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SUPPORTING INFORMATION

Perhydroquinolylbenzamides as Novel Inhibitors of 11β-Hydroxysteroid Dehydrogenase Type 1

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Contents

Experimental

N-[4-(Bicyclo[2.2.1]hept-2-yl-methylcarbamoyl)-phenyl]-2,4-dichlorobenzamide (9k). Prepared in 100% yield from **16** by Method A (product is a foam); ^{1}H NMR ($CDCl_3$): δ 8.52 (s, 1H), 7.71-7.57 (m, 3H), 7.46 (s, 1H), 7.33 (t, 3H), 5.30 (s, 2H), 2.93, (s 3H), 2.30 (d, J = 12.81 Hz, 1H), 1.74-0.98 (m, 9H); MS (ES^+) m/z 417 $(M+1)^+$. Anal. ($C_{22}H_{22}Cl_2N_2O_2$) C, H, N.

2,4-Dichloro-N-{4-[methyl-((1S*,2S*)-2-methylcyclohexyl)-carbamoyl]-phenyl}-benzamide (9l). Prepared in 88% yield from **20** by Method A (product is a foam); IR (KBr), ν (cm^{-1}): 3427, 3254, 1604; ^{1}H NMR ($CDCl_3$): δ 8.13 (s, 1H), 7.74 (d, J = 8.09 Hz, 1H), 7.69-7.61 (m, 2H), 7.49 (s, 1H), 7.42-7.31 (m, 3H), 2.92 (s, 2H), 2.79 (s, 1H), 1.89-1.35 (m, 8H), 1.27-1.06 (m, 2H), 0.94 (d, J = 6.25 Hz, 1H), 0.78 (d, J = 6.62 Hz, 2H); MS (ES^+) m/z 419 $(M+1)^+$.

2,4-Dichloro-N-[4-((4aS*,8aS*)-octahydrobenzo[1,4]oxazine-4-carbonyl)-phenyl]-benzamide (9n). Prepared in 81% yield from **66** by Method A, mp = 227 – 230 °C; ^{1}H NMR (DMSO-d_6): δ 10.74 (s, 1H), 7.80-7.72 (m, 3H), 7.68-7.54 (m, 2H), 7.46 (d, J = 8.29 Hz, 2H), 3.83-3.56 (m, 4H), 3.49-3.28 (m, 2H), 2.38-2.28 (m 1H), 1.93-1.81 (m, 1H), 1.75-1.58 (m, 2H), 1.48-1.15 (m, 4H); MS (ES$^+$) m/z 433 $(M+1)^+$.

4-Fluoro-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (25). Prepared in 79% yield from **23** and 4-fluorobenzoyl chloride according to Method A (step c), mp = 251 – 253 °C; IR (KBr), ν (cm^{-1}): 3434, 3294, 1674, 1602; ^{1}H NMR (DMSO-d_6): δ 10.41 (s, 1H), 8.10-8.00 (m, 2H), 7.82 (d, J = 8.67 Hz, 2H), 7.44-7.32 (m, 4H), 3.46-3.25 (m, 3H), 2.10 (d, J = 12.44 Hz, 1H), 1.79-0.95 (m, 12H); MS (ES$^+$) m/z 381 $(M+1)^+$; Anal. ($C_{23}H_{25}N_2O_2F$) C, H, N.

4-Methoxy-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (27). Prepared in 84% yield from **23** and 4-methoxybenzoyl chloride according to Method A (step c), mp = 243 – 246 °C; ^{1}H NMR (DMSO-d_6): δ 10.23 (s, 1H), 7.96 (d, J = 8.83 Hz, 2H), 7.82 (d, J = 8.82 Hz, 2H), 7.34 (d, J = 8.83 Hz, 4H), 7.08 (d, J = 8.82 Hz, 2H), 3.84 (s, 3H), 3.44-3.27 (m, 3H), 2.15-2.04 (m, 1H), 1.78-0.96 (m, 12H); MS (ES$^+$) m/z 393 $(M+1)^+$;Anal. ($C_{24}H_{28}N_2O_3$) C, H, N.

