Journal of the Serbian Chemical Society
JSCS@tmf.bg.ac.rs • www.shd.org.rs/JSCS
J. Serb. Chem. Soc. 77 (6) S87–S89 (2012) *Supplementary material*

SUPPLEMENTARY MATERIAL TO

Synthesis and antimicrobial activity of novel tetrabromo-bis(substituted benzyl)cycloalkanones

A. F. M. MOTIUR RAHMAN[1]*, MOHAMMAD SAYED ALAM[2,3]** and ADNAN A. KADI[1]

[1]*Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, Riyadh 11451, Saudi Arabia,* [2]*Division of Bioscience, Dongguk University, Geyonju 780–714, Republic of Korea and* [3]*Department of Chemistry, Jagannath Univeristy, Dhaka 1100, Bangladesh*

J. Serb. Chem. Soc. 77 (6) (2012) 717–723

ANALYTICAL AND SPECTRAL DATA OF THE SYNTHESIZED COMPOUNDS

*2,5-Dibromo-2,5-bis(α-bromobenzyl)cyclopentanone (**4a**)*. Yield: 418 mg, 70%; m.p.: 178–180 °C (lit. 175 °C[1]); IR (KBr, cm^{-1}): 3245, 1664, 1213, 1025, 846; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.53–7.50 (4H, *m*, Ar–H), 7.43–7.36 (6H, *m*, Ar–H), 5.57 (2H, s), 3.32 (2H, *ABq*, J_{AB} = 13.3 Hz), 2.42 (2H, *ABq*, J_{AB} = = 13.2 Hz); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 198.35, 137.35, 129.61, 129.03, 128.35, 67.42, 52.26, 31.67; ESI-MS (*m*/*z*): 579.8 $[M(^{79}Br)+H]^+$.

*2,5-Dibromo-2,5-bis(α-bromo-*p*-methylbenzyl)cyclopentanone (**4b**)*. Yield: 462 mg, 76 %; m.p.: 184–185 °C; IR (KBr, cm^{-1}): 3252, 1671, 1312, 1007, 820; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.39 (4H, *d*, *J* = 8.1 Hz, Ar–H), 7.18 (4H, *d*, *J* = 7.9 Hz, Ar–H), 5.54 (2H, *s*), 3.31 (2H, *ABq*, J_{AB} = 13.3 Hz), 2.42 (2H, *ABq*, J_{AB} = 13.2 Hz), 2.37 (6H, *s*, 2×CH_3); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 198.50, 139.06, 134.48, 129.47, 129.08, 67.64, 52.36, 32.76, 21.26; ESI-MS (*m*/*z*): 607.5 $[M(^{79}Br)+H]^+$.

*2,5-Dibromo-2,5-bis(α-bromo-*m*-chlorobenzyl)cyclopentanone (**4c**)*. Yield: 571 mg, 88 %); m.p.: 190–192 °C; IR (KBr, cm^{-1}): 3271, 1665, 1200, 1142, 830; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.50 (2H, *m*, Ar–H), 7.41–7.28 (6H, *m*, Ar–H), 5.49 (2H, *s*), 3.28 (2H, *ABq*, J_{AB} = 13.3 Hz), 2.40 (2H, *ABq*, J_{AB} = 13.0 Hz); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 197.82, 139.18, 134.24, 129.63, 129.62, 129.30, 127.90, 66.78, 51.07, 31.58; ESI-MS (*m*/*z*): 648.0 $[M(^{79}Br)+H]^+$.

*2,5-Dibromo-2,5-bis(α-bromo-*p*-methoxybenzyl)cyclopentanone (**4d**)*. Yield: 595 mg, 93 %; m.p. 170–172 °C; IR (KBr, cm^{-1}): 3301, 1681, 1425, 986, 725; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.43 (4H, *d*, *J* = 8.6 Hz, Ar–H), 6.89 (4H,

*,** Corresponding authors. E-mail: *afmrahman@ksu.edu.sa; **alam_sms@yahoo.com

d, *J* = 8.6 Hz, Ar–H), 5.55 (2H, *s*), 3.82 (6H, *s*, 2×OCH_3), 3.30 (2H, *ABq*, J_{AB} = = 13.0 Hz), 2.41 (2H, *ABq*, J_{AB} = 13.0 Hz); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 198.57, 159.87, 130.83, 129.46, 113.68, 67.98, 55.31, 52.38, 31.73; ESI-MS (*m*/*z*): 640.0 [M(^{79}Br)+H]$^+$.

*2,5-Dibromo-2,5-bis(α-bromo-*p*-fluorobenzyl)cyclopentanone* ***(4e)***. Yield: 542 mg, 85 %; m.p.: 205–207 °C; IR (KBr, cm^{-1}): 3205, 1672, 1201, 1017, 825; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.49 (4H, *dd*, *J* = 8.6, 5.2 Hz, Ar–H), 7.07 (4H, *t*, *J* = 8.6 Hz, Ar–H), 5.53 (2H, *s*), 3.28 (2H, *ABq*, J_{AB} = 13.1 Hz), 2.37 (2H, *ABq*, J_{AB} = 13.2 Hz); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 198.10, 162.70 ($J_{C–F}$ = 247.8 Hz), 133.23 ($J_{C–F}$ = 3.6 Hz), 131.43 ($J_{C–F}$ = 8.4 Hz), 115.45 ($J_{C–F}$ = 21.8 Hz), 67.44, 51.40, 31.61; ESI-MS (*m*/*z*): 615.9 [M(^{79}Br)+H]$^+$.

