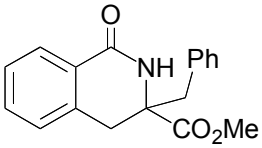
Supplementary Material (ESI) for Chemical Communications

Preparation of benzolactams by Pd(II)-catalyzed carbonylation of N-unprotected arylethylamines

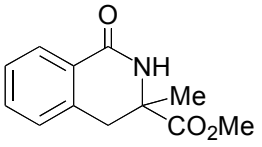
Blanca López, Aleix Rodriguez, David Santos, Joan Albert, Xavier Ariza, Jordi Garcia, and Jaume Granell,
Departaments de Química Orgànica, Inorgànica i IBUB, Fac. De Química, Universitat de Barcelona,
jordigarciagomez@ub.edu; jaume.granell@qi.ub.es

Typical Procedure: A stirred suspension of methyl 2-amino-2-benzyl-3-phenylpropanonate **1c** (100 mg, 0.38 mmol), benzoquinone (83 mg, 0.76 mmol) and palladium acetate (4.5 mg, 0.02 mmol) in AcOH (25 mL) was gently refluxed in an oil bath at 120 ºC in a 100-mL three-necked flask provided with a reflux condenser closed by a toy balloon under atmosphere of carbon monoxide (overal gas volume ~150 mL) for 6 h. Internal presure (~1 atm) was checked during all the process. The reaction mixture was cooled, a filtered through a thin pad of Celite®. The volatiles were removed under vacuum to obtain a solid corresponding to almost pure benzolactam **5c**. The residue was purified by flash chromatography to afford **5c** (105 mg, 93%).



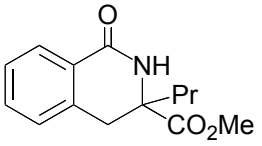
5c

Methyl 3-benzyl-1-oxo-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **5c**: White solid; mp 128-129 ºC; R_f (hexane/EtAcO 7:3): 0.30; ^{1}H NMR (400 MHz; $CDCl_3$; Me_4Si): δ 8.09 (1 H, d, J = 7.6 Hz), 7.49 (1 H, m), 7.38 (1 H, m), 7.23-7.31 (4 H, m), 7.06 (2 H, m), 6.26 (1 H, br s, NH), 3.63 (3 H, s, OCH_3), 3.44 (1 H, d, J = 15.8 Hz, CHH), 3.24 (1 H, d, J = 13.4 Hz, CHH), 3.23 (1 H, d, J = 15.8 Hz, CHH), 3.03 (1 H, d, J = 13.4 Hz CHH); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 172.6 (*C*OO), 165.0 (*C*ONH), 135.5 (q), 134.0 (q), 132.7, 129.7, 128.7, 128.2, 127.8, 127.6, 127.5, 62.8 (q), 52.7 (O*C*H$_3$), 44.6, 36.8; IR (ATR): ν_{max} 3191, 1731, 1663, 1603, 1384. HRMS (ESI$^+$) calcd for $C_{18}H_{18}NO_3$ $(M+H)^+$ 296.1281, found 296.1283.



5a

Methyl 3-methyl-1-oxo-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **5a**: White solid; mp 180-182 ºC (lit.[1] 181-183 ºC); R_f (CH_2Cl_2/MeOH 98:2): 0.31; ^{1}H NMR (400 MHz; $CDCl_3$; Me_4Si): δ 8.07 (1 H, d, J = 7.7 Hz), 7.46 (1 H, m), 7.36 (1 H, m), 7.22 (1 H, d, J = 7.5 Hz), 6.58 (1 H, br s, NH), 3.69 (3 H, s, OCH_3), 3.40 (1 H, d, J = 15.7 Hz, CHH), 3.11 (1 H, d, J = 15.7 Hz, CHH), 1.56 (3 H, s, CCH_3); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 174.6 (*C*OO), 165.5 (*C*ONH), 136.1 (q), 132.8, 128.3, 127.9, 127.8, 127.6, 59.0 (q), 53.1 (O*C*H$_3$), 37.9 (*C*H$_2$), 25.9 (*C*H$_3$); IR (ATR): ν_{max} 3195, 1737, 1665. HRMS (ESI$^+$) calcd for $C_{12}H_{14}NO_3$ $(M+H)^+$ 220.0968, found 220.0968.