4-Methyl-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (28). Prepared in 19% yield from **23** and *p*-toluoyl chloride according to Method A (step c), mp = 185 – 190 °C; ^{1}H NMR (DMSO-d_6): δ 10.30 (s, 1H), 7.91-7.80 (m, 4H), 7.34 (d, J = 8.09 Hz, 4H), 3.47-3.26 (m, 3H), 2.39 (s, 3H), 2.15-2.04 (m, 1H), 1.78-0.96 (m, 12H); MS (ES$^+$) m/z 377 $(M+1)^+$; Anal. ($C_{24}H_{28}N_2O_4 \cdot 0.05\ CH_2Cl_2$) C, H, N.

4-Cyano-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (29). Prepared in 23% yield from **23** and 4-cyanobenzoyl chloride according to Method A (step c), mp = 265 – 266 °C; ^{1}H NMR ($CDCl_3$): δ 9.09 (s, 1H), 8.13 (d, J = 8.29 Hz, 2H), 7.78 (d, J = 8.29 Hz, 2H), 7.41 (d, J =

8.29 Hz, 2H), 7.23 (d, J = 8.67 Hz, 2H), 3.53-3.26 (m, 3H), 2.28-2.14 (m, 1H), 1.91-1.02 (m, 12H); MS (ES^+) m/z 388 $(M+1)^+$; Anal. ($C_{24}H_{25}N_3O_3$) C, H, N.

N-[4-((4aR*,8aS*)-Octahydro-quinoline-1-carbonyl)-phenyl]-terephthalamic acid (30). Methyl ester prepared in 64% yield from **23** and 4-carbomethoxybenzoyl chloride according to Method A (step c), mp = 224 – 226 °C; IR (KBr), ν (cm^{-1}): 3331, 1722, 1674; ^1H NMR (DMSO-d_6): δ 10.23 (s, 1H), 7.96 (d, J = 8.83 Hz, 2H), 7.81 (d, J = 8.82 Hz, 2H), 7.34 (d, J = 8.83 Hz, 2H), 7.07 (d, J = 8.82 Hz, 2H), 3.84 (s, 3H), 3.44-3.27 (m, 3H), 2.15-2.04 (m, 1H), 1.78-0.95 (m, 12H); MS (ES^+) m/z 421 $(M+1)^+$; Anal. ($C_{25}H_{28}N_2O_4$) C, H, N.

To a suspension of 0.78 mmol of the above ester in 15 mL of EtOH was added 0.8 mL of 1.0 N NaOH. The mixture was stirred at 50 °C for 5 h then the solvent was removed under reduced pressure. The resulting solid was stirred with 2 N HCl for 30 min then the solid was filtered, washed with H_2O and dried to give 272 mg (86%) of **30**, mp = >265 °C; ^1H NMR (DMSO-d_6): δ 7.96-7.78 (m, 6H), 7.33 (d, J = 8.45 Hz, 2H), 3.50-3.22 (m, 3H), 2.15-2.04 (m, 1H), 1.79-0.94 (m, 12H); MS (ES^+) m/z 407 $(M+1)^+$; Anal. ($C_{24}H_{26}N_2O_4 \cdot 0.25\ H_2O$) C, H, N.

4-Methanesulfonyl-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (31). Prepared in 53% yield from **23** and 4-methanesulfonylbenzoyl chloride[62] according to Method A (step c), mp = 249 – 252 °C; IR (KBr), ν (cm^{-1}): 3283, 1667, 1585, 1154; ^1H NMR (DMSO-d_6): δ 10.65 (s, 1H), 8.18 (d, J = 8.46 Hz, 2H), 8.10 (d, J = 8.46 Hz, 2H), 7.84 (d, J = 8.45 Hz, 2H), 7.39 (d, J = 8.46 Hz, 2H), 3.43-3.25 (m, 3H), 3.30 (s, 3H), 2.15-2.04 (m, 1H), 1.79-0.96 (m, 12H); MS (ES^+) m/z 441 $(M+1)^+$; Anal. ($C_{24}H_{28}N_2O_4S$) C, H, N.

4-Dipropylsulfamoyl-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (33). Prepared in 75% yield from **23** and 4-dipropylsulfamoylbenzoyl chloride[63] according to Method A (step c), mp = 188 – 190 °C; ^1H NMR ($CDCl_3$): δ 9.12 (s, 1H), 8.07 (d, J = 8.45 Hz, 2H), 7.82 (d, J = 8.46

Hz, 2H), 7.56 (d, J = 8.45 Hz, 2H), 7.27 (d, J = 8.46 Hz, 2H), 3.56-3.30 (m, 3H), 3.17-3.04 (m, 4H), 2.32-2.22 (m, 1H), 1.87 –1.02 (m, 16H), 0.95-0.83 (m, 6H); MS (ES^+) m/z 526 $(M+1)^+$; Anal. ($C_{29}H_{39}N_3O_4S$) C, H, N.

