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FUNCTIONALLY SUBSTITUTED N- AND P-CONTAINING SALTS OF RESINOUS AND BILE ACIDS

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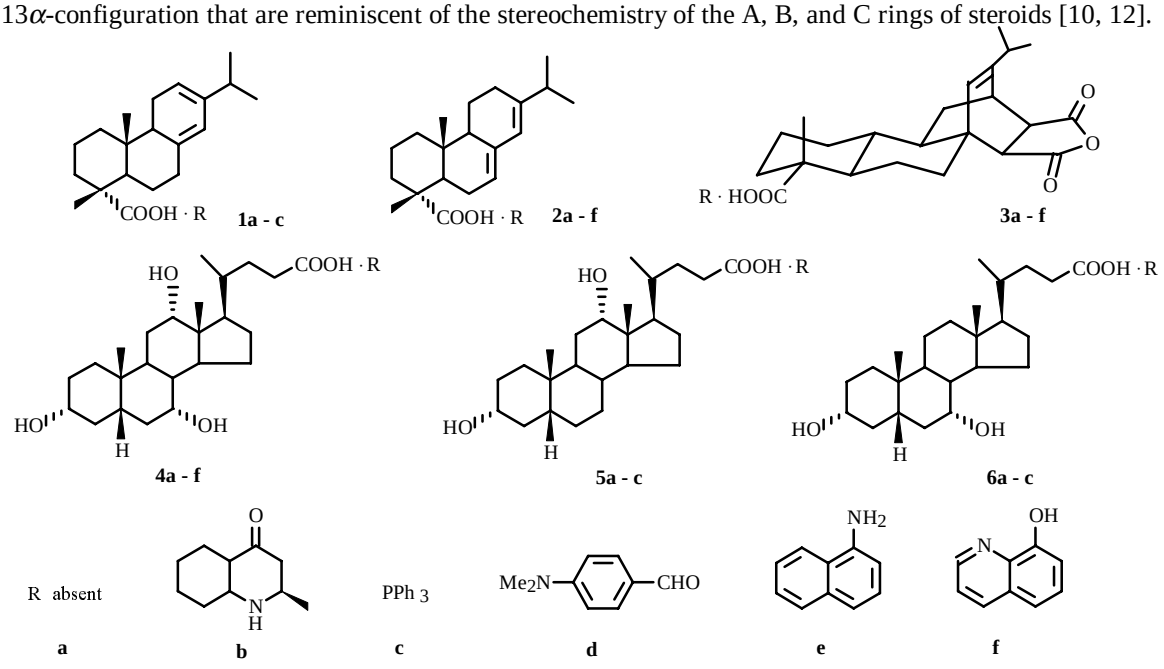
*New functionally substituted N- and P-containing salts of resinous and bile acids **1b, c**, **2b-f**, **3b-f**, **4b-f**, **5b, c**, and **6b, c** were prepared by reaction of the corresponding acids with amines or triphenylphosphine in anhydrous acetone.*

Key words: substituted N- and P-containing salts of carboxylic acids, biological and adhesive activity, amines, triphenylphosphine, synthesis, maleopimaric acid.

Because natural carboxylic acids possess a wide range of useful properties, their functionally substituted N- and P-containing salts are of interest. Examples include their high biological [1-4] and adhesive [5-7] activities. Levopimaric (**1a**), abietinic (**2a**), maleopimaric (**3a**), cholic (**4a**), deoxycholic (**5a**), and chenodeoxycholic (**6a**) carboxylic acids were selected as starting materials because of the similarity of their chemical structures and the need for comparison of the biological activity elicited by the molecular pharmacophores associated with the carboxylic and amino groups in the salts prepared from them [8-12]. Maleopimaric acid (**3a**) is the diene adduct prepared via Diels—Alder reaction of levopimaric acid (**1a**) and maleic anhydride [8, 12, 13]. It represents a convenient and readily available synthon for the preparation of compounds with a wide spectrum of biological, in particular, anti-inflammatory, nematocidal, and fungicidal activities [14-19], and for the synthesis of monomers [20, 21]. The high biological activity of maleopimaric acid derivatives is explained by the stereochemical features of their 13α-configuration that are reminiscent of the stereochemistry of the A, B, and C rings of steroids [10, 12].



