Supporting information

Mesoporous poly-melamine-formaldehyde stabilized palladium nanoparticles (Pd@mPMF) catalyzed mono and double carbonylation of aryl halides with amines

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Data for the products of mono carbonylation

N-benzoylmorpholine (Table 4, entry 1): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.95(d, *J*= 8.0Hz, 2H), 7.66(t, *J*= 7.6 Hz, 1H), 7.52(t, *J*= 7.6 Hz, 2H), 3.85-3.76(m, 4H), 3.65 (t, *J*= 4.8 Hz, 2H), 3.37(t, *J*= 4.8 Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ = 42.8, 48.5, 67.2, 127.3, 128.9, 130.2, 135.5, 170.7; HR-MS (M+H)⁺ m/z 191.61.

N-(4-toluoyl)morpholine (Table 4, entry 2): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.85 (d, *J*= 8.0 Hz, 2H), 7.31 (d, *J*= 8.0 Hz, 2H), 3.77 (s, 4H), 3.63 (t, *J*= 4.4 Hz, 2H), 3.36 (t, *J*= 4.4 Hz, 2H), 2.41 (s, 3H). ¹³C NMR (CDCl3, 100 MHz) δ = 21.5, 42.6, 48.3, 67.0, 127.3, 129.3, 132.5, 140.2, 171.7; HR-MS (M+H)⁺ m/z 206.21.

N-(2-toluoyl)morpholine (Table 4, entry 3): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.71 (dd, *J*= 7.6 Hz, *J*= 0.8 Hz, 1H), 7.50 (dt, *J*= 7.6 Hz, *J*= 1.2 Hz, 1H), 7.35-7.31 (m, 2H), 3.83-3.76 (m, 4H), 3.67 (t, *J*= 4.8 Hz, 2H), 3.40 (t, *J*= 4.8 Hz, 2H), 2.67 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ = 19.1, 42.0, 47.4, 67.1, 125.9, 126.1, 129.2, 130.6, 134.3, 135.8, 170.3; HR-MS (M+H)⁺ m/z 206.22.

N-(3-toluoyl)morpholine (Table 4, entry 4): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.76 (d, *J*= 7.6 Hz, 2H), 7.46 (d, *J*= 7.6 Hz, 1H), 7.40 (t, *J*= 7.8 Hz, 1H), 3.81-3.77(m, 4H), 3.65 (t, *J*= 4.8Hz, 2H), 3.37 (t, *J*= 4.8 Hz, 2H), 2.43 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz) δ = 21.4, 42.5, 48.2, 66.9, 124.0, 127.7, 128.4, 130.6, 135.3, 138.5, 170.8.; HR-MS (M+H)⁺ m/z 206.11.

N-(4-methoxybenzoyl)morpholine (Table 4, entry 5): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.92 (dt, *J*= 8.8 Hz, *J*= 2.4 Hz, 2H), 6.98 (dt, *J*= 8.8 Hz, *J*= 2.4 Hz, 2H), 3.89 (s, 3H), 3.81-3.76 (m, 4H), 3.65 (t, *J*= 4.8 Hz, 2H), 3.37(d, *J*= 4.8Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ = 42.6, 48.2, 55.5, 67.0, 113.8, 129.3, 161.0, 170.5; HR-MS (M+H)⁺ m/z 222.12.

(4-(dimethylamino)phenyl)(morpholino)methanone: (Table 4, entry 6): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.81(d, *J*= 8.8 Hz, 2H), 6.67 (d, *J*= 8.8 Hz, 2H), 3.82-3.72 (m, 4H), 3.63(t, *J*= 4.8Hz, 2H), 3.38 (t, *J*= 4.8Hz, 2H), 3.11 (s, 6H). ¹³C NMR (CDCl₃, 100 MHz) δ = 40.2, 44.0, 66.2, 111.6, 124.7, 130.0, 152.1, 168.8; HR-MS (M+H)⁺ m/z 234.51.

