Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information

2,2-Dimethyl cyclopentanones by acid catalyzed ring expansion of isopropenylcyclobutanols. A short synthesis of (±)-α-cuparenone and (±)-herbertene

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Preparation of cyclopropyl carbinols 2a-f. Typical procedure.

To a solution of cyclopropylphenylsulfide (1.8 g, 12 mmol) in dry THF (40 mL), under Argon, n-BuLi (7.5 mL, 12 mmol, 1.6 M solution in hexane) was added dropwise at 0°C. After stirring for 5h the resulting mixture was cooled at –78°C and the ketone or the aldehyde (12 mmol) was added. After 14h, the reaction mixture was quenched with saturated aqueous NH_4Cl (20 ml) and extracted with diethyl ether, dried on sodium sulfate and concentrated under reduced pressure. The crude oil was chromatographed on silica gel with light petroleum / diethyl ether (10:1) to give **2a-e,**[1] **f**.

1- (3-Methylphenyl)-1-[1-phenylthiocyclopropyl]ethanol. (2f)

Yellow oil. Yield 68 %. IR (neat): 3450 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 0.93-0.97 (m, 1H), 1.02-1.04 (m, 1H), 1.16-1.21 (m, 1H), 1.24-1.29 (m, 1H), 1.60 (s, 3H), 2.28 (s, 3H), 2.63 (br s, 1H), 6.98-7.74 (m, 9H). ^{13}C NMR ($CDCl_3$) δ: 13.3, 15.2, 21.5, 27.3, 36.4, 76.4, 123.1, 125.7, 126.8, 127.7, 128.2, 129.3, 133.7, 137.1, 145.4.

MS (*m/z*): 284 (M^+ (14)), 266 (49), 239 (9), 174 (8), 161 (9), 150 (86), 135 (100), 117 (94), 105(16), 91(29), 77(10).

General procedure for preparation of cyclobutanones 3a-f.[2]

A solution of cyclopropylphenylthio carbinol **2a-f** (2.9 mmol) and PTSA (5 g, 2.9 mmol) in wet benzene (70 mL) was stirred at reflux for 5 h. The resulting mixture was cooled to room temperature and washed with a 1,0 M solution of NaOH and brine. The resulting organic phase was dried on sodium sulfate and

concentrated in vacuo. The crude oil chromatographed with light petroleum / diethyl ether (5:1) gave **3a-f**.

General procedure for the synthesis of cyclobutanols 6a-l.

To a stirred solution of cyclobutanone **3a-h** (2.8 mmol) in dry THF (15 mL), under Argon, isopropenylmagnesium bromide [prepared as usual from magnesium turning (201 mg, 8.4 mmol) and 2-bromopropene (0.75 mL, 8.4 mmol)] in dry THF was added dropwise at -20°C . The solution was allowed to gradually reach room temperature and stirring was continued for 14 h. The reaction mixture was then quenched with saturated aqueous NH_4Cl and extracted with diethyl ether. The organic layer was dried on sodium sulfate and concentrated under vacuum to give crude the cyclobutanols **6** which were chromatographed with a 10:1 to 5:1 light petroleum / diethyl ether.

1-Isopropenyl-2-methyl-2-(2-phenylethyl)cyclobutanol. (6a)

Overall yield 71%. 35:65 of mixture *trans*:*cis* of two diastereoisomers. Major *cis* isomer: colourless oil. IR (neat): 3450 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.05 (s, 3H), 1.36-1.44 (m, 1H), 1.58 (s, 1H), 1.62-1.79 (m, 2H), 1.71 (s, 3H), 1.96 (q, 1H, J = 8.7Hz), 2,06-2.16 (m, 1H); 2.40-2.65 (m, 3H), 4.88 (s, 1H), 4.91 (s, 1H), 7.19-7.26 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 18.9, 21.5, 27.6, 28.4, 30.6, 37.7, 45.4, 82.4, 111.4, 125.5, 128.2, 128.3, 143.1, 147.1. MS (*m/z*): 230 (M^+ (5)), 215 (3), 187 (8), 160 (7), 146 (16), 139 (5), 125 (11), 118 (6), 111 (21,6), 91 (100), 84 (70), 77 (6), 69 (43), 65 (13), 55 (10), 43 (25), 41 (30).