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Our goal was to develop a highly effective synthetic method for potentially biologically active functionally substituted N- and P-containing salts of **1a-6a**. The previously unknown salts **1b, c**, **2b-f**, **3b-f**, **4b-f**, **5b, c**, and **6b, c** were prepared by reacting the corresponding acids **1a-6a** with aromatic and heterocyclic amines and triphenylphosphine in 1:1 stoichiometries in anhydrous acetone. The reactions were complete in 5-10 min at 20-23°C. After removal of solvent in vacuum, the yields of the aforementioned salts were 91-96%.

The salts are all colorless or lightly colored substances that form large crystals and are soluble in acetone and C_{1-4} alcohols and poorly soluble in water. They are not hygroscopic and well preserved in sealed ampuls at 0-5°C in the dark. The structures of the synthesized compounds were confirmed by elemental analysis and PMR, IR, and UV spectra. The purity of the prepared compounds was 99 ± 1% according to PMR data.

EXPERIMENTAL

IR spectra were recorded on a Protege-460 Fourier spectrometer in KBr disks; PMR spectra, on a Tesla-567A (100 MHz) spectrometer in $(CD_3)_2CO$ solution. Chemical shifts were measured against TMS as an internal standard. UV spectra were recorded on a Specord UV—Vis instrument using solutions ($1 \cdot 10^{-3}$ M) in *n*-butanol.

N- and P-containing salts of Carboxylic Acids, 1b, c, 2b-f, 3b-f, 4b-f, 5b, c, and 6b, c (General Method). A solution of carboxylic acid **1a-6a** (0.005 mol) in anhydrous acetone (50 mL) was treated with the appropriate amine or triphenylphosphine (0.005 mol). The solvent was removed after all components had dissolved. The solid was heated (40-50°C) in vacuo for 5-6 h. Salts **1b, c**, **2b-f**, **3b-f**, **4b-f**, **5b, c**, and **6b, c** were prepared as homogeneous friable porous masses.

This method was used to prepare:

2*e*-Methyl-4-oxo-*trans*-decahydroquinolonium Levopimarate (1b). Yield 96%, mp 44-45°C. Found (%): C 77.03, H 10.24, N 2.66. Cald. for $C_{30}H_{47}NO_3$ (%): C 76.71, H 10.09, N 2.98. IR spectrum (ν, cm^{-1}): 3035 (=CH); 2932, 2864 (CH_{Alk}); 1721, 1670 (C=O); 1620 (C=C); 1460, 1450 (CH_2); 1385, 1353, 1244, 1132 (C–O). UV spectrum (λ_{max}, ε): 202 (4000), 270 (5000). PMR spectrum (δ, ppm, J/Hz): 0.89 (CH_3, s), 0.96 (2×CH_3, d, J = 5.6), 1.16 (CH_3, s), 1.16 (CH_3, d, ^3J = 5.6), 4.90-5.25 m, 5.55 s (2H, 2=CH).

Triphenylphosphonium Levopimarate (1c). Yield 92%, mp 62-63°C. Found (%): C 81.03, H 8.16, P 5.19. Cald. for $C_{38}H_{45}PO_2$ (%): C 80.82, H 8.03, P 5.48. IR spectrum (ν, cm^{-1}): 3064, 3048, 3025, 3015 (=CH and CH_{Ar}); 2956, 2926, 2890, 2865 (CH_{Alk}); 1689 (C=O); 1620 (C=C); 1580, 1474, 1435 (Ar); 1386, 1279, 1179, 1089, 1024 (C–O); 742, 693, 541, 513, 498, 490 (CH_{Ar}). UV spectrum (λ_{max}, ε): 204 (37000), 268 (13000). PMR spectrum (δ, ppm): 0.93 (CH_3, s), 0.97 (2×CH_3, d), 1.15 (CH_3, s), 4.95-5.20 m, 5.53 s (2H, 2=CH), 7.05-7.85 (15H, m, $3C_6H_5$).

