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An Effective Procedure for the Acylation of Azaindoles at C-3

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HRMS Results

Compound Number	Formula [M+H]	Theoretic m/z.	Observed m/z	Deviation in ppm
7	C ₁₄ H ₁₁ N ₂ O	223.0871	223.0873	-0.8
13	C ₁₄ H ₁₁ N ₂ O	223.0871	223.0872	-0.3
14	C ₁₀ H ₉ N ₂ O ₃	205.0613	205.0616	-1.4
15	C ₉ H ₉ N ₂ O	161.0715	161.0721	-3.7
16	C ₁₄ H ₁₁ N ₂ O	223.0871	223.0879	-3.4
Compound Number	Formula [M-H]	Theoretic m/z.	Observed m/z	Deviation in ppm
5	C ₁₀ H ₇ N ₂ O ₃	203.0457	203.0448	4.4
6	C ₉ H ₇ N ₂ O	159.0558	159.0565	-4.0
8	C ₁₀ H ₇ N ₂ O ₃	203.0457	203.0457	0.0
9	C ₉ H ₇ N ₂ O	159.0558	159.0562	-2.2
10	C ₁₄ H ₉ N ₂ O	221.0715	221.0714	0.5
11	C ₁₀ H ₇ N ₂ O ₃	203.0457	203.0447	4.7
12	C ₉ H ₇ N ₂ O	159.0558	159.0562	-2.0

Supporting Material: Spectra

Methyl α -oxo-7-azaindole-3-acetate, 5. ^1H NMR (300 MHz, DMSO- d_6) δ 8.60 (s, 1H), 8.47 (d, 1H, J = 7.86 Hz), 8.40 (d, 1H, J = 4.71 Hz), 7.34 (dd, 1H, J = 7.86, 4.77 Hz), 3.99 (s, 3H); ^{13}C NMR (75 MHz, DMSO- d_6) δ 178.7, 163.3, 149.0, 145.1, 138.8, 129.7, 119.0, 118.0, 111.2, 52.7; HRMS m/z : (M-H) $^+$ calcd for $\text{C}_{10}\text{H}_7\text{N}_2\text{O}_3$ 203.0457, found 203.0448.

3-Acetyl-7-azaindole, 6. ^1H NMR (500 MHz, CD_3OD) δ 8.55 (m, 1H), 8.29 (m, 2H), 7.24 (m, 1H), 2.51 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 196.3, 150.1, 145.3, 135.7, 132.1, 119.9, 119.4, 117.4, 27.0; HRMS m/z : (M-H) $^+$ calcd for $\text{C}_9\text{H}_7\text{N}_2\text{O}$ 159.0558, found 159.0565.

3-Benzoyl-7-azaindole, 7. ^1H NMR (500 MHz, DMSO- d_6) δ 8.53 (d, 1H, J = 7.85 Hz), 8.38 (d, 1H, J = 4.70 Hz), 8.09 (s, 1H), 7.81 (d, 2H, J = 6.85 Hz), 7.63 (t, 1H, J = 6.5 Hz), 7.56 (t, 2H, J = 7.65 Hz), 7.30 (m, 1H); ^{13}C NMR (125 MHz, DMSO- d_6) δ 189.7, 149.0, 144.4, 139.6, 135.8, 131.3, 129.7, 128.41, 128.38, 118.6, 118.1, 113.6; HRMS m/z : (M+H) $^+$ calcd for $\text{C}_{14}\text{H}_{11}\text{N}_2\text{O}$ 223.0871, found 223.0873.

Methyl α -oxo-6-azaindole-3-acetate, 8. ^1H NMR (500 MHz, DMSO- d_6 & CDCl_3) δ 9.34 (s, 1H), 9.15 (s, 1H), 8.55 (m, 2H), 3.93 (s, 3H); ^{13}C NMR (125 MHz, DMSO- d_6 & CDCl_3) δ 177.9, 161.9, 146.2, 136.0, 132.4, 132.1, 129.1, 117.6, 112.7, 52.8; HRMS m/z : (M-H) $^+$ calcd for $\text{C}_{10}\text{H}_7\text{N}_2\text{O}_3$ 203.0457, found 203.0457.

3-Acetyl-6-azaindole, 9. ^1H NMR (500 MHz, CD_3OD) δ 8.76 (s, 1H), 8.39 (s, 1H), 8.23 (d, 1H, $J = 5.5$ Hz), 8.16 (d, 1H, $J = 5.55$ Hz), 2.55 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 196.2, 141.1, 138.9, 135.9, 132.6, 118.2, 117.8, 27.6; HRMS m/z : (M-H) $^+$ calcd for $\text{C}_9\text{H}_7\text{N}_2\text{O}$ 159.0558, found 159.0562.