5b

Methyl 1-oxo-3-propyl-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **5b**: White solid; mp 120-121 ºC; R_f (hexane/EtAcO 1:1): 0.48; ^{1}H NMR (400 MHz; $CDCl_3$; Me_4Si): δ 8.06 (1 H, d, J = 7.6 Hz), 7.46 (1 H, m), 7.36 (1 H, m), 7.22 (1 H, d, J = 7.5 Hz), 6.43 (1 H, br s, NH), 3.71 (3 H, s, OCH_3), 3.37

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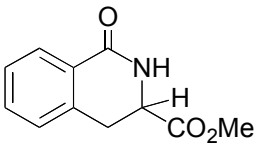
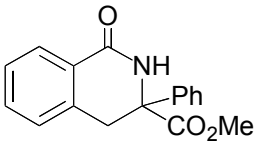
[1] L. Cohenm *J. Biol. Chem.*, 1970, **245**, 5718.

(1 H, d, *J* = 15.7 Hz, C*H*H), 3.13 (1 H, d, *J* = 15.7 Hz, CH*H*), 1.79 (2 H, m), 1.40 (1 H, m), 1.25 (1 H, m), 0.90 (3 H, t, *J* = 7.3 Hz); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 173.3 (*C*OO), 165.1 (*C*ONH), 135.9, 132.6, 128.1, 127.9, 127.8, 127.4, 61.9 (q), 52.8 (O*C*H$_3$), 40.7, 36.6 (*C*H$_2$), 17.0 (*C*H$_2$), 13.9 (*C*H$_3$); IR (ATR): ν_{max} 3209, 1727, 1664, 1606, 1578, 1390. HRMS (ESI$^+$) calcd for $C_{14}H_{18}NO_3$ $(M+H)^+$ 248.1287, found 248.1294.



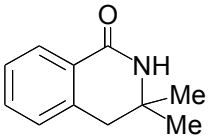
5e

Methyl 1-oxo-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **5e**: Oil;[2] ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.04 (1 H, d, *J* = 7.5 Hz), 7.47 (1 H, m), 7.37 (1 H, m), 7.25 (1 H, m), 6.48 (1 H, br s, NH), 4.41 (1 H, ddd, *J* = 9.9, 5.4, 2.1 Hz, NHC*H*), 3.79 (3 H, s, OC*H*$_3$), 3.33 (1 H, dd, *J* = 15.7, 5.2 Hz, C*H*H), 3.22 (1 H, dd, *J* = 15.7, 9.9 Hz, CH*H*); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 170.8 (*C*OO), 165.1 (*C*ONH), 136.1, 132.5, 129.3, 128.1, 127.5, 127.4, 53.0 (O*C*H$_3$), 52.8 (NH*C*H), 31.1 (*C*H$_2$); IR (ATR): ν_{max} 3271, 1737, 1656, 1546, 1536, 1215, 1176. HRMS (ESI$^+$) calcd for $C_{11}H_{12}NO_3$ $(M+H)^+$ 206.0817, found 206.0803.