2*e*-Methyl-4-oxo-*trans*-decahydroquinolonium Abietate (2b). Yield 95%, mp 55-56°C. Found (%): C 76.97, H 10.20, N 2.74. Cald. for $C_{30}H_{47}NO_3$ (%): C 76.71, H 10.09, N 2.98. IR spectrum (ν, cm^{-1}): 3025 (=CH); 2950, 2933, 2863 (CH_{Alk}); 1722 (C=O); 1621 (C–C); 1460, 1449 (CH_2); 1385, 1355, 1243, 1152, 1132 (C–O). UV spectrum (λ_{max}, ε): 202 (5000), 233 (21000), 243 (23000), 251 (15000). PMR spectrum (δ, ppm, J/Hz): 0.73 (CH_3, s), 1.01 (2×CH_3, d, ^3J = 7.0), 1.11 (CH_3, d, ^3J = 6.2), 1.27 (CH_3, s), 5.25-5.45 m, 5.78 s (2H, 2=CH).

Triphenylphosphonium Abietate (2c). Yield 92%, mp 57-58°C. Found (%): C 81.11, H 8.21, P 5.22. Cald. for $C_{38}H_{45}PO_2$ (%): C 80.82, H 8.03, P 5.48. IR spectrum (ν, cm^{-1}): 3075, 3055, 3025, 3015 (=CH and CH_{Ar}); 2956, 2930, 2880, 2865, 2847, 2828 (CH_{Alk}); 1684 (C=O); 1645, 1635, 1625 (C=C); 1580, 1474, 1431 (Ar); 1465 (CH_2); 1383, 1279, 1190, 1155, 1119, 1087, 1026 (C–O); 741, 721, 694, 668, 541, 494 (CH_{Ar}). UV spectrum (λ_{max}, ε): 204 (38000), 234 (20000), 244 (22000), 250 (16000), 265 (8000). PMR spectrum (δ, ppm, J/Hz): 0.84 (CH_3, s), 1.00 (2×CH_3, d, ^3J = 7.5), 1.25 (CH_3, s), 5.20-5.45 m, 5.75 s (2H, 2=CH), 7.05-7.90 (15H, m, $3C_6H_5$).

***p*-Dimethylammoniumbenzaldehyde Abietate (2d).** Yield 96%, mp 98-99°C. Found (%): C 77.24, H 9.28, N 2.95. Cald. for $C_{29}H_{41}NO_3$ (%): C 77.12, H 9.15, N 3.10. IR spectrum (ν, cm^{-1}): 3080, 3055, 3025 (=CH and CH_{Ar}); 2958, 2933, 2880, 2865, 2850, 2830, 2797 (CH_{Alk}); 1689, 1661 (C=O); 1600, 1549, 1535 (Ar); 1462, 1445, 1430 (CH_2); 1372, 1282, 1232, 1166, 1065 (C–O); 825, 813, 728, 596 (CH_{Ar}). UV spectrum (λ_{max}, ε): 204 (15000), 234 (23000), 244 (27000), 251 (16000), 340 (25000). PMR spectrum (δ, ppm, J/Hz): 0.84 (CH_3, s), 1.00 (2×CH_3, d, ^3J = 7.3), 1.25 (CH_3, s), 3.09 [$(CH_3)N$, s], 5.25-5.40 m, 5.75 s (2H, 2=CH), 6.65-7.80 (4H, m, C_6H_4), 9.72 (CHO, s).

α-Naphthylammonium Abietate (2e). Yield 94%, mp 96-97°C. Found (%): C 81.11, H 9.03, N 2.96. Cald. for $C_{30}H_{39}NO_2$ (%): C 80.86, H 8.82, N 3.14. IR spectrum (ν, cm^{-1}): 3383 (NH); 3080, 3060, 3044, 3020 (=CH and CH_{Ar}); 2953,

2930, 2880, 2871, 2850, 2831 (CH_{Alk}); 1688 (C=O); 1622, 1590, 1547, 1511, 1456, 1404 (Ar); 1287, 1254, 1228, 1192, 1154 (C–O); 789, 771, 721 (CH_{Ar}). UV spectrum (λ_{max}, ε): 203 (7000), 210 (44000), 234 (28000), 244 (48000), 253 (22000), 321 (7000). PMR spectrum (δ, ppm, J/Hz): 0.84 (CH_3, s), 1.01 (2×CH_3, d, ^3J = 7.5), 1.24 (CH_3, s), 5.25-5.43 m, 5.75 s (2H, 2=CH), 6.55-8.10 (7H, m, $C_{10}H_7$).