N-(4-chlorobenzoyl)morpholine (Table 4, entry 7): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.42-7.35(m, 4H), 3.85-3.56(m, 6H), 3.56-3.33 (m, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ = 42.8, 48.2, 66.9, 128.7, 128.9, 133.7, 136.1, 169.5; HR-MS (M+H)⁺ m/z 248.65.

N-(4-florobenzoyl)morpholine (Table 4, entry 8): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 8.01-7.98 (m, 2H), 7.23-7.18(m, 2H),3.82-3.76(m, 4H), 3.65 (t, *J*= 4.8 Hz, 2H), 3.39 (t, *J*= 4.8 Hz,2H). ¹³C NMR (CDCl₃, 100 MHz) δ = 41.9, 47.2, 65.9, 114.6, 114.9, 128.5, 128.6, 130.5, 161.4, 163.8, 168.6; HR-MS (M+H)⁺ m/z 232.08. **morpholino(4-(trifluoromethyl)phenyl)methanone: (Table 4, entry 9):** ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.68 (d, *J*= 8.0Hz, 2H), 7.52(d, *J*= 8.0Hz, 2H), 3.88-3.71 (m, 4H), 3.71-3.56 (m,2H), 3.50-3.30 (m, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ = 44.0, 66.2, 124.1, 124.8, 127.5, 132.0, 138.5, 168.9; HR-MS (M+H)⁺ m/z 259.52.

4-(morpholine-4-carbonyl)benzaldehyde (Table 4, entry 10): 1H NMR (CDCl₃, 400 MHz, 298 K): δ= 10.06 (s, 1H), 7.95 (d, *J*= 8.0 Hz, 2H), 7.55 (d, *J*= 8.0 Hz, 2H), 3.88-3.71 (m, 4H), 3.69-3.56 (m,2H), 3.52-3.30 (m, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ= 42.7, 48.2, 66.9, 127.8, 130.0, 137.2, 141.1, 169.1, 191.5; HR-MS (M+H)⁺ m/z 242.05.

Ethyl 3-(morpholine-4-carbonyl)benzoate (Table 4, entry 11): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ= 8.11(dt, *J*= 8.4 Hz, *J*= 1.6 Hz, 2H), 7.47 (dt, *J*= 8.4 Hz, *J*= 1.6Hz, 2H), 4.40 (q, *J*= 7.2 Hz, 2H), 3.88-3.72 (m, 4H), 3.68-3.56 (m,2H), 3.48-3.32 (m, 2H),1.40 (t, *J*=7.2 Hz, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ= 14.1, 44.0, 60.8, 66.2, 127.1, 129.7, 131.1, 139.5, 165.9, 168.9; HR-MS (M+H)⁺ m/z 263.51.

Methyl 3-(morpholine-4-carbonyl)benzoate: (Table 4, entry 12): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 8.00 (d, *J*= 8 Hz, 2H), 7.50 (d, *J*= 8.4 Hz, 2H), 3.92-3.69 (m, 4H), 3.68-3.55 (m, 2H), 3.51-3.26 (m, 2H), 2.62 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ = 42.5, 48.1, 52.2, 66.6, 128.6, 128.7, 130.2, 130.7, 131.4, 135.6, 166.0, 169.2; HR-MS (M+H)⁺ m/z 256.09.

mesityl(morpholino)methanone (Table 4, entry 13): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ= 6.84 (s, 2H), 3.82 (t, J= 4.4 Hz, 2H), 3.76 (t, J= 4.4Hz, 2H), 3.56 (t, J= 4.8Hz, 2H), 3.18 (t, J= 4.8 Hz, 2H), 2.26 (s, 3H), 2.21 (s, 6H). ¹³C NMR (CDCl₃, 100 MHz) δ= 18.7, 21.8, 44.0, 66.2, 127.7, 133.2, 134.3, 139.2, 166.3; HR-MS (M+H)⁺ m/z 233.53.