3-Fluoro-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (34). Prepared in 93% yield from **23** and 3-fluorobenzoyl chloride according to Method A (step c), mp = 183 – 186 °C; ^1H NMR ($CDCl_3$): δ 8.79 (s, 1H), 7.80-7.70 (m, 2H), 7.48 (d, J = 8.09 Hz, 2H), 7.48-7.42 (m, 1H), 7.30-7.20 (m, 3H), 3.58-3.25 (m, 3H), 2.31-2.19 (m, 1H), 1.86 –1.01 (m, 12H); MS (ES^+) m/z 381 $(M+1)^+$; Anal. ($C_{23}H_{25}N_2O_2F$) C, H, N.

2,4-Dichloro-N-[2-methyl-4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (41). Prepared in 84% yield from **56** and 2,4-dichlorobenzoyl chloride according to Method A (step c), mp = 209 – 211 °C; IR (KBr), ν (cm^{-1}): 3282, 1690, 1603; ^1H NMR (DMSO-d_6): δ 10.09 (s, 1H), 7.78 (s, 1H), 7.67 (d, J = 8.09 Hz, 1H), 7.61-7.50 (m, 2H), 7.28-7.17 (m, 2H), 3.46-3.22 (m, 3H), 2.30 (s, 3H), 2.16-2.06 (m,1H), 1.79-0.96 (m, 12H); MS (ES^+) m/z 445 $(M+1)^+$; Anal. ($C_{24}H_{26}N_2O_2Cl_2$) C, H, N.

2,4-Dichloro-N-[2-methoxy-4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-benzamide (42). Prepared in 83% yield from 12b and 21 according to Method B, mp = 184 – 186 °C; IR (KBr), ν (cm^{-1}): 3418, 1675, 1608; ^1H NMR ($CDCl_3$): δ 8.65 (s, 1H), 8.50 (d, J = 8.82 Hz, 1H), 7.76 (d, J = 8.09 Hz, 1H), 7.49 (d, J = 2.20 Hz, 1H), 7.41-7.35 (m, 1H), 7.07-7.00 (m, 2H), 3.92 (s, 3H), 3.58-3.32 (m, 3H), 2.35-2.25 (m,1H), 1.87-1.01 (m, 12H); MS (ES^+) m/z 461 $(M+1)^+$; Anal. ($C_{24}H_{26}N_2O_3Cl_2$) C, H, N.

2,4-Dichloro-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-naphthalen-1-yl]-benzamide (49). Prepared in 59% yield from **62** and 2,4-dichlorobenzoyl chloride according to Method A (step c), mp = 231 – 233 °C; IR (KBr), ν (cm^{-1}): 3430, 1683, 1606; ^1H NMR (DMSO-d_6): δ 10.68 (s, 1H),

8.26-8.17 (m, 1H), 7.87-7.34 (m, 8H), 3.61-3.11 (m, 3H), 2.31-2.14 (m,1H), 2.05-0.95 (m, 12H); MS (ES^+) m/z 481 $(M+1)^+$; Anal. ($C_{27}H_{26}N_2O_2Cl_2$) C, H, N.

N-[4-((4aR*,8aS*)-Octahydroquinoline-1-carbonyl)-phenyl]-isonicotinamide (50). Prepared in 75% yield from **23** and isonicotinoyl chloride according to Method A (step c), mp = 220 – 228 °C; ^1H NMR ($CDCl_3$): δ 9.50 (s, 1H), 8.78 (d, J = 5.52 Hz, 2H), 7.90 (d, J = 5.52 Hz, 2H), 7.41 (d, J = 8.46 Hz, 2H), 7.17 (d, J = 8.09 Hz, 2H), 3.57-3.28 (m, 3H), 2.30-2.18 (m, 1H), 1.90-1.01 (m, 12H); MS (ES^+) m/z 364 $(M+1)^+$; Anal. ($C_{22}H_{25}N_3O_2$) C, H, N.