8-Hydroxyquinolonium Abietate (2f). Yield 95%, mp 57-58°C. Found (%): C 78.06, H 8.52, N 2.90. Cald. for $C_{29}H_{37}NO_3$ (%): C 77.82, H 8.33, N 3.13. IR spectrum (ν, cm^{-1}): 3100, 3070, 3045, 3025 (=CH and CH_{Ar}); 2959, 2932, 2880, 2860, 2850, 2830 (CH_{Alk}); 1690 (C=O); 1625 (C=C); 1580, 1508, 1471, 1410 (Ar); 1382, 1285, 1223, 1206, 1155, 1094 (C–O); 891, 818, 781, 742, 711 (CH_{Ar}). UV spectrum (λ_{max}, ε): 204 (35000), 240 (63000), 250 (17000), 309 (3000). PMR spectrum (δ, ppm, J/Hz): 0.85 (CH_3, s), 0.98 (2×CH_3, d, ^3J = 7.4), 1.25 (CH_3, s), 5.20-5.40 m, 5.74 s (2H, 2=CH), 6.95-8.90 (6H, m, C_9H_6N).

2*e*-Methyl-4-oxo-*trans*-decahydroquinolonium maleopimarate (3b). Yield 95%, mp 57-58°C. Found (%): C 72.13, H 8.91, N 2.31. Cald. for $C_{34}H_{49}NO_6$ (%): C 71.93, H 8.70, N 2.47. IR spectrum (ν, cm^{-1}): 3035 (=CH); 2960, 2936, 2865 (CH_{Alk}); 1860, 1842, 1779, 1716 (C=O); 1621 (C=C); 1466, 1449 (CH_2); 1231, 1087, 1003, 947, 923, 905 (C–O). UV spectrum (λ_{max}, ε): 205 (7000). PMR spectrum (δ, ppm, J/Hz): 0.56 (CH_3, s), 0.97 (2×CH_3, d, ^3J = 7.4), 1.11 (CH_3, s), 1.22 (CH_3, d, ^3J = 6.0), 5.48 (1H, s, =CH), 5.75-7.00 (2H, br.s, NH_2).

Triphenylphosphonium Maleopimarate (3c). Yield 96%, mp 47-48°C. Found (%): C 76.29, H 7.25, P 4.60. Cald. for $C_{42}H_{47}PO_5$ (%): C 76.11, H 7.15, P 4.67. IR spectrum (ν, cm^{-1}): 3070, 3053, 3030, 3000 (=CH and CH_{Ar}); 2955, 2928, 2867 (CH_{Alk}); 1855, 1841, 1778, 1690 (C=O); 1640 (C=C); 1585, 1433, 1386 (Ar); 1476, 1460 (CH_2); 1278, 1228, 1085, 1027, 1001, 946, 921, 904 (C–O); 852, 742, 694, 671, 566, 540 (CH_{Ar}). UV spectrum (λ_{max}, ε): 205 (57000), 262 (7000). PMR spectrum (δ, ppm, J/Hz): 0.56 (CH_3, s), 0.96 (CH_3, d, ^3J = 7.6), 1.13 (CH_3, s), 5.48 (1H, s, =CH), 7.00-7.50 (15H, m, $3C_6H_5$), 7.65 (1H, br.s, PH).

***p*-Dimethylammoniumbenzaldehyde Maleopimarate (3d).** Yield 94%, mp 36-37°C. Found (%): C 72.34, H 8.03, N 2.27. Cald. for $C_{33}H_{43}NO_6$ (%): C 72.10, H 7.88, N 2.55. IR spectrum (ν, cm^{-1}): 3065, 3040, 3025 (=CH and CH_{Ar}); 2960, 2932, 2868, 2825, 2739 (CH_{Alk}); 1855, 1842, 1779, 1716, 1691, 1660 (C=O); 1640 (C=C); 1596, 1552, 1534, 1373 (Ar); 1465, 1440 (CH_2), 1230, 1166, 1084, 1001, 946, 921, 905 (C–O); 815, 753, 728, 693, 670, 595 (CH_{Ar}). UV spectrum (λ_{max}, ε): 204 (11000), 240 (4000), 350 (13000). PMR spectrum (δ, ppm, J/Hz): 0.57 (CH_3, s), 0.95 (2×CH_3, d, ^3J = 7.3), 1.14 (CH_3, s), 3.04 [s, $(CH_3)_2N$], 5.48 (1H, s, =CH), 6.45-7.85 (4H, m, C_6H_4), 9.69 (1H, s, CHO).