(4-tert-butylphenyl)(morpholino)methanone (Table 4, entry 14): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ= 7.88(d, *J*= 8.4 Hz, 2H), 7.53(d, *J*= 8.4 Hz, 2H), 3.85-3.74(m,4H), 3.65 (t, *J*= 4.8 Hz, 2H), 3.37 (t, *J*= 4.8 Hz, 2H), 1.34(s, 9H). ¹³C NMR (CDCl₃, 100 MHz) δ= 31.3, 34.2, 44.0, 66.2, 124.8, 126.8, 132.1, 152.3, 168.9; HR-MS (M+H)⁺ m/z 247.58.

Morpholino(pyridin-3-yl)methanone (Table 4, entry 15): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 8.69-8.62(m, 1H), 7.76 (dt, *J*= 7.6 Hz, *J*= 2.0 Hz, 1H), 7.38-7.34 (m, 1H), 3.83-3.56 (m, 6H), 3.54-3.33 (m, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ = 42.7, 48.4, 66.9, 123.6, 131.3, 135.2, 148.1, 151.1, 167.9; HR-MS (M+H)⁺ m/z 192.11.

morpholino(thiophen-2-yl)methanone ((Table 4, entry 16): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.46 (d, *J*= 4.8 Hz, 1H), 7.30-7.27(m, 1H), 7.05 (t, *J*= 4.8 Hz, 1H), 3.79-3.69(m, 8H). ¹³C NMR (CDCl₃, 100 MHz) δ = 45.8, 66.8, 126.7, 128.8, 128.9, 136.6, 163.6; HR-MS (M+H)⁺ m/z 197.58.

morpholino(pyrazin-2-yl)methanone (Table 4, entry 17): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ= 8.95(s, 1H), 8.63(d, *J*= 6.4 Hz, 1H), 8.56-8.51(m, 1H), 3.85-3.77 (m, 4H), 3.72 - 3.64 (m, 4H).

¹³C NMR (CDCl₃, 100 MHz) δ = 26.7, 42.8, 48.3, 66.8, 129.4, 155.6 159.6, 165.2; HR-MS (M+H)⁺ m/z 193.82.

morpholino(naphthalen-1-yl)methanone (Table 4, entry 18): ¹H NMR (CDCl₃, 400 MHz, 298 K): δ = 7.94-7.86 (m, 2H), 7.82-7.77(m, 1H), 7.58-7.46 (m, 4H), 4.31-4.23(m, 2H), 3.58-3.52(m, 2H), 3.18-3.06(m, 2H), 2.84-2.67(m, 2H),2.62(s, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ =43.9, 66.2, 123.3, 126.7, 127.3, 128.7, 128.4, 129.2, 129.6, 133.5, 134.8, 168.9; HR-MS (M+H)⁺ m/z 241.54.

Data for the products of double carbonylation

1-morpholino-2-phenylethane-1,2-dione (Table 5, entry 1): ¹H NMR (250 MHz, CDCl₃) δ (ppm) 3.35-3.38 (2H, m), 3.62-3.69 (2H, m), 3.76- 3.83 (4H, m), 7.47-7.57 (2H, m), 7.62-7.70 (1H, m), 7.92-8.00 (2H, m). ¹³C (75 MHz, CDCl₃) δ (ppm): 41.7, 46.4, 66.8, 66.8, 129.2, 129.8, 133.2, 135.1, 165.6, 191.3; HR-MS (M+H)⁺ m/z 218.78.

N,*N*-diethyl-2-oxo-2-phenylacetamid (Table 5, entry 2): ¹H NMR (250 MHz, CDCl₃) δ (ppm) 1.16 (3H, t, 3 *J*H-H = 7.1 Hz, 3H), 1.28 (3H, t, 3*J*H-H= 7.2 Hz, 3H), 3.24 (2H, q, 3*J*H-H = 7.1 Hz, 2H), 3.57 (2H, q, 3*J*H-H = 7.2 Hz, 2H), 7.45 – 7.55 (2H, m), 7.58 – 7.69 (1H, m), 7.90 – 7.99 (2H, m). ¹³C NMR (75 MHz, CDCl₃) δ (ppm): 12.8, 14.2, 38.9, 42.2, 129.1, 129.7, 133.3, 134.7, 166.8, 191.8; HR-MS (M+H)⁺ m/z 205.02.

