c). Anal. Calcd for $\text{C}_{11}\text{H}_8\text{ClNO}$: C, 64,16; H, 3,77; N, 6,68 %. Found: C, 64,25; H, 3,92; N, 6,81 %.

2-chloro-8-methylquinoline-3-carbaldehyde (5d): Recrystalization eluent: Ethyl acetate; Yield 492 mg (72%); yellow pale solid; mp 133-135 °C. IR (KBr): 3056 (st. C-H), 1687 (st. C=O), 1628-1578-1520 (st. C=C) cm^{-1} . $^1\text{H-NMR}$ (270 MHz, J Hz, CDCl_3) δ : 10,54 (s, 1H); 7,78 (d, $J=8,2$; 1H); 7,69 (d, $J=7,4$; 1H); 7,54 (d, $J=7,6$; 1H); 7,48 (s, 1H), 2,74 (s, 3H, CH_3). Anal. Calcd for $\text{C}_{11}\text{H}_8\text{ClNO}$: C, 64,14; H, 3,72; N, 6,73 %. Found: C, 64,25; H, 3,92; N, 6,81 %.

2-chloro-6-methoxyquinoline-3-carbaldehyde (5e): Recrystalization eluent: Ethyl acetate; Yield 435 mg (49%); yellow pale solid, mp 142-144 °C. IR (KBr): 3054 (st. C-H), 2872 (st. C-H), 1690 (st. C=O), 1617-1578-1503 (st. C=C) cm^{-1} . $^1\text{H-NMR}$ (270 MHz, J Hz, CDCl_3) δ : 10,53 (s, 1H); 8,63 (s, 1H); 7,95 (d, $J=9,16$, 1H); 7,50 (dd, $J_1=9,36$; $J_2=2,96$; 1H); 7,18 (d, $J=2,6$, 1H); 3,94 (s, 3H, OCH_3). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ : 189,6 (d); 158,9 (s); 147,8 (d), 145,9 (s), 138,8 (d); 130,0 (d); 127,7 (d); 126,4 (d); 125,9 (d); 106,5 (d) ; 55,9 (c) (OCH_3). Anal. Calcd for $\text{C}_{11}\text{H}_8\text{ClNO}_2$: C, 59,47; H, 3,56; N, 6,26 %. Found: C, 59,61; H, 3,64; N, 6,32.

2-chloro-7-methoxyquinoline-3-carbaldehyde (5f): Recrystalization eluent: Ethyl acetate; Yield 655 mg (74%); yellow pale solid, mp 189-191 °C. IR (KBr): 3046 (st. C-H), 2878 (st. C-H), 1687 (st. C=O), 1625-1585 (st. C=C) cm^{-1} . $^1\text{H-NMR}$ (270 MHz, J Hz, CDCl_3) δ : 10,48 (s, 1H); 8,63 (s, 1H); 7,82 (d, $J=8,80$; 1H); 7,35 (d, $J=2,20$; 1H); 7,25 (dd, $J_1=9,02$; $J_2=2,20$;

1H); 3,96 (s, 3H, OCH₃). ¹³C-NMR (100 MHz, CDCl₃) δ: 189,2 (d); 164,3 (s); 152,0 (d); 139,6 (d); 130,9 (d); 124,4 (d); 122,5 (s), 121,8 (s); 120,8 (s); 106,9 (d); 56,0 (c) (OCH₃).
 Anal. Calcd for C₁₁H₈ClNO₂: C, 59,54; H, 3,59; N, 6,27 %. Found: C, 59,61; H, 3,64; N, 6,32 %.

2-chloro-6-bromoquinoline-3-carbaldehyde (5g): Recrystallization eluent: Acetonitrile; Yield 302 mg (28%); yellow pale solid; mp 186-188 °C. IR (KBr): 3046 (st. C-H), 2878 (st. C-H), 1687 (st. C=O), 1625-1585 (st. C=C) cm⁻¹. ¹H-NMR (270 MHz, *J* Hz, CDCl₃) δ: 10,54 (s, 1H); 8,65 (s, 1H); 8,12 (d, *J*=8,8; 1H); 7,92-7,93 (m, 2H). Anal. Calcd for C₁₀H₅BrClNO: C, 44,28; H, 1,76; N, 5,03 %. Found: C, 44,40; H, 5,18; N, 5,91 %.