Furan-2-carboxylic acid [4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-amide (51). Prepared in 67% yield from **23** and 2-furoic acid according to Method A (similar to example **32**), mp = 229 – 232 °C; ^1H NMR (DMSO-d_6): δ 10.33 (s, 1H), 7.96 (m, 1H), 7.80 (d, J = 8.45 Hz, 2H), 7.40-7.31 (m, 3H), 6.75-6.69 (m, 1H), 3.48-3.26 (m, 3H), 2.14-2.04 (m, 1H), 1.79-0.94 (m, 12H); MS (ES^+) m/z 353 $(M+1)^+$; Anal. ($C_{21}H_{24}N_2O_3$) C, H, N.

1-Methyl-1H-pyrrole-2-carboxylic acid [4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-amide (52). Prepared in 32% yield from **23** and 1-methyl-1H-pyrrole-2-carboxylic acid according to Method A (similar to example **32**), mp = 169 – 171 °C; IR (KBr), ν (cm^{-1}): 3276, 3087, 3063, 3032, 1725, 1687, 1638; ^1H NMR ($CDCl_3$): δ 7.77 (s, broad, 1H), 7.55 (d, J = 8.45 Hz, 2H), 7.37 (d, J = 8.45 Hz, 2H), 6.83-6.75 (m, 2H), 6.18-6.13 (m, 1H), 3.98 (s, 3H), 3.56-3.32 (m, 3H), 2.35-2.22 (m, 1H), 1.86-0.99 (m, 12H); MS (ES^+) m/z 366 $(M+1)^+$; Anal. ($C_{22}H_{27}N_3O_2$) C, H, N.

Cyclohexanecarboxylic acid [4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-amide (53). Prepared in 35% yield from **23** and cyclohexanecarbonyl chloride according to Method A (step c), mp = 205 – 206 °C; ^1H NMR ($CDCl_3$): δ 7.51 (s, broad, 1H), 7.48 (d, J = 8.29 Hz, 2H), 7.31 (d, J = 8.29 Hz, 2H), 3.56-3.28 (m, 3H), 2.35-2.18 (m, 2H), 2.02-1.00 (m, 22H); MS (ES^+) m/z 369 $(M+1)^+$; Anal. ($C_{27}H_{32}N_2O_2$) C, H, N.

N-[4-((4aR*,8aS*)-Octahydroquinoline-1-carbonyl)-phenyl]-isobutyramide (54). Prepared in 44% yield from **23** and isobutyryl chloride according to Method A (step c), mp = 170 – 171 °C; ^{1}H NMR ($CDCl_3$): δ 7.52 (d, J = 8.29 Hz, 2H), 7.49 (s, broad, 1H), 7.34 (d, J = 8.29 Hz, 2H), 3.56-3.30 (m, 3H), 2.60-2.46 (m, 1H), 2.31-2.22 (m, 1H), 1.83-1.52 (m, 5H), 1.49-1.02 (m, 7H), 1.25 (d, J = 6.79 Hz, 6H); MS (ES^+) m/z 329 $(M+1)^+$; Anal. ($C_{20}H_{28}N_2O_2$) C, H, N.

3,7-Dimethyl-bicyclo[3.3.1]nonane-1-carboxylic acid [4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-amide (55). Prepared in 80% yield from **23** and 1-adamantanecarbonyl chloride according to Method A (step c), mp = 239 – 243 °C; IR (KBr), ν (cm^{-1}): 3298, 1669; ^{1}H NMR ($CDCl_3$): δ 7.54 (d, J = 8.45 Hz, 2H), 7.40 (s, 1H), 7.37 (d, J = 8.45 Hz, 2H), 3.58-3.29 (m, 3H), 2.34-2.22 (m, 1H), 2.17-2.07 (m, 3H), 1.97 (s, 6H), 1.86-1.00 (m, 18H); MS (ES^+) m/z 421 $(M+1)^+$; Anal. ($C_{27}H_{36}N_2O_2$) C, H, N.

