Supporting Information

An Efficient Method for the Synthesis of α , α' -Bis(arylmethylidene) Cycloalkanones Catalyzed by Bromodimethylsulfonium Bromide

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Experimental Section

General procedure. All chemicals were purchased from Aldrich Co. and used as received. Melting points were measured using a X-4 apparatus and are uncorrected. IR spectra were obtained using Shimadzu FTIR-8900 spectrometer instrument. NMR spectra were taken with a Varian 400 spectrometer. Elemental analyses were carried out on MT-3 analyzer. Physical and spectral characterization of the products was confirmed by comparison with available literature data.^{S1, S2}

Physical and Spectral Data of the Products

α , α' -bis(benzylidene) cyclopentanone 3a

IR (KBr): 3052, 3017, 2910, 1688, 1625, 1600 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.58–7.60 (m, 6 H), 7.35–7.45 (m, 6 H), 3.12 (s, 4 H). ^{13}C NMR (CDCl_3): δ = 188.1, 144.4, 142.3, 133.3, 128.5, 127.5, 126.7, 22.6. Anal. Calcd for $\text{C}_{19}\text{H}_{16}\text{O}$: C, 87.66; H, 6.19; Found: C, 87.57; H, 6.28.

α, α' -bis(p-methoxyphenylmethylidene) cyclopentanone 3b

IR (KBr): 2934, 1660, 1591, 1550, 1503, 1247, 1020, 835 cm^{-1} . ^1H NMR (CDCl_3): δ = 6.89–7.57 (m, 10 H), 3.84 (s, 6 H), 3.07 (s, 4 H). ^{13}C NMR (CDCl_3): δ = 187.0, 161.2, 143.6, 142.3, 127.2, 114.8, 56.5, 23.3. Anal. Calcd for $\text{C}_{21}\text{H}_{20}\text{O}_3$: C, 78.73; H, 6.29; Found: C, 78.77; H, 6.25.

α, α' -bis(p-methylphenylmethylidene) cyclopentanone 3c

IR (KBr): 2940, 2923, 1658, 1601 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.43–7.50 (m, 6 H), 7.17–7.18 (m, 4 H), 3.03 (s, 4 H), 2.32 (s, 6 H). ^{13}C NMR (CDCl_3): δ = 188.0, 144.2, 142.5, 136.9, 131.8, 128.9, 126.1, 23.6, 20.6. Anal. Calcd for $\text{C}_{21}\text{H}_{20}\text{O}$: C, 87.46; H, 6.99; Found: C, 87.57; H, 6.95.

α, α' -bis(p-chlorophenylmethylidene) cyclopentanone 3d

IR (KBr): 2911, 1692, 1620, 1605, 1580 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.45–7.47 (m, 6 H), 7.33–7.35 (m, 4 H), 3.01 (s, 4 H). ^{13}C NMR (CDCl_3): δ = 187.8, 143.8, 143.6, 133.3, 128.8, 23.5. Anal. Calcd for $\text{C}_{19}\text{H}_{14}\text{Cl}_2\text{O}$: C, 69.32; H, 4.29; Found: C, 69.37; H, 4.35.

α, α' -bis(2,4-dichlorophenylmethylidene) cyclopentanone 3e

IR (KBr): 3071, 2911, 1692, 1614, 1576 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.82 (s, 2 H), 7.48–7.30 (m, 6 H), 2.98 (s, 4 H). ^{13}C NMR (CDCl_3): δ = 187.3, 143.5, 142.6, 134.3,

132.8, 129.7, 23.6. Anal. Calcd for C₁₉H₁₂Cl₄O: C, 57.32; H, 3.04; Found: C, 57.37; H, 3.05.

α, α' -bis(p-nitrophenylmethylidene) cyclopentanone 3f

IR (KBr): 3106, 2844, 1706, 1608, 1522 cm⁻¹. ¹H NMR (CDCl₃): δ = 8.30–8.32 (m, 4 H), 7.65–7.76 (m, 6 H), 3.21 (s, 4 H). ¹³C NMR (CDCl₃): δ = 187.7, 147.7, 143.5, 142.1, 141.0, 127.5, 123.6, 23.6. Anal. Calcd for C₁₉H₁₄N₂O₅: C, 65.14; H, 4.03; Found: C, 65.11; H, 4.10.