2-oxo-2-phenyl-*N***,***N***-dipropylacetamide (Table 5, entry 3):** ¹H NMR (250 MHz, CDCl₃) δ (ppm) 0.79 (3H, t, 3*J*H-H = 7.31 Hz), 1.00 (3H, t, 3*J*H-H = 7.3 Hz), 1.42-1.82 (3H, m), 3.13 (2H, q, 3*J*H-H = 7.55), 3.46 (2H, 2H, q, 3*J*H-H = 7.53), 7.44-7.57 (2H, m), 7.57-7.69 (1H, m), 7.89-8.00 (2H, m). ¹³C NMR (75 MHz, CDCl₃) δ (ppm): 11.1, 11.6, 20.7, 21.9, 45.9, 49.4, 129.0, 129.7, 133.4, 134.6, 167.3, 191.6; HR-MS (M+H)⁺ m/z 233.76.

N-benzyl-*N*-methyl-2-oxo-2-phenylacetamide (Table 5, entry 4): ¹H NMR (250 MHz, CDCl₃) δ (ppm); 2.72 and 2.88 (3H, s), 4.28 and 4.63 (2H, s), 7.10-7.32 (6H, m), 7.36-7.45 (2H, m,), 7.49-7.58 (1H, m), 7.82- 7.92 (2H, m). ¹³C (75 MHz, CDCl₃) δ (ppm): 31.5, 34.6, 53.6, 49.9, 128.0, 128.4, 128.5, 129.0, 129.2, 129.8, 133.2, 133.4, 135.0, 135.9, 167.3, 167.5, 191.6, 191.7; HR-MS (M+H)⁺ m/z 253.64.

1-phenyl-2-(piperidin-1-yl)ethane-1,2-dione (Table 5, entry 5): ¹H NMR (250 MHz, CDCl₃) δ (ppm) 1.49-1.61 (2H, m), 1.65-1.74 (4H, m), 3.26- 3.33 (2H, m), 3.67-3.75 (2H, m), 7.46-7.56 (2H, m), 7.60-7.69 (1H, m), 7.91-7.99 (2H, m). ¹³C NMR (75 MHz, CDCl₃) δ (ppm): 24.6, 25.6, 26.3, 42.4, 47.2, 129.1, 129.7, 133.4, 134.8, 165.5, 192.1; HR-MS (M+H)⁺ m/z 217.09.

1-(3,4-dihydroisoquinolin-2(1*H***)-yl)-2-phenylethane-1,2-dione (Table 5, entry 6):** ¹H NMR (250 MHz, CDCl₃) δ (ppm) 2.87 and 3.01 (2H, 2 t, 3*J*H-H = 5.8 and 3*J*H-H = 6.0 Hz), 3.57-3.67 and 3.96-4.04 (2H, m), 4.54 and 4.92 (2H, s), 6.90-7.25 (4H, m), 7.44- 7.58 (2H, m), 7.59-7.71 (1H, m) 7.90-8.04 (2H, m). ¹³C (75 MHZ, CDCl₃) δ (ppm): 28.4, 29.3, 39.5, 43.6, 47.5, 126.2,

126.7, 126.8, 127.0, 127.3, 128.9, 129.1, 129.3, 129.8, 131.6, 131.9, 133.2, 133.5, 134.9, 135.0, 165.9, 166.2, 191.1, 191.6; HR-MS (M+H)⁺ m/z 266.11.