4-Fluoro-N-[4-((4aR*,8aS*)-2-octahydroquinolin-1-yl-2-oxo-ethyl)-phenyl]-benzamide (76). Prepared in 33% yield from **75** and 4-fluorobenzoyl chloride according to Method A (step c), mp = 129 – 130 °C; ^{1}H NMR ($CDCl_3$): δ 8.06 (s, broad, 1H), 7.96-7.87 (m, 2H), 7.55 (d, J = 8.29 Hz, 2H), 7.22 (d, J = 8.66 Hz, 2H), 7.15 (t, 2H), 3.74-3.57 (m, 1H), 3.67 (s, 2H), 3.37 (t, broad, 1H), 3.23-3.05 (m, 1H), 2.19-2.08 (m, 1H), 1.83 –1.16 (m, 10H), 1.11-0.94 (m, 2H); MS (ES^+) m/z 395 $(M+1)^+$; Anal. ($C_{24}H_{27}N_2O_2F$) C, H, N.

2-(4-Fluoro-phenyl)-N-[4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-phenyl]-acetamide (77). Prepared in 38% yield from **23** and 4-fluorophenylacetyl chloride according to Method A (step c), mp = 173 – 174 °C; IR (KBr), ν (cm^{-1}): 3324, 1697, 1603; ^{1}H NMR ($CDCl_3$): δ 7.74 (s, broad, 1H), 7.38 (d, J = 8.45 Hz, 2H), 7.34-7.26 (m, 2H), 7.27 (d, J = 8.09 Hz, 2H), 7.07 (t, 2H), 3.69 (s, 2H), 3.54-3.27 (m, 3H), 2.33-2.22 (m, 1H), 1.84 –1.51 (m, 7H), 1.47-1.00 (m, 5H); MS (ES^+) m/z 395 $(M+1)^+$; Anal. ($C_{24}H_{27}N_2O_2F$) C, H, N.

N-Cyclohexyl-4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-benzamide (93). Prepared using multiparallel conditions similar to Method B using **91** and cyclohexylamine; MS (ES^+) m/z 369 $(M+1)^+$.

N-Isopropyl-4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-benzamide (94). Prepared in 34% yield from **91** and isopropylamine according to Method D, mp = 156 – 156 °C; ^1H NMR ($CDCl_3$): δ 7. 76 (d, J = 8.29 Hz, 2H), 7.42 (d, J = 8.29 Hz, 2H), 5.97 (d, broad, J = 7.53 Hz, 1H), 4.36 – 4.21 (m, 1H), 3.55 – 3.44 (m, 1H), 3.37 – 3.28 (m, 2H), 2.32 – 2.20 (m, 1H), 1.84 – 1.02 (m, 12H), 1.27 (d, J = 6.78 Hz, 6H); MS (ES^+) m/z 329 $(M+1)^+$. Anal. ($C_{20}H_{28}N_2O_2$) C, H, N.

N-Isobutyl-4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-benzamide (95). Prepared in 45% yield from **91** and isobutylamine according to Method D, mp = 134 – 135 °C; ^1H NMR ($CDCl_3$): δ 7. 76 (d, J = 8.29 Hz, 2H), 7.42 (d, J = 8.29 Hz, 2H), 6.28 (m, 1H), 3.56 – 3.43 (m, 1H), 3.38 – 3.25 (m, 4H), 2.33 – 2.20 (m, 1H), 1.99 – 1.03 (m, 13H), 0.99 (d, J = 6.78 Hz, 6H); MS (ES^+) m/z 343 $(M+1)^+$; Anal. ($C_{21}H_{30}N_2O_2$) C, H, N.

N-Benzyl-4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-benzamide (96). Prepared in 19% yield from **91** and benzylamine according to Method D, mp = 140 – 147 °C; ^1H NMR ($CDCl_3$): δ 7. 80 (d, J = 8.09 Hz, 2H), 7.43 (d, J = 8.09 Hz, 2H), 7.40 – 7.27 (m, 5H), 6.42 (s, broad, 1H), 4.66 (d, J = 5.51 Hz, 2H), 3.56 – 3.43 (m, 1H), 3.40 – 3.28 (m, 2H), 2.32 – 2.23 (m, 1H), 1.86 – 1.01 (m, 12H); MS (ES^+) m/z 377 $(M+1)^+$; Anal. ($C_{24}H_{28}N_2O_2 \cdot 0.25\ H_2O$) C, H, N.