α, α' -bis(m-pyridylmethylidene) cyclopentanone 3g

IR (KBr): 3101, 2930, 2850, 1645, 1583, 1512, 1365, 1158, 818 cm⁻¹. ¹H NMR (CDCl₃): δ = 8.87 (s, 2H), 8.61 (s, 2H), 7.92 (d, J = 7.7 Hz, 2H), 7.59 (s, 2H), 7.42–7.44 (m, 4 H), 3.19 (s, 2 H). ¹³C NMR (CDCl₃): δ = 187.0, 155.8, 149.6, 149.4, 136.7, 125.5, 121.1, 23.5. Anal. Calcd for C₁₇H₁₄N₂O: C, 77.84; H, 5.38; Found: C, 77.81; H, 5.30.

α, α' -bis(p-hydroxyphenylmethylidene) cyclopentanone 3h

IR (KBr): 3109, 2845, 1700, 1604, 1500 cm⁻¹. ¹H NMR (DMSO-d₆): δ = 9.99 (s, 2 H), 6.82–7.49 (m, 10 H), 2.97 (s, 4 H). ¹³C NMR (CDCl₃): δ = 187.4, 156.2, 143.6, 142.1, 127.6, 115.8, 23.4. Anal. Calcd for C₁₉H₁₆O₃: C, 78.06; H, 5.52; Found: C, 78.11; H, 5.58.

α, α' -bis(styrylmethylidene) cyclopentanone 3i

IR (KBr): 3026, 1669, 1618, 1584 cm⁻¹. ¹H NMR (CDCl₃): δ = 7.52 (d, J = 7.2 Hz, 4 H), 7.23–7.46 (m, 8 H), 6.98–7.01 (m, 4 H), 2.93 (s, 4 H). ¹³C NMR (CDCl₃): δ = 187.2, 147.5, 143.6, 134.9, 128.4, 127.7, 24.6. Anal. Calcd for C₂₃H₂₀O: C, 88.43; H,

6.45; Found: C, 88.51; H, 6.54.

α , α' -bis(2-furylmethylidene) cyclopentanone 3j

IR (KBr): 3133, 2917, 1682, 1624, 1600, 1290, 1238, 755 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.60 (s, 2 H), 7.36 (s, 2 H), 6.71 (d, J = 3.3 Hz, 2 H), 6.55 (s, 2 H), 3.09 (s, 4 H). ^{13}C NMR (CDCl_3): δ = 187.8, 155.3, 149.6, 142.1, 127.7, 111.8, 20.8. Anal. Calcd for $\text{C}_{15}\text{H}_{12}\text{O}_3$: C, 74.99; H, 5.03; Found: C, 74.91; H, 5.14.

α , α' -bis(benzylidene)cyclohexanone 3k

IR (KBr): 3024, 2926, 1657, 1609, 1570, 1440, 1268, 1144, 773 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.81 (s, 2 H), 7.33–7.48 (m, 10 H), 2.96 (t, J = 6.4 Hz, 4 H), 1.83–1.76 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.2, 144.3, 141.5, 134.7, 128.6, 127.5, 27.8. Anal. Calcd for $\text{C}_{20}\text{H}_{18}\text{O}$: C, 87.56; H, 6.61; Found: C, 87.61; H, 6.74.

α , α' -bis(p-methoxyphenylmethylidene) cyclohexanone 3l

IR (KBr): 2937, 1661, 1595, 1552, 1505, 1248, 1021, 833 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.77 (s, 2 H), 7.48–6.96 (m, 8 H), 3.78 (s, 6 H), 2.88 (t, J = 5.4 Hz, 4 H), 1.73–1.75 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.4, 161.2, 144.3, 141.5, 127.2, 114.0, 56.0, 27.5. Anal. Calcd for $\text{C}_{22}\text{H}_{22}\text{O}_3$: C, 79.02; H, 6.63; Found: C, 79.11; H, 6.70.

α , α' -bis(p-methylphenylmethylidene) cyclohexanone 3m

IR (KBr): 2942, 2918, 1660, 1600 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.78 (s, 2 H), 7.18–7.39 (m, 8 H), 2.92 (t, J = 5.6 Hz, 4 H), 2.39 (s, 6 H), 1.75–1.79 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.1, 144.3, 141.6, 131.9, 136.9, 129.1, 27.8; 20.9. Anal. Calcd for $\text{C}_{22}\text{H}_{22}\text{O}$: C, 87.38; H, 7.33; Found: C, 87.31; H, 7.30.