α-Naphthylammonium Maleopimarate (3e). Yield 93%, mp 62-63°C. Found (%): C 75.34, H 7.66, N 2.34. Cald. for $C_{34}H_{41}NO_5$ (%): C 75.11, H 7.60, N 2.58. IR spectrum (ν, cm^{-1}): 3385 (NH); 3080, 3060, 3049, 3020 (=CH and CH_{Ar}); 2956, 2932, 2869 (CH_{Alk}); 1855, 1841, 1778, 1708, 1692 (C=O); 1640 (C=C); 1629, 1590, 1578, 1514, 1460, 1406 (Ar); 1387, 1375, 1278, 1230, 1087, 946, 923 (C–O); 791, 772 (CH_{Ar}). UV spectrum (λ_{max}, ε): 210 (40000), 220 (31000), 240 (16000), 308 (4000). PMR spectrum (δ, ppm, J/Hz): 0.65 (CH_3, s), 1.00 (2×CH_3, d, ^3J = 7.4), 1.16 (CH_3, s), 5.61 (1H, s, =CH), 6.60-8.15 (7H, m, $C_{10}H_7$).

8-Hydroxyquinolonium Maleopimarate (3f). Yield 94%, mp 47-48°C. Found (%): C 72.73, H 7.34, N 2.40. Cald. for $C_{33}H_{39}NO_6$ (%): C 72.64, H 7.20, N 2.57. IR spectrum (ν, cm^{-1}): 3405 (OH), 3075, 3053, 3040 (=CH and CH_{Ar}); 2957, 2934, 2869 (CH_{Alk}); 1855, 1842, 1779, 1691 (C=O); 1637 (C=C); 1580, 1505, 1472, 1406, 1380 (Ar); 1464, 1446 (CH_2); 1279, 1226, 1208, 1193, 1163, 1088, 947, 923, 905 (C–O); 853, 825, 790, 709, 672 (CH_{Ar}). UV spectrum (λ_{max}, ε): 203 (31000), 242 (34000). PMR spectrum (δ, ppm, J/Hz): 0.57 (CH_3, s), 0.95 (2×CH_3, d, ^3J = 7.5), 1.14 (CH_3, s), 5.48 (1H, s, =CH), 7.05-8.85 (8H, m, C_9H_6N, NH, and OH).

2*e*-Methyl-4-oxo-*trans*-decahydroquinolonium Cholate (4b). Yield 91%, mp 73-74°C. Found (%): C 71.19, H 10.11, N 2.31. Cald. for $C_{34}H_{57}NO_6$ (%): C 70.92, H 9.98, N 2.43. IR spectrum (ν cm^{-1}): 2935, 2864 (CH_{Alk}); 1713, 1625 (C=O); 1470, 1449 (CH_2); 1400, 1375, 1304, 1226, 1078, 1047, 981, 946, 913 (C–O). UV spectrum (λ_{max}, ε): 215 (1000), 305 (100). PMR spectrum (δ, ppm, J/Hz): 0.68 (CH_3-18, s), 0.91 (CH_3-19, s), 1.04 (CH_3-21, d, ^3J = 5.3), 1.16 (CH_3, d, ^3J = 6.7), 3.55-4.05 (m, H-3, H-7, H-12).