2,7-dichloroquinoline-3-carbaldehyde (5h): Recrystallization eluent: Acetonitrile. Yield 271 mg (30%). IR (KBr, cm⁻¹): 3054 (st. C-H), 1689 (st. C=O), 1609-1581-1559 (st. C=C). ¹H-NMR (270 MHz, *J* Hz, CDCl₃) δ: 10,53 (s, 1H); 8,72 (s, 1H); 8,05 (s, 1H); 7,91 (d, *J*=8,9; 1H); 7,59 (dd, *J*₁=8,8; *J*₂=1,7; 1H). Anal. Calcd for C₁₀H₅Cl₂NO: C, 52,98; H, 2,17; N, 6,09 %. Found: C, 53,13; H, 2,23; N, 6,20 %.

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% Transmitancia

SI_AR_6Me_quinolina.3
SI_AR_6Me_quinolina_carbono desacop

SL_AR_6Me_quinolina.3
SL_AR_6Me_quinolina_carbono desacop

% Transmittancia

Número de Ondas (cm⁻¹)

SL_AR_7Me_quinol.5
SL_AR_7Me_quinol

X : parts per Million : 1H

SL_AR_7Me_quinolina.5
SL_AR_7Me_quinolina_carbono acop

SL_AR_7Me_quinolina.5
SL_AR_7Me_quinolina_carbono acop

X : parts per Million : ¹³C

% Transmitancia

Número de Ondas (cm⁻¹)

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% Transmitancia

Número de Ondas (cm⁻¹)

SL_AR_6OMequinol.3
protones

X : parts per Million : 1H

SL_AR_6OMequinol.3
protones

6-OMe

SL_AR_pOMequinolina_carbon.5
SL_AR_pOMequinolina_carbon acopl

SL_AR_pOMequinolina_carbon.5
SL_AR_pOMequinolina_carbon.acopl

127,886

SL_AR_pOMequinolina_carbon.5
SL_AR_pOMequinolina_carbon acopl

% Transmitancia

Número de Ondas (cm⁻¹)

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SL_AR_7OMe_quinolina2_proton

X : parts per Million : 1H

SL_AR_7OMe_quinolina2.4
SL_AR_7OMe_quinolina2_carbono acop

SL_AR_7OMe_quinolina2.4
SL_AR_7OMe_quinolina2_carbono acop

SL_AR_7OMe_quinolina2.4
SL_AR_7OMe_quinolina2_carbono acop

% Transmitancia

Número de Ondas (cm⁻¹)

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% Transmitancia

Número de Ondas (cm⁻¹)

References

- [1] (a) Baruah, B., Bhuyan, P.J., *Tetrahedron*, **2009**, 65, 7099. (b) Chandra, A., Singh, B., Khanna, R.S., Singh, R.M. *J. Org. Chem.* **2009**, 74, 5664. (c) Verma, A.K., Aggarwal, T., Rustagi, V., Larock, R.C., *Chem. Commun.* **2010**, 46, 4064. (d) Kalita, P.K., Baruah, B., Bhuyan, P.J., *Tetrahedron Lett.* **2006**, 47, 7779. (e) Li, P., Majireck, M.M., Korboukh, I., Weinreb, S.M., *Tetrahedron Lett.* **2008**, 49, 3162. (f) Abdel-Wahab, B.F., Khidre, R.E., Farahat, A.A., Sayed El-Ahl, A.A., *ARKIVOC*, **2012**, 211-276. (g) Abdel-Wahab, B.F., Khidre, R.E., *J. Chem.*, **2013**. (<http://dx.doi.org/10.1155/2013/851297>).
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