α , α' -bis(p-chlorophenylmethylidene) cyclohexanone 3n

IR (KBr): 2920, 1693, 1624, 1603, 1581 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.75 (s, 2H), 7.40 (m, 8H), 2.93-2.89 (m, 4H), 1.84-1.81(m, 2H). ^{13}C NMR (CDCl_3): δ = 189.8, 136.4, 135.7, 134.6, 134.3, 131.5, 128.6, 28.3, 22.8. Anal. Calcd for $\text{C}_{20}\text{H}_{16}\text{Cl}_2\text{O}$: C, 69.98; H, 4.70; Found: C, 69.91; H, 4.63.

α, α' -bis(2,4-dichlorophenylmethylidene) cyclohexanone 3o

IR (KBr): 3096, 2968, 1658, 1593, 1575, 1464, 1138, 826 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.76 (s, 2H), 7.40–7.18 (m, 6H), 2.68 (t, J = 5.6 Hz, 4H), 1.72–1.68(m, 2H). ^{13}C NMR (CDCl_3): δ = 187.6, 144.4, 134.7, 133.4, 129.3, 126.8, 27.8. Anal. Calcd for $\text{C}_{20}\text{H}_{14}\text{Cl}_4\text{O}$: C, 58.29; H, 3.42; Found: C, 58.31; H, 3.53.

α, α' -bis(p-nitrophenylmethylidene) cyclohexanone 3p

IR (KBr): 3107, 2930, 1705, 1600, 1515, 1342, 850 cm^{-1} . ^1H NMR (CDCl_3): δ = 8.3 (s, 2 H), 8.2–7.58 (m, 8 H), 2.94 (t, J = 6.0 Hz, 4 H), 1.87–1.85 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.8, 147.6, 144.3, 141.5, 141.0, 127.1, 123.5, 27.6. Anal. Calcd for $\text{C}_{20}\text{H}_{16}\text{N}_2\text{O}_5$: C, 65.93; H, 4.43; Found: C, 65.89; H, 4.53.

α, α' -bis(p-dimethylaminophenylmethylidene) cyclohexanone 3q

IR (KBr): 2932, 2852, 1648, 1580, 1515, 1361, 1156, 815 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.76 (s, 2H), 7.62–6.87 (m, 8 H), 3.02 (s, 12H), 2.95 (t, J = 6.1 Hz, 4 H), 1.81–1.83 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.7, 144.2, 143.7, 141.5, 127.1, 113.0, 43.5, 27.6. Anal. Calcd for $\text{C}_{24}\text{H}_{28}\text{N}_2\text{O}$: C, 79.96; H, 7.83; Found: C, 79.89; H, 7.89.

α, α' -bis(p-hydroxyphenylmethylidene) cyclohexanone 3r

IR (KBr): 3012, 2930, 2842, 1640, 1500, 1519, 1331, 1160, 818 cm^{-1} . ^1H NMR (CDCl_3): δ = 9.96 (s, 2 H), 7.54 (s, 2 H), 7.49 (s, 4 H), 6.84 (s, 4 H), 2.86 (t, J = 6.2

Hz, 4 H), 1.71–1.73 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.0, 156.5, 144.4, 141.5, 127.6, 115.6, 27.6. Anal. Calcd for $\text{C}_{20}\text{H}_{18}\text{O}_3$: C, 78.41; H, 5.92; Found: C, 78.49; H, 5.89.

α, α' -bis(3-phenylallylidene) cyclohexanone 3s

IR (KBr): 3052, 2900, 1690, 1600 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.44–7.40 (m, 6 H), 7.30–6.90 (m, 10 H), 2.74–2.72 (t, J = 5.6 Hz, 4 H), 1.75 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.3, 148.3, 142.7, 134.8, 128.2, 127.8, 126.1, 125.3, 27.8. Anal. Calcd for $\text{C}_{24}\text{H}_{22}\text{O}$: C, 88.31; H, 6.79; Found: C, 88.39; H, 6.87.

α, α' -bis(2-thienylmethylidene) cyclohexanone 3t

IR (KBr): 3140, 2927, 1678, 1621, 1601, 1233, 750 cm^{-1} . ^1H NMR (CDCl_3): δ = 7.99 (s, 2 H), 7.54 (d, J = 4.6 Hz, 2 H), 7.39 (d, J = 2.5 Hz, 2 H), 7.16 (t, J = 3.7 Hz, 2 H), 2.94 (t, J = 4.7 Hz, 4 H), 2.00–1.97 (m, 2 H). ^{13}C NMR (CDCl_3): δ = 187.3, 150.6, 141.5, 136.6, 130.3, 127.8, 126.4, 27.4. Anal. Calcd for $\text{C}_{16}\text{H}_{14}\text{OS}_2$: C, 67.10; H, 4.93; Found: C, 67.19; H, 4.88.

References

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