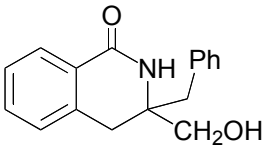
5f

Methyl 1-oxo-3-phenyl-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **5f**: White solid; mp 146-148 ºC; R_f (hexane/EtAcO 7:3): 0.43; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.04 (1 H, d, *J* = 7.7 Hz), 7.25-7.48 (7 H, m), 7.20 (1 H, d, *J* = 7.6 Hz), 6.84 (1 H, br s, NH), 3.75 (3 H, s, OC*H*$_3$), 3.71 (1 H, d, *J* = 15.6 Hz, C*H*H), 3.63 (1 H, d, *J* = 15.6 Hz, CH*H*); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 171.5 (*C*OO), 165.5 (*C*ONH), 139.1, 135.6, 132.9, 128.9, 128.5, 128.2, 128.0, 127.8, 127.7, 127.5, 125.3, 64.3 (q), 53.3 (O*C*H$_3$), 38.0 (*C*H$_2$); IR (ATR): ν_{max} 3174, 3064, 1733, 1691, 1661, 1602, 1464, 1377, 1250. HRMS (ESI$^+$) calcd for $C_{17}H_{16}NO_3$ $(M+H)^+$ 282.1130, found 282.1144.



9

3,3-Dimethyl-3,4-dihydroisoquinolin-1(2*H*)-one, **9**[3]: White solid; mp 146-147 ºC (lit[3] 146-147); ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.06 (1 H, d, *J* = 7.6 Hz), 7.45 (1 H, m), 7.36-7.13 (2 H, m), 6.37 (1 H, br s, NH), 2.92 (2 H, s, C*H*$_2$), 1.32 (6 H, s, C*H*$_3$); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 165.5 (*C*ONH), 137.5, 132.2, 130.4, 127.8, 126.9, 126.2, 52.0 (q), 41.6 (*C*H$_2$), 28.8 (*C*H$_3$); IR (KBr): ν_{max} 3395, 1660. HRMS (ESI$^+$) calcd for $C_{11}H_{14}NO$ $(M+H)^+$ 176.1075, found 176.1069.



10

3-Benzyl-3-(hydroxymethyl)-3,4-dihydroisoquinolin-1(2*H*)-one, **10**: Brownish oil; R_f (hexane/EtAcO 1:1): 0.20; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.05 (1 H, d, *J* = 7.6 Hz), 7.50 (1 H, m), 7.36 (1 H, m), 7.15-7.32 (6 H, m), 6.91 (1 H, br s, N*H*), 3.60 (1 H, d, *J* = 11.2 Hz, C*H*H), 3.52 (1 H, d, *J* = 11.2 Hz, CH*H*), 3.06 (1 H, *J* = 13.6 Hz), 2.95-2.80 (4 H, m); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 165.8 (*C*ONH), 136.7, 135.9, 132.8, 130.4, 128.5, 128.1, 128.0, 127.1, 126.9, 65.7 (q), 58.4, 33.7, 30.9; IR (ATR): ν_{max} 3383, 2927, 1651, 1387, 1253, 1094. HRMS (ESI$^+$) calcd for $C_{17}H_{18}NO_2$ $(M+H)^+$ 268.1338, found 268.1345.

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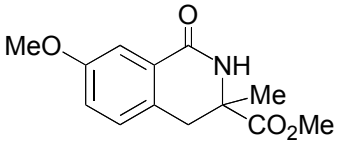
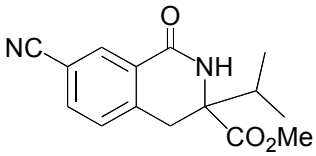
[2] L. Janin, E. Roulland, A. Beurdeley-Thomas, D. Deucadin, C. Monneret and M. Poupon, *J. Chem. Soc.,* 2002, **4**, 529.

[3] D. M. Bayley and C. G. De Grazia, *J. Org. Chem.*, 1970, **35**, 4088.