1-(3,4-dihydroisoquinolin-2(1*H***)-yl)-2-(4-methoxyphe nyl)ethane-1,2-dione (Table 5, entry 7): ¹H NMR (250 MHz, CDCl₃) \delta (ppm) 2.85 and 3.00 (2H, 2 t, 3***J***H-H = 5.8 and 3***J***H-H = 6.0 Hz), 3.57-3.66 and 3.94-4.02 (2H, m), 3.87 and 3.89 (3H, s), 4.54 and 4.90 (2H, s), 6.90-7.25 (6H, m), 7.86-8.00 (2H, m). ¹³C (75 MHZ, CDCl₃) \delta (ppm): 28.4, 29.4, 39.4, 43.5, 43.7, 47.5, 55,8, 114,5, 126.2, 126.4, 126.7, 126.9, 127.0, 127.3, 129.1, 131.9, 132.1, 132.3, 132.4, 133.6, 134.4, 165.1, 166.3, 166.6, 190.2, 190.4; HR-MS (M+H)⁺ m/z 296.32.**

1-(3,4-dihydroisoquinolin-2(1*H***)-yl)-2-(2-methoxyphenyl) ethane-1,2-dione (Table 5, entry 8):** ¹H NMR (250 MHz, CDCl₃) δ (ppm) 2.90 and 2.99 (2H, 2 t, 3*J*H-H = 5.9 and 3*J*H-H = 6.0 Hz), 3.42 and 3.69 (3H, s), 3.60-3.68 and 3.88-3.98 (2H, m), 4.56 and 4.84 (2H, s), 6.78- 7.24 (6H, m), 7.47-7.63 (1H, m), 7.88-8.02 (1H, m). ¹³C (75 MHZ, CDCl₃) δ (ppm): 28.0, 28.7, 39.3, 43.2, 43.4, 47.4, 55.3, 56.0, 112.2, 112.8, 123.7, 126.3, 126.6, 126.9, 127.1, 128.8, 129.1, 131.3, 132.5, 132.6, 134.0, 134.4, 136.1, 136.3, 160.0, 160.3, 167.9, 190.4, 190.9; HR-MS (M+H)⁺ m/z 296.54.

1-(3,4-dihydroisoquinolin-2(1*H***)-yl)-2-p-tolylethane-1,2-dione (Table 5, entry 9): ¹H NMR (250 MHz, CDCl3) \delta (ppm) 2.42 and 2.44 (3H, s), 2.85 and 3.00 (2H, 2 t, 3***J***H-H = 5.8 and 3***J***H-H = 6.0 Hz), 3.53-3.70 and 3.90-4.06 (2H, m), 4.53 and 4.91 (2H, s), 6.88-7.39 (6H, m), 7.79-7.96 (2H, m). ¹³C (75 MHZ, CDCl₃) \delta (ppm): 22.1, 28.4, 29.4, 39.4, 43.5, 43.6, 47.5, 126.2, 126.7, 126.9, 127.0, 127.3, 128.9, 129.1, 130.0, 130.8, 131.7, 132.0, 133.6, 134.3, 146.3, 166.1, 166.4, 193.3; HR-MS (M+H)⁺ m/z 280.75.**

1-(3,4-dihydroisoquinolin-2(1*H***)-yl)-2-o-tolylethane-1,2-dione (Table 5, entry 10): ¹H NMR (250 MHz, CDCl3) \delta (ppm) 2.67 and 2.71 (3H, s), 2.88 and 3.01 (2H, 2 t, 3***J***H-H = 5.8 and 3***J***H-H = 6.0 Hz), 3.57-3.69 and 3.93-4.04 (2H, m), 4.57 and 4.90 (2H, s), 6.91- 7.37 (6H, m), 7.42-7.55 (1H, m), 7.62-7.78(1H, m). ¹³C (75 MHZ, CDCl₃) \delta (ppm): 22.0, 28.3, 29.3, 39.5, 43.6, 47.5, 126.2, 126.7, 126.9, 127.0, 127.3, 128.9, 129.1, 131.6, 131.7, 132.0, 132.7, 132.8, 133.0, 133.7, 134.4, 133.9, 141.7, 141.7, 166.7, 166.9, 193.4, 193.6; HR-MS (M+H)⁺ m/z 280.27.**

Characterization of mPMF-Pd⁰

CHN analysis: To determine the different element contents in the mesoporous polymer supported Pd nano composite (mPMF-Pd⁰), CHN analysis was done. Elemental analysis of mPMF-Pd⁰ showed that it contained C- 31.12%, N-38.66%, H-3.70% and S-4.31% (S from solvent, DMSO).