4-((4aR*,8aS*)-Octahydroquinoline-1-carbonyl)-N-pyridin-3-ylmethyl-benzamide (97). Prepared in 46% yield from **90** and 3-(aminomethyl)pyridine according to Method E, mp = 122 – 123 °C; ^1H NMR ($CDCl_3$): δ 8.60 – 8.50 (m, 2H), 7.76 (d, J = 8.29 Hz, 2H), 7.71 (m, 1H), 7.32 (d, J = 8.29 Hz, 2H), 7.29 – 7.21 (m, 2H), 4.65 (d, J = 6.02 Hz, 2H), 3.52 – 3.40 (m, 1H), 3.36 – 3.25 (m, 2H), 2.30 – 2.17 (m, 1H), 1.86 – 1.00 (m, 12H); MS (ES^+) m/z 378 $(M+1)^+$; Anal. ($C_{23}H_{27}N_3O_2$) C, H, N.

N-(4-Fluorophenyl)-4-((4aR*,8aS*)-octahydroquinoline-1-carbonyl)-benzamide (98). Prepared in 37% yield from **91** and 4-fluoroaniline according to Method D, mp = 196 – 198 °C; ^{1}H NMR ($CDCl_3$): δ 8.77 (s, 1H), 7. 81 (d, J = 7.92 Hz, 2H), 7.77 – 7.68 (m, 2H), 7.29 (d, J = 8.29 Hz, 2H), 7.07 (t, 2H), 3.55 – 3.44 (m, 1H), 3.35 – 3.25 (m, 2H), 2.30 – 2.21 (m, 1H), 1.86 – 1.00 (m, 12H); MS (ES^+) m/z 381 $(M+1)^+$; Anal. ($C_{23}H_{27}N_2O_2F$) C, H, N.

(4aR*,8aS*)-Octahydroquinolin-1-yl-[4-(piperidine-1-carbonyl)-phenyl]-methanone (99). Prepared in 46% yield from **90** and piperidine according to Method E, mp = 136 – 137 °C; ^{1}H NMR ($CDCl_3$): δ 7.41 (s, 4H), 3.71 (m, 2H, broad), 3.52 (m, 1H), 3.40 – 3.26 (m, 4H), 2.28 (m, 1H), 1.84 – 1.02 (m, 18H); MS (ES^+) m/z 355 $(M+1)^+$; Anal. ($C_{22}H_{30}N_2O_2$) C, H, N.

[4-(3,4-Dihydro-1H-isoquinoline-2-carbonyl)-phenyl]-(4aR*,8aS*)-octahydroquinolin-1-yl-methanone (100). Prepared in 78% yield from **90** and 1,2,3,4-tetrahydroisoquinoline according to Method E, mp = 143 – 148 °C; ^{1}H NMR ($CDCl_3$): δ 7.46 (s, 4H), 7.25 – 7.10 (m, 4H), 4.90 (s, broad, 1H), 4.56 (s, broad, 1H), 4.00 (s, broad, 1H), 3.70 – 3.58 (m, 1H), 3.58 – 3.46 (m, 1H), 3.43 – 3.30 (m, 2H), 3.05 – 2.80 (m, 2H), 2.34 – 2.23 (m, 1H), 1.89 – 1.02 (m, 12H); MS (ES^+) m/z 403 $(M+1)^+$; Anal. ($C_{26}H_{30}N_2O_2$) C, H, N.