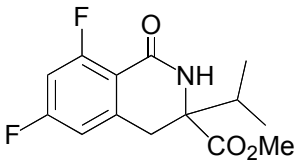
11

Methyl 7-methoxy-3-methyl-1-oxo-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **11**: White solid; mp 171-174 ºC; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.58 (1 H, d, *J* = 2.8 Hz), 7.11 (1 H, d, *J* = 8.4 Hz), 7.01 (1 H, dd, *J* = 8.4, 2.8 Hz), 6.27 (1 H, br s, NH), 3.84 (3 H, s, OC*H*$_3$), 3.70 (3 H, s, OC*H*$_3$), 3.33 (1 H, d, *J* = 15.6 Hz, C*H*H), 3.03 (1 H, d, *J* = 15.6 Hz, CH*H*), 1.53 (3 H, s, CC*H*$_3$); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 174.0 (*C*OO), 165.4 (*C*ONH), 158.9, 130.7, 128.8, 127.9, 120.1, 111.2, 58.9 (q), 55.4 (O*C*H$_3$), 52.7 (O*C*H$_3$), 37.0 (*C*H$_2$), 25.5 (*C*H$_3$); IR (KBr): ν_{max} 3195, 3075, 2951, 1736, 1668, 1493, 1451, 1437, 1382. HRMS (ESI$^+$) calcd for $C_{13}H_{16}NO_4$ $(M+H)^+$ 250.1079, found 250.1068.



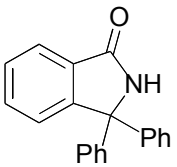
12

Methyl 7-cyano-3-isopropyl-1-oxo-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **12**: White solid; mp 176-178 ºC; R_f (hexane/EtAcO 1:1): 0.43; ^{1}H NMR (400 MHz; $CDCl_3$; Me_4Si): δ 8.44 (1 H, d, *J* = 1.6 Hz), 7.72 (1 H, dd, *J* = 8.0, 1.6 Hz), 7.36 (1 H, d, *J* = 8.0 Hz), 6.29 (1 H, br s, NH), 3.68 (3 H, s, OC*H*$_3$), 3.38 (1 H, d, *J* = 16.4, C*H*H), 3.26 (1 H, d, *J* = 16.4 Hz, CH*H*), 2.18 (1 H, m), 1.00 (3 H, d, *J* = 6.8 Hz), 0.98 (3 H, d, *J* = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 172.7 (*C*OO), 163.7 (*C*ONH), 141.5, 135.4, 132.8, 128.9, 128.7, 117.9 (*C*N), 111.5, 65.3 (q), 52.8 (O*C*H$_3$), 35.0, 33.5, 17.1 (*C*H$_3$), 16.8 (*C*H$_3$); IR (ATR): ν_{max} 3205, 2963, 2229, 1670, 1610, 1433, 1330, 1277, 1189. HRMS (ESI$^+$) calcd for $C_{15}H_{17}N_2O_3$ $(M+H)^+$ 273.1239, found 273.1230.



13

Methyl 6,8-difluoro-3-isopropyl-1-oxo-1,2,3,4-tetrahydroisoquinoline-3-carboxylate, **13**: White solid; mp 138-140 ºC; R_f (hexane/EtAcO 1:1): 0.45; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 6.77 (2 H, m), 6.29 (1 H, br s, NH), 3.68 (3 H, s, OC*H*$_3$), 3.27 (1 H, d, *J* = 16.0 Hz, C*H*H), 3.16 (1 H, d, *J* = 16.0 Hz, CH*H*), 2.12 (1 H, m), 0.99 (3 H, d, *J* = 6.8 Hz), 0.96 (3 H, d, *J* = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 172.5 (*C*OO), 166.0 (*C*ONH), 164.7, 165.0 (dd, $J_{C\text{-}F}$ = 251.2, 13.3 Hz), 163.3 (dd, $J_{C\text{-}F}$ = 274.2, 8.0 Hz), 141.3 (d, $J_{C\text{-}F}$ = 10.4 Hz), 110.9 (dd, $J_{C\text{-}F}$ = 21.9, 4 Hz), 104.4 (t, $J_{C\text{-}F}$ = 25.5 Hz), 64.9 (q), 52.7 (O*C*H$_3$), 34.9, 29.7, 17.2 (*C*H$_3$), 16.9 (*C*H$_3$); IR (ATR): ν_{max} 3226, 3090, 2960, 1724, 1667, 1615, 1307, 1228, 1122. HRMS (ESI$^+$) calcd for $C_{14}H_{16}F_2NO_3$ $(M+H)^+$ 284.1098, found 284.1085.