Thermal analysis:

The quantitative determination of the organic content and the framework stability of the mPMF and mPMF-Pd⁰ samples are obtained from the thermogravimetric (TG) analysis under N₂ flow. TGA of mPMF-Pd⁰ material is shown in Figure 5. The TGA of this material showed the first weight loss around 150 $^{\circ}$ C due to desorption of physisorbed water. This was followed by a gradual decrease in the weight after 400 $^{\circ}$ C. Thus, this thermal analysis data suggested that mPMF-Pd⁰ sample is stable up to 550 $^{\circ}$ C (Fig. S1).



Figure S1: TGA of mPMF and mPMF-Pd⁰ materials

UV-vis spectroscopy study:

Optical absorption of the as-prepared mPMF-Pd⁰ was investigated in Fig. 6. UV-vis absorbance spectra (Fig. S2) of mPMF exhibits no strong characteristic absorbance, except two peaks at 255 and 308 nm due to π - π * and n- π * transitions respectively. But a distinguishable

change in absorbance is observed in case of mPMF-Pd⁰ material. This material displays a broad shoulder at around 320-390 nm, thereby indicating the formation of Pd NPs.¹



Figure S2: DRS-UV-visible absorption spectra of the mPMF and mPMF-Pd⁰ materials.

FT-IR analysis:

The FT-IR spectrum of mPMF (Fig.S3) shows a broad peak at 3409 cm⁻¹ due to -NHstretching which is shifted to 3405 cm⁻¹ and decreased in intensity after palladium nano loading. The imine (C=N) function of the mesoporous polymer shows two stretches at 1631 and 1199 cm⁻¹. The distinct bands related to the quadrant (1547 cm⁻¹) and semicircle stretching (1474 cm⁻¹) of the triazine ring are present in the spectrum of the mPMF material, indicating the successful incorporation of melamine into the network.²



Figure S3: FT-IR Spectra of mesoporous polymer and mPMF-Pd⁰

Specific surface area:

The surface area of the mPMF-Pd⁰ determined by N₂-adsorption study reveals that mPMF-Pd⁰ exhibits specific surface area of about 94.180 m²/g. The BET surface areas of mPMF is 930 m² g⁻¹ with an average pore size of 15.7 nm.³ Considerable decrease in the surface area to 94.180 m²/g in mPMF-Pd⁰ suggests that palladium nano particles are anchored in the inner surface of the pores (Fig.S4).



Figure S4: N₂ adsorption isotherm of mPMF-Pd⁰



Figure S5: Powder XRD pattern of mPMF material.



Figure S6 Image of mesoporous polymer before and after palladium nano loading.

¹H and ¹³C NMR Spectra of mono carbonylation products (Table 4)





Table 4, entry 3





Table 4, entry 5





Table 4, entry 7





Table 4, entry 9







Table 4, entry 11









Table 4, entry 15





Table 4, entry 17





Table 4, entry1





Table 4, entry3









Table 4, entry 7







Table 4, entry 9







Table 4, entry 11





Table 4, entry 13





Table 4, entry 15



ppm (fl)



Table 4, entry 17





¹H and ¹³C NMR Spectra of double carbonylation products (Table 5)

Table 5, entry 1







Table 5, entry 3







Table 5, entry 5







Table 5, entry 7







Table 5, entry 9







Table 5, entry 1



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Table 5, entry 3



Table 5, entry 4



Table 5, entry 5







Table 5, entry 7



Table 5, entry 8



Table 5, entry 9



Table 5, entry 10



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