Triphenylphosphonium Cholate (4c). Yield 92%, mp 67-68°C. Found (%): C 75.29, H 8.35, P 4.51. Cald. for $C_{42}H_{55}PO_5$ (%): C 75.20, H 8.26, P 4.62. IR spectrum (ν, cm^{-1}): 3522 (OH); 3064, 3050, 3025, 3005 (CH_{Ar}); 2980, 2965, 2955, 2934, 2920, 2885, 2872 (CH_{Alk}); 1715 (C=O); 1580, 1475, 1434, 1400 (Ar); 1375, 1329, 1287, 1254, 1241, 1120, 1091, 1070, 1045, 1000, 982, 915 (C–O); 742, 695 (CH_{Ar}). UV spectrum (λ_{max}, ε): 205 (33000), 265 (8000). PMR spectrum (δ, ppm, J/Hz): 0.70 (CH_3-18, s), 0.91 (CH_3-19, s), 1.06 (CH_3-21, d, ^3J = 4.2), 7.10-7.90 (15H, m, $3C_6H_5$).

p-Dimethylammoniumbenzaldehyde Cholate (4d). Yield 93%, mp 55-56°C. Found (%): C 71.22, H 9.41, N 2.31. Cald. for $C_{33}H_{51}NO_6$ (%): C 71.06, H 9.22, N 2.51. IR spectrum (ν, cm^{-1}): 3523 (OH); 3050 (CH_{Ar}); 2980, 2966, 2955, 2934, 2965, 2890, 2872, 2830 (CH_{Alk}); 1716, 1660 (C=O); 1600, 1553, 1534 (Ar); 1467, 1445 (CH_2); 1374, 1330, 1260, 1288, 1243, 1230, 1167, 1122, 1092, 1087, 1046, 981, 914 (C–O); 813, 729, 681 (CH_{Ar}). UV spectrum (λ_{max}, ε): 204 (10000), 244 (5000), 341 (24000). PMR spectrum (δ, ppm, J/Hz): 0.71 (CH_3-18, s), 0.90 (CH_3-19, s), 1.06 (CH_3-21, d, ^3J = 4.3), 3.09 [s, $(CH_3)_2N$], 2.95-4.00 (m, H-3, H-7, H-12).

***α*-Naphthylammonium Cholate (4e).** Yield 95%, mp 141-142°C. Found (%): C 74.24, H 9.16, N 2.33. Cald. for $C_{34}H_{49}NO_5$ (%): C 74.01, H 8.95, N 2.54. IR spectrum (ν, cm^{-1}): 3521 (OH); 3345 (NH); 3080, 3060, 3045, 3020 (CH_{Ar}); 2965, 2934, 2920, 2871 (CH_{Alk}); 1713 (C=O); 1629, 1595, 1576, 1514, 1406 (Ar); 1462, 1447 (CH_2); 1376, 1335, 1289, 1253, 1197, 1121, 1090, 1077, 1044, 1016, 980, 950, 914 (C–O); 789, 770, 612 (CH_{Ar}). UV spectrum (λ_{max}, ε): 210 (42000), 245 (23000), 320 (7000). PMR spectrum (δ, ppm, J/Hz): 0.70 (CH_3-18, s), 0.91 (CH_3-19, s), 1.07 (CH_3-21, d, ^3J = 4.5), 3.10-4.05 (m, H-3, H-7, H-12), 6.55-8.10 (10H, m, $C_{10}H_7$ and NH_3).

8-Hydroxyquinolonium Cholate (4f). Yield 94%, mp 191-192°C. Found (%): C 71.84, H 8.64, N 2.41. Cald. for $C_{33}H_{47}NO_6$ (%): C 71.58, H 8.55, N 2.53. IR spectrum (ν, cm^{-1}): 3522 (OH); 3095, 3070, 3050, 3010 (CH_{Ar}); 2980, 2966, 2955, 2934, 2915, 2885, 2872, 2850 (CH_{Alk}); 1715 (C=O); 1577, 1506, 1473, 1403 (Ar); 1446 (CH_2); 1375, 1335, 1286, 1255, 1229, 1197, 1164, 1092, 1078, 1045, 982, 915 (C–O); 823, 788, 750, 710, 612 (CH_{Ar}). UV spectrum (λ_{max}, ε): 205 (31000), 244 (40000), 312 (2000). PMR spectrum (δ, ppm, J/Hz): 0.69 (CH_3-18, s), 0.90 (CH_3-19, s), 1.05 (CH_3-21, d, ^3J = 4.6), 2.90-4.05 (m, H-3, H-7, H-12), 6.95-8.95 (8H, m, C_9H_6N, NH, and OH).