3-Benzoyl-6-azaindole, 10. ^1H NMR (500 MHz, CD_3OD) δ 8.81 (s, 1H), 8.29 (d, 1H, $J = 5.55$ Hz), 8.21 (d, 1H, $J = 5.55$ Hz), 8.10 (s, 1H), 7.81 (d, 2H, $J = 7.00$ Hz), 7.62 (m, 1H), 7.53 (m, 2H); ^{13}C NMR (125 MHz, CD_3OD) δ 193.2, 141.4, 141.3, 140.2, 135.7, 133.7, 133.0, 129.8, 129.7, 117.9, 116.9; HRMS m/z : (M-H) $^+$ calcd for $\text{C}_{14}\text{H}_9\text{N}_2\text{O}$ 221.0715, found 221.0714.

Methyl α -oxo-5-azaindole-3-acetate, 11. ^1H NMR (500 MHz, CD_3OD) δ 9.61 (s, 1H), 9.02 (s, 1H), 8.59 (d, 1H, $J = 6.63$ Hz), 8.15 (d, 1H, $J = 6.60$ Hz), 4.00 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 178.9, 163.0, 145.6, 144.2, 138.3, 135.0, 124.7, 116.3, 112.1, 53.8; HRMS m/z : (M-H) $^+$ calcd for $\text{C}_{10}\text{H}_7\text{N}_2\text{O}_3$ 203.0457, found 203.0447.

3-Acetyl-5-azaindole, 12. ^1H NMR (500 MHz, CD_3OD) δ 9.38 (s, 1H), 8.26 (m, 2H), 7.49 (d, 1H, $J = 5.65$ Hz), 2.55 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 196.0, 145.1, 142.8, 142.2, 136.8, 123.8, 118.8, 109.0, 27.3; HRMS m/z : (M-H) $^+$ calcd for $\text{C}_9\text{H}_7\text{N}_2\text{O}$ 159.0558, found 159.0562.

3-Benzoyl-5-azaindole, 13. ^1H NMR (500 MHz, DMSO- d_6) δ 9.43 (s, 1H), 8.34 (d, 1H, J = 5.55 Hz), 8.08 (s, 1H as "7.83 (d, 2H, J = 7.00 Hz), 7.63 (m, 1H), 7.57 (m, 2H), 7.52 (d, 1H, J = 5.55 Hz); ^{13}C NMR (125 MHz, DMSO- d_6) δ 189.6, 144.0, 142.0, 140.4, 139.7, 136.4, 131.4, 128.4, 128.3, 122.9, 114.7, 107.5; HRMS m/z : (M+H) $^+$ calcd for $\text{C}_{14}\text{H}_{11}\text{N}_2\text{O}$ 223.0871, found 223.0872.

Methyl α -oxo-4-azaindole-3-acetate, 14. ^1H NMR (500 MHz, CD_3OD) δ 9.17 (s, 1H), 8.75 (d, 1H, J = 8.30 Hz), 8.69 (d, 1H, J = 5.85 Hz), 7.91 (m, 1H), 4.01 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 178.5, 162.7, 145.2, 138.2, 137.1, 134.9, 131.6, 120.9, 110.5, 53.8; HRMS m/z : (M+H) $^+$ calcd for $\text{C}_{10}\text{H}_9\text{N}_2\text{O}_3$ 205.0613, found 205.0616.

3-Acetyl-4-azaindole, 15. ^1H NMR (500 MHz, CD_3OD) δ 8.49 (d, 1H, J = 4.65 Hz), 8.31 (s, 1H), 7.87 (d, 1H, J = 8.25 Hz), 7.25 (m, 1H), 2.70 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 196.2, 145.8, 144.6, 136.8, 131.7, 121.4, 119.0, 118.2, 29.0; HRMS m/z : (M+H) $^+$ calcd for $\text{C}_9\text{H}_9\text{N}_2\text{O}$ 161.0715, found 161.0721.

3-Benzoyl-4-azaindole, 16. ^1H NMR (500 MHz, CDCl_3) δ 8.87 (d, 1H, J = 8.25 Hz), 8.68 (d, 1H, J = 5.90 Hz), 8.53 (s, 1H), to "7.73 (d, 2H, J = 7.85 Hz), 7.66 (m, 1H), 7.61 (t, 1H, J = 7.30 Hz), 7.45 (t, 2H, J = 7.60 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 190.1, 142.2, 137.3, 136.5, 135.2, 134.0, 133.3, 131.3, 129.0, 128.5, 118.6, 110.4; HRMS m/z : m/z : (M+H) $^+$ calcd for $\text{C}_{14}\text{H}_{11}\text{N}_2\text{O}$ 223.0871, found 223.0879.

Figure Captions

S7

8S

6S