14

3,3-Diphenylisoindolin-1-one, **14**[4]: White solid; mp 208-210 ºC (lit.[4] 210-211 ºC); ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.88 (1 H, d, *J* = 7.5 Hz), 7.56 (1 H, m), 7.31-7.51 (2 H, m), 7.35-7.20 (9 H, m), 7.15 (1 H, m), 6.65 (1 H, br s, N*H*); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 169.7 (*C*ONH), 150.1, 142.7, 138.1, 132.4, 130.5, 128.7, 128.5, 128.0, 127.9, 127.0, 126.3, 124.5, 124.3, 71.1 (q); IR (ATR): ν_{max} 3291, 1692, 1651, 1446, 1258. HRMS (ESI$^+$) calcd for $C_{20}H_{16}NO$ $(M+H)^+$ 286.1232, found 286.1256.

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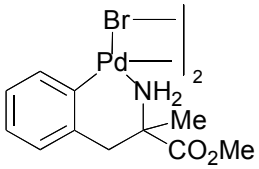
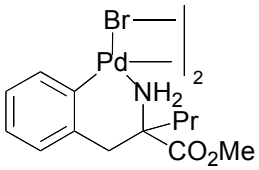
[4] K. Y. Koltunov, G. K. S. Prakash, G. Rasul and G. A. Olah, *Eur. J. Org. Chem.*, 2006, 4861

Analitical data of isolated palladacycles:



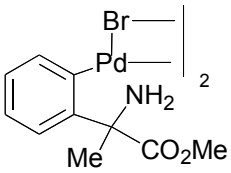
3a

Orange-brown solid; mp 107-109 ºC; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.36 (1 H, d, *J* = 7.5 Hz), 6.90 (1 H, m), 6.82 (1 H, m), 6.75 (1 H, d, *J* = 7.3 Hz), 4.63 (1 H, br d, *J* = 11.0 Hz, NH*H*), 3.71 (3 H, s, OC*H*$_3$), 3.53 (1 H, d, *J* = 13.7, C*H*H), 3.32 (1 H, d, *J* = 13.7, CH*H*), 2.99 (1 H, br d, *J* = 11.0 Hz, N*H*H), 1.56 (3 H, s, CC*H*$_3$); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 174.6 (*C*OO), 137.2, 135.1, 127.7, 125.9, 124.7, 56.2, 53.5 (O*C*H$_3$), 52.2 (*C*H$_2$), 25.1 (*C*H$_3$); IR (KBr): ν_{max} 3325, 3238, 1730, 1573, 1560, 1212, 1125. HRMS (MALDI-TOF) calcd for $C_{22}H_{28}BrN_2O_4Pd_2$ $(M\text{-}Br)^+$ 674,9302, found 674.4302; Anal. calcd for $C_{22}H_{28}Br_2N_2O_4Pd_2$: C, 34.90; H, 3.73; N, 3.70. Found: C, 35.12; H, 3.43; N, 3.62.