CHN Analyses for HSD Compounds

No.	Formula	Calcd.			Found		
		C	H	N	C	H	N
9a	$C_{22}H_{26}N_2O_5$+0.5 H_2O	64.85	6.68	6.87	65.07	6.59	6.79
9c	$C_{19}H_{18}Cl_2N_2O_2$+0.3 H_2O	59.64	4.90	7.32	59.31	4.52	7.19
9d	$C_{20}H_{14}Cl_2N_2O_2$	61.39	5.15	7.16	61.20	5.13	7.10
9e	$C_{21}H_{22}Cl_2N_2O_2$	62.28	5.77	6.91	62.05	5.50	6.89
9g	$C_{21}H_{22}Cl_2N_2O_2$	62.23	5.47	6.91	62.18	5.50	6.84
9h	$C_{21}H_{16}Cl_2N_2O_2$+0.4 H_2O	62.05	4.17	6.89	61.74	3.89	6.81
9i	$C_{26}H_{30}Cl_2N_2O_2$+0.3 CH_2Cl_2	63.31	6.18	5.61	63.11	5.78	5.57
9k	$C_{22}H_{22}Cl_2N_2O_2$	63.32	5.31	6.71	63.05	5.27	6.55
9l	$C_{22}H_{24}Cl_2N_2O_2$+0.4 H_2O	61.95	5.86	6.68	61.79	5.83	6.34
9m	$C_{23}H_{24}Cl_2N_2O_2$	64.04	5.61	6.49	63.68	5.67	6.37
9n	$C_{22}H_{22}Cl_2N_2O_3$+0.4 H_2O	59.98	5.22	6.36	59.64	5.03	6.24
11	$C_{16}H_{13}Cl_2NO_3$	56.82	3.87	4.14	56.72	3.95	4.00
12a	$C_{14}H_8Cl_3NO_2$	51.18	2.45	4.26	50.87	2.61	4.58
14	$C_{14}H_{16}N_2O_3$	64.60	6.20	10.76	64.53	5.92	10.73
16	$C_{15}H_{20}N_2O$+0.4 H_2O	71.62	8.33	11.14	71.35	7.99	10.96
18	$C_{14}H_{18}N_2O_3$	64.11	6.92	10.68	64.22	6.86	10.62
19	$C_{15}H_{20}N_2O_3$	65.20	7.29	10.14	65.05	7.12	9.98
22	$C_{16}H_{20}N_2O_3$	66.65	6.99	9.72	66.87	6.95	9.80
24	$C_{23}H_{26}N_2O_2$+0.4 H_2O	74.73	7.31	7.58	74.56	7.31	7.58
25	$C_{23}H_{25}FN_2O_2$	72.61	6.62	7.36	72.73	6.64	7.35
26	$C_{23}H_{25}Cl\ N_2O_2$+0.5 H_2O	68.66	6.41	6.96	68.66	6.19	7.08
27	$C_{24}H_{28}N_2O_3$	73.44	7.19	7.14	73.09	7.08	7.00
28	$C_{24}H_{28}N_2O_2$+.05 CH_2Cl_2	75.87	7.04	7.36	75.57	7.02	7.34
29	$C_{24}H_{25}N_3O_2$	74.39	6.50	10.84	74.11	6.50	10.81
30	$C_{24}H_{26}N_2O_4$+0.2 H_2O	70.29	6.49	6.83	69.96	6.50	6.77
31	$C_{24}H_{28}N_2O_4S$	65.43	6.41	6.36	65.36	6.37	6.31
32	$C_{23}H_{27}N_3O_4S$	62.56	6.16	9.52	62.38	6.08	9.43
33	$C_{29}H_{39}N_3O_4S$	66.26	7.48	7.99	66.12	7.43	7.68
34	$C_{23}H_{25}FN_2O_2$	72.61	6.62	7.36	72.25	6.51	7.32
35	$C_{23}H_{25}FN_2O_2$	72.61	6.62	7.36	72.30	6.66	7.18
37	$C_{23}H_{25}ClN_2O_2$	69.60	6.35	7.06	69.49	6.28	6.92
38	$C_{27}H_{36}N_2O_2$	69.33	6.07	7.03	69.12	6.15	6.87
39	$C_{24}H_{27}ClN_2O_2$	70.15	6.62	6.82	69.86	6.43	6.62
40	$C_{24}H_{27}ClN_2O_3$	67.52	6.37	6.56	67.38	6.16	6.35
41	$C_{24}H_{26}Cl_2N_2O_2$	64.72	5.88	6.29	64.97	5.63	6.15
42	$C_{24}H_{26}Cl_2N_2O_3$	62.48	5.68	6.07	62.25	5.60	6.00
43	$C_{23}H_{25}Cl_2N_3O_2$+0.2 H_2O	61.39	5.69	9.34	61.26	5.83	9.29
44	$C_{25}H_{27}Cl_2N_3O_3$	61.48	5.57	8.60	61.82	5.88	8.26
45	$C_{22}H_{23}Cl_2N_3O_2$	61.12	5.36	9.72	60.75	5.49	9.14
48	$C_{26}H_{30}Cl_2N_3O_3$	63.80	6.18	5.72	63.59	6.13	5.69