2*e*-Methyl-4-oxo-*trans*-decahydroquinolonium Deoxycholate (5b). Yield 96%, mp 71-72°C. Found (%): C 73.13, H 10.31, N 2.32. Cald. for $C_{34}H_{57}NO_5$ (%): C 72.95, H 10.26, N 2.50. IR spectrum (ν, cm^{-1}): 2935, 2862 (CH_{Alk}); 1713, 1625 (C=O); 1449 (CH_2); 1376, 1304, 1245, 1219, 1133, 1092, 1066, 1045, 1014, 970, 944 (C–O). UV spectrum (λ_{max}, ε): 214 (1000), 305 (100). PMR spectrum (δ, ppm, J/Hz): 0.71 (CH_3-18, s), 0.94 (CH_3-19, s), 1.06 (CH_3-21, d, ^3J = 5.6), 1.16 (CH_3, d, ^3J = 6.8), 3.55-4.05 (m, H-3, H-12).

Triphenylphosphonium Deoxycholate (5c). Yield 95%, mp 134-135°C. Found (%): C 77.20, H 8.55, P 4.50. Cald. for $C_{42}H_{55}PO_4$ (%): C 77.03, H 8.46, P 4.73. IR spectrum (ν, cm^{-1}): 3066, 3053, 3025, 3005 (CH_{Ar}); 2975, 2932, 2863 (CH_{Alk}); 1696 (C=O); 1580, 1473, 1433, 1410 (Ar); 1452, 1445 (CH_2); 1381, 1363, 1305, 1293, 1270, 1254, 1220, 1199, 1157, 1110, 1094, 1069, 1042, 1000, 969, 944, 912 (C–O); 741, 715, 694 (CH_{Ar}). UV spectrum (λ_{max}, ε): 205 (35000), 264 (8000). PMR spectrum (δ, ppm, J/Hz): 0.71 (CH_3-18, s), 0.92 (CH_3-19, s), 1.08 (CH_3-21, d, ^3J = 4.4), 3.80-4.35 (m, H-3, H-12), 7.05-7.80 (15H, m, $3C_6H_5$).

2*e*-Methyl-4-oxo-*trans*-decahydroquinolonium Chenodeoxycholate (6b). Yield 92%, mp 69-70°C. Found (%): C 73.18, H 10.34, N 2.27. Cald. for $C_{34}H_{57}NO_5$ (%): C 72.95, H 10.26, N 2.50. IR spectrum (ν, cm^{-1}): 2933, 2864 (CH_{Alk}); 1714, 1625 (C=O); 1464, 1449 (CH_2); 1375, 1364, 1334, 1305, 1245, 1220, 1166, 1131, 1079, 1050, 1001, 980 (C–O). UV spectrum (λ_{max}, ε): 215 (1000), 305 (100). PMR spectrum (δ, ppm, J/Hz): 0.68 (CH_3-18, s), 0.93 (CH_3-19, s), 0.97 (CH_3-21, d, ^3J = 6.0), 1.16 (CH_3, d, ^3J = 6.8), 3.70-4.10 (m, H-3, H-7).

Triphenylphosphonium Chenodeoxycholate (6c). Yield 93%, mp 123-124°C. Found (%): C 77.24, H 8.59, P 4.53. Cald. for $C_{42}H_{55}PO_4$ (%): C 77.03, H 8.46, P 4.73. IR spectrum (ν, cm^{-1}): 3066, 3048, 3025, 3007 (CH_{Ar}); 2975, 2935, 2900, 2865, 2850 (CH_{Alk}); 1708 (C=O); 1581, 1475, 1435, 1417 (Ar); 1447 (CH_2); 1368, 1329, 1308, 1267, 1245, 1202, 1160, 1120, 1077, 1025, 1000, 977 (C–O); 742, 717, 696 (CH_{Ar}). UV spectrum (λ_{max}, ε): 205 (35000), 264 (8000). PMR spectrum (δ, ppm, J/Hz): 0.70 (CH_3-18, s), 0.92 (CH_3-19, s), 0.98 (CH_3-21, d, ^3J = 4.5), 3.10-4.80 (m, H-3, H-7), 7.00-7.70 (15H, m, $3C_6H_5$).

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