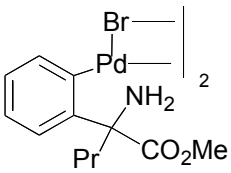
3b

Brownish solid; mp 110-112 ºC; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.38 (1 H, d, *J* = 7.8 Hz), 6.90 (1 H, m), 6.83 (1 H, m), 6.75 (1 H, dd, *J* = 7.3, 1.8 Hz), 4.46 (1 H, br d, *J* = 11.3 Hz, NH*H*), 3.68 (3 H, s, OC*H*$_3$), 3.59 (1 H, d, *J* = 13.8, C*H*H), 3.25 (1 H, d, *J* = 13.8, CH*H*), 3.14 (1 H, br d, *J* = 11.3 Hz, N*H*H), 1.98 (1 H, m), 1.84 (1 H, m), 1.43 (1 H, m), 1.24 (1 H, m), 0.95 (3 H, t, *J* = 7.2 Hz, C*H*$_3$); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 173.9 (*C*OO), 136.8, 135.0, 127.5, 127.4, 125.6, 124.6, 59.7 (q), 53.2 (O*C*H$_3$), 51.5 (*C*H$_2$), 40.0 (*C*H$_2$), 17.7 (*C*H$_2$), 14.0 (*C*H$_3$); IR (KBr): ν_{max} 3302, 3237, 1726, 1571, 1558, 1435, 1230. HRMS (MALDI-TOF) calcd for $C_{26}H_{36}BrN_2O_4Pd_2$ $(M\text{-}Br)^+$ 730,9928, found 730.9899; Anal. calcd for $C_{26}H_{36}Br_2N_2O_4Pd_2$: C, 38.40; H, 4.46; N, 3.44. Found: C, 38.67; H, 4.33; N, 3.60.



4a

Brownish solid; mp 207-209 ºC; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.38 (1 H, d, *J* = 7.5 Hz), 7.0 (1 H, m), 6.90 (2 H, m), 5.15 (1 H, br d, *J* = 9.6 Hz, NH*H*), 3.85 (3 H, s, OC*H*$_3$), 3.52 (1 H, br d, *J* = 9.6 Hz, N*H*H), 1.56 (3 H, s, CC*H*$_3$); ^{13}C NMR ($CDCl_3$ + drop of pyridine-d_5, 101 MHz; Me_4Si): δ 172.8 (*C*OO), 152.6, 149.5, 132.2, 126.8, 124.7, 123.5, 71.9 (q), 53.3 (O*C*H$_3$), 28.3 (*C*H$_3$); IR (KBr): ν_{max} 3286, 3236, 1724, 1550, 1432, 1215, 1143. HRMS (MALDI-TOF) calcd for $C_{20}H_{24}BrN_2O_4Pd_2$ $(M\text{-}Br)^+$ 646,8989, found 646.8977; Anal. calcd for $C_{20}H_{24}Br_2N_2O_4Pd_2$: C, 32.95; H, 3.32; N, 3.84. Found: C, 32.65; H, 3.55; N, 3.65.

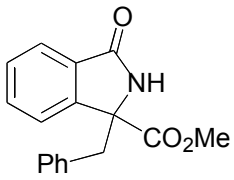


4b

Brownish solid; mp 122-124 ºC; ^{1}H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.38 (1 H, d, *J* = 7.8 Hz), 6.98 (2 H, m), 6.88 (1 H, m), 5.10 (1 H, br d, *J* = 10.3 Hz, NH*H*), 3.83 (3 H, s, OC*H*$_3$), 3.63 (1 H, br d, *J* = 10.3 Hz, N*H*H), 2.18 (2 H, m), 1.45 (1 H, m, CH*H*), 1.34 (1 H, m, C*H*H), 0.99 (3 H, t, *J* = 7.2 Hz, C*H*$_3$); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 172.4 (*C*OO), 150.6, 144.9, 135.6, 127.1, 124.8, 123.9, 74.7 (q), 53.5 (O*C*H$_3$), 42.7 (*C*H$_2$), 17.3 (*C*H$_2$), 14.0; IR (KBr): ν_{max} 3296, 3252, 1729, 1571, 1433, 1210. HRMS (MALDI-TOF) calcd for $C_{24}H_{32}BrN_2O_4Pd_2$ $(M\text{-}Br)^+$ 702,9615, found 702.9609; Anal. calcd for $C_{24}H_{32}Br_2N_2O_4Pd_2$: C, 36.71; H, 4.11; N, 3.57. Found: C, 36.65; H, 4.05; N, 3.75.