49	$C_{27}H_{26}Cl_2N_2O_2$	67.36	5.44	5.82	67.06	5.43	5.83
50	$C_{22}H_{25}N_3O_2$	72.70	6.93	11.56	72.65	7.17	11.48
51	$C_{21}H_{24}N_2O_3$+0.15 CH_2Cl_2	69.57	6.71	7.67	69.59	6.65	7.88
52	$C_{22}H_{27}N_3O_2$+0.25 CH_2Cl_2	69.11	7.17	10.87	68.86	7.13	10.79
53	$C_{23}H_{32}N_2O_2$	74.96	8.85	7.60	74.67	8.67	7.52
54	$C_{20}H_{28}N_2O_2$	73.14	8.59	8.53	73.13	8.40	8.47
55	$C_{26}H_{36}N_2O_2$	77.10	8.63	6.66	77.21	8.28	6.45
56	$C_{17}H_{24}N_2O$+0.5 H_2O	72.56	8.95	9.95	72.63	9.03	9.97
57	$C_{16}H_{21}N_3O_3$+0.2 H_2O	62.61	7.03	13.69	62.51	6.70	13.40
61	$C_{20}H_{22}N_2O_3$	70.98	6.55	8.28	70.75	6.38	8.56
63	$C_{16}H_{20}N_2O_3$	66.65	6.99	9.72	66.46	6.81	9.60
64	$C_{16}H_{22}N_2O$	74.38	8.58	10.84	74.04	8.74	10.55
65	$C_{15}H_{18}N_2O_4$	62.06	6.25	9.65	62.16	6.11	9.70
66	$C_{15}H_{20}N_2O_2$+0.1 H_2O	68.73	7.77	10.69	68.60	8.09	10.67
70	$C_{16}H_{20}N_2O_3$+0.7 H_2O	63.86	7.17	9.31	63.75	6.89	9.09
71	$C_{16}H_{22}N_2O$	74.38	8.80	10.84	74.24	8.80	10.70
72	$C_{23}H_{26}N_2O_2$+0.4 H_2O	74.73	7.31	7.58	74.54	7.44	7.72
76	$C_{24}H_{27}FN_2O_2$	73.07	6.90	7.10	73.15	7.05	7.08
77	$C_{24}H_{27}FN_2O_2$	73.07	6.90	7.10	72.79	6.80	6.97
78	$C_{15}H_{20}N_2O_4S$	55.54	6.21	8.64	55.70	6.12	8.58
80	$C_{22}H_{25}FN_2O_3S$	63.44	6.05	6.73	63.41	5.98	6.73
81	$C_{22}H_{25}FN_2O_3S$	63.44	6.05	6.73	63.24	5.97	6.66
82	$C_{16}H_{20}N_2O_2S$	63.13	6.62	9.20	63.19	6.56	8.98
83	$C_{16}H_{22}N_2S$	68.42	8.16	9.75	68.42	8.16	9.55
84	$C_{23}H_{25}FN_2OS$	69.67	6.35	7.06	69.41	6.37	6.91
85	$C_{23}H_{27}FN_2O$	75.38	7.43	7.64	75.42	7.37	7.55
87	$C_{24}H_{27}FN_2O_2$	73.07	6.90	7.10	72.76	6.87	7.03
89	$C_{18}H_{23}NO_3$	71.74	7.69	4.65	71.43	7.46	4.05
90	$C_{17}H_{21}NO_3$	71.06	7.37	4.87	71.04	7.25	4.76
92	$C_{23}H_{34}N_2O_2$	74.56	9.25	7.56	74.71	9.24	7.51
94	$C_{20}H_{28}N_2O_2$	73.14	8.59	8.53	73.42	8.67	8.45
95	$C_{21}H_{30}N_2O_2$	73.65	8.83	8.18	73.79	8.80	8.06
96	$C_{24}H_{28}N_2O_2$+0.25H_2O	75.66	7.54	7.35	75.53	7.82	7.11
97	$C_{23}H_{27}N_3O_2$	73.18	7.21	11.13	72.87	6.82	11.11
98	$C_{23}H_{25}FN_2O_2$	72.61	6.62	7.36	72.67	6.71	7.40
99	$C_{22}H_{30}N_2O_2$	74.54	8.53	7.90	74.19	8.65	7.80
100	$C_{26}H_{30}N_2O_2$	77.58	7.51	6.96	77.50	7.28	6.83