2,5-Dibromo-2,5-bis(α-bromobenzyl)cyclohexanone ***(4f)***. Yield: 523 mg, 76 %; m.p.: 195–196 °C (lit. 193 °C[15]); IR (KBr, cm^{-1}): 3201, 1672, 1313, 1102, 742; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.54–7.50 (4H, *m*, Ar–H), 7.37–7.32 (6H, *m*, Ar–H), 6.03 (2H, s), 3.07 (2H, *td*, *J* = 14.8, 2.9 Hz), 2.46–2.32 (3H, *m*), 2.03–1.95 (1H, *m*); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 198.13, 136.11, 131.14, 128.85, 127.75, 64.45, 55.30, 33.75, 16.83; ESI-MS (*m*/*z*): 593.9 [M(^{79}Br)+H]$^+$.

*2,5-Dibromo-2,5-bis(α-bromo-*p*-methylbenzyl)cyclohexanone* ***(4g)***. Yield: 412 mg, 79 %; m.p.: 187–189 °C; IR (KBr, cm^{-1}): 3212, 1666, 1210, 1009, 841; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.39 (4H, *d*, *J* = 8.0 Hz, Ar–H), 7.13 (4H, *d*, *J* = 7.9 Hz, Ar–H), 5.99 (2H, *s*), 3.04 (2H, *td*, *J* = 14.6, 2.8 Hz), 2.38–2.31 (3H, *m*), 2.34 (6H, *s*), 2.00–1.95 (1H, *m*); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 198.20, 138.85, 133.24, 130.97, 128.48, 64.77, 55.30, 33.78, 21.19, 16.86; ESI--MS (*m*/*z*): 622.0 [M(^{79}Br)+H]$^+$.

*2,5-Dibromo-2,5-bis(α-bromo-*p*-fluorobenzyl)cyclohexanone* ***(4h)***. Yield: 555 mg, 88 %; m.p.: 155–158 °C; IR (KBr, cm^{-1}): 3195, 1675, 1301, 955, 759; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.52–7.35 (4H, *dd*, *J* = 8.4, 5.3 Hz, Ar–H), 7.03 (4H, *t*, *J* = 8.4 Hz, Ar–H), 5.99 (2H, *s*), 3.00 (2H, *td*, *J* = 12.3, 2.8 Hz), 2.40–2.26 (3H, *m*), 2.00–1.96, (1H, *m*); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 197.92, 162.70 ($J_{C–F}$ = 247.6 Hz), 132.80 ($J_{C–F}$ = 82.5 Hz), 131.93 ($J_{C–F}$ = 3.4 Hz), 114.80 ($J_{C–F}$ = 21.6 Hz), 64.37, 54.33, 33.61, 16.74; ESI-MS (*m*/*z*): 629.9 [M(^{79}Br)+H]$^+$.

*2,5-Dibromo-2,5-bis(α-bromo-*p*-methoxybenzyl)cyclohexanone* ***(4i)***. Yield: 523 mg, 80 %; m.p. 170–172 °C; IR (KBr, cm^{-1}): 3240, 1662, 1201, 980, 744; ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 7.45 (4H, *d*, *J* = 8.7 Hz, Ar–H), 6.86 (4H, *dd*, *J* = 8.8, 2.2 Hz, Ar–H), 5.99 (1H, *s*), 5.93 (1H, *s*) 3.81 (6H, *s*, 2×OCH_3), 3.34–3.25 (1H, *m*), 3.02 (1H, *t*, *J* = 12.88 Hz), 2.39–2.31 (3H, *m*), 2.02–1.90 (1H, *m*); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 198.19, 159.79, 159.76, 132.26, 128.25, 128.08, 113.13, 113.09, 66.72, 65.16, 55.30, 55.21, 53.90, 33.80, 16.89; ESI–MS (*m*/*z*): 654.0 [M(^{79}Br)+H]$^+$.

*2,5-Dibromo-2,5-bis(*α*-bromo-*p*-bromobenzyl)cyclohexanone (**4j**)*. Yield: 638 mg, 85 %; m.p.: 210–212 °C; IR (KBr, cm^{-1}): 3285, 1660, 1201, 955, 885; ^{1}H--NMR (250 MHz, $CDCl_3$, δ / ppm): 7.50 (4H, *d*, *J* = 8.5 Hz, Ar–H), 7.38 (4H, *d*, *J* = 8.6 Hz, Ar–H), 5.95 (2H, *s*), 2.99 (2H, *t*, *J* = 14.6 Hz), 2.40–2.14 (3H, *m*), 2.02–1.96 (1H, *dd*, *J* = 14.0, 3.0 Hz); ^{13}C-NMR (62.5 MHz, $CDCl_3$, δ / ppm): 197.72, 135.11, 132.67, 130.99, 123.11, 63.95, 54.32, 33.65, 16.73; ESI-MS (*m*/*z*): 751.8 $[M(^{79}Br)+H]^+$.

REFERENCES

1. P.-Y. Yeh, *Bull. Chem. Soc. Jpn*. **27** (1954) 60.