Stepwise preparation of lactam 6c: A mixture of methyl 2-amino-2,3-diphenylpropanoate **9** (500 mg, 1.96 mmol) and palladium acetate (439 mg, 1.96 mmol) in 40 mL of toluene was stirred at 80 ºC for 22 h. The solvent was removed under vacuum and the residue was treated with lithium bromide (213 mg, 2.45 mmol) in acetone (40 mL) for 1 h at rt. The suspension was filtered to obtain 783 mg (91%) of a 86:14 mixture of 5-membered and 6-membered palladacycles (^{1}H NMR of the crude). The crude was purified by flash chromatography (hexane/EtAcO 8:2) to afford 5-membered palladacycle **16** (625 mg, 72%). Compound **16**: Brownish solid; mp 212–214 ºC; R_f (hexane/EtAcO 8:2): 0.32; ^{1}H NMR (400 MHz; $CDCl_3$; Me_4Si): δ 7.44 (3 H, m), 7.33 (3 H, m), 7.18 (1 H, dd, *J* = 7.7, 1.2 Hz), 7.04 (1 H, m), 6.94 (1 H, m), 4.75 (1 H, br d, *J* = 10.3, NH*H*), 3.86 (3 H, s, OC*H*$_3$), 3.72 (1 H, d, *J* = 14.1 Hz, C*H*H), 3.61 (1 H, d, *J* = 14.1 Hz, CH*H*), 3.67 (1H, br d, *J* = 10.3, N*H*H); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 171.1, 136.2, 133,9, 130.2, 130.0, 129.5, 129.4, 128.3, 127.2, 125.0, 123.0, 74.9 (q), 53.3 (O*C*H$_3$), 47.4 (*C*H$_2$); IR (KBr): ν_{max} 3296, 3256, 1730. HRMS (Maldi-TOF) calcd for $C_{32}H_{32}BrN_2O_4Pd_2$ $(M\text{-}Br)^+$ 798.9609, found 798.9626.

A stirred solution of palladacycle **16** (110 mg, 0.125 mmol) in AcOH (25 mL) was gently refluxed in an oil bath at 120 ºC in a 100-mL three-necked flask provided with a reflux condenser closed by a toy balloon under atmosphere of carbon monoxide (overal gas volume ~150 mL) for 3 h. The reaction mixture was cooled, a filtered through a thin pad of Celite®. The volatiles were removed under vacuum to obtain a solid corresponding to a 93:7 mixture (65 mg, 93%) of **6c** and **5f** . The residue was purified by flash chromatography to afford **6c** (61 mg, 86%).



6c

Methyl 1-benzyl-3-oxoisoindoline-1-carboxylate, **6c**: White solid; mp 140-142 ºC; R_f (hexane/EtAcO 8:2): 0.32; ^{1}H NMR (400 MHz; $CDCl_3$; Me_4Si): δ 7.82 (1 H, d, *J* = 7.7 Hz), 7.79 (1 H, d, *J* = 7.5 Hz), 7.64 (1 H, m), 7.52 (1 H, m), 7.27 (3 H, m), 7.12 (2 H, m), 6.53 (1 H, br s, NH), 3.77 (1 H, d, *J* = 13.4 Hz, C*H*H), 3.71 (3 H, s, OC*H*$_3$), 2.96 (1 H, d, *J* = 13.4 Hz, CH*H*); ^{13}C NMR ($CDCl_3$, 101 MHz; Me_4Si): δ 170.6 (CO), 169.5 (CO), 144.9 (q), 134.5, 132.5, 130.8, 129.8, 129.5, 128.7, 127.7, 124.0, 123.3, 68.8 (q), 53.0 (O*C*H$_3$), 45.0 (*C*H$_2$); IR (ATR): ν_{max} 3219, 1733, 1695, 1611, 1250. HRMS (ESI^+) calcd for $C_{17}H_{16}NO_3$ $(M+H)^+$ 282.1130, found 282.1128.