Minor *trans* isomer: colourless oil. IR (neat): 3450 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.23 (s, 3H), 1.50-1.83 (m, 6H), 1.69 (br s, 1H), 1.73 (s, 3H), 2.40-2.60 (m, 2H), 4,90 (br s, 2H), 7.14-7.26 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 19.5, 26.4, 29.4, 29.7, 30.9, 39.0, 46.4, 81.8, 111.6, 125.6, 128.3, 128.3, 143.1, 146.6. MS (*m/z*): 230 (M^+ (6)), 215 (3), 187 (8), 160 (7), 146 (16), 139 (5), 125 (11), 118 (6), 111 (22), 91 (100), 84 (70), 77 (6), 69 (43), 65 (13), 55 (10), 43 (25), 41 (30).

***Cis* -1-isopropenyl-2-(2-phenylethyl)cyclobutanol. (6b)**

Colourless oil. Yield 98 %. IR (neat): 3450 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.66-1.92 (m, 5H), 1.74 (s, 3H), 2.12-2.22 (m, 5H), 4,77 (br s, 1H), 4.88 (br s, 1H), 7.14-7.27 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 18.1, 21.3, 31.3, 31.5, 33.2, 42.2, 79.6, 109.2, 125.6, 128.2, 128.3, 142.5, 149.2. MS (*m/z*): 216 (M^+ (6)), 198 (6), 183 (6), 173 (7), 142 (4), 130 (12), 112 (15), 91 (100), 84 (60), 77 (9), 69 (44), 65 (15), 55 (10).

1-Isopropenyl-2-[1-methyl-2-(phenysulfanyl)ethyl]cyclobutanol.(6c)

Colourless oil. Yield 98%. 50:50 Mixture of two *cis* separable diastereoisomers. First diastereoisomer. IR (neat): 3410 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 0.95 (d, 3H, *J* = 6.6 Hz), 1.71-1.92 (m, 4H), 1.82 (s, 3H), 2.03-2.18 (m, 2H), 2.35-2.44 (m, 1H), 2.53 (dd, 1H, *J* = 7.8 Hz, *J* = 13.2 Hz), 3.04 (dd, 1H, *J* = 4.8 Hz, *J* = 13.2 Hz), 4.82 (s, 1H), 5.01 (s, 1H), 7.20-7.35 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 16.6, 18.9, 21.2, 31.7, 33.9, 39.1, 46.9, 79.9, 109.3, 125.7, 128.8, 128.9, 137.0, 149.4. MS (*m/z*): 262 (M^+ (21)), 178 (23), 153 (10), 123 (100), 110 (29), 77 (11).

Second diastereoisomer. IR (neat): 3410 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 0.96 (d, 3H, *J* = 6.6 Hz), 1.71-1.92 (m, 4H), 1.80 (s, 3H), 2.03-2.18 (m, 2H), 2.38-2.45 (m, 1H), 2.60 (dd, 1H, *J* = 8.7 Hz, *J* = 12.6 Hz), 3.07 (dd, 1H, *J* = 4.8 Hz, *J* = 12.6 Hz), 4.82 (br s, 1H), 4.99 (s, 1H), 7.15-7.35 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 17.8, 18.9, 21.1, 31.8, 33.8, 38.6, 46.6, 80.5, 109.4, 125.6, 128.8, 128.9, 137.4, 149.2. MS (*m/z*): 262 (M^+ (9), 178 (18), 153 (10), 123 (100), 110 (27), 77 (11).

***Cis*-1-Isopropenyl-2-(4-metylphenyl)cyclobutanol. (6d)**

Colourless oil. Yield 95 % . IR (neat): 3300 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.81 (s, 3H), 1.92-2.00 (m, 1H), 2.05-2.14 (m, 1H), 2,32 (s, 3H), 2.34-2.50 (m, 3H), 3.76 (t, 1H, *J* = 8.4), 4.86 (br s, 1H), 5.01 (br s, 1H), 7.15 (m, 4H). ^{13}C NMR ($CDCl_3$) δ: 18.5, 22.1, 28.4, 31.1, 47.3, 82.4, 109.9, 126.7, 128.4, 134.5, 138.7, 146.9.

MS (*m/z*): 202 (M^+ (4)), 174 (2), 159 (11), 131 (3), 118 (100), 115 (8), 91 (8), 69 (3), 41 (5).

1-Isopropenyl-2-methyl-2-(4-metylphenyl)cyclobutanol. (6e)

Colourless oil. Yield 76%. 64:36 Mixture of two *cis*:*trans* separable diastereoisomers. Major *cis* diastereoisomer. IR (neat): 3410 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.38 (s, 3H), 1.61-1.70 (m, 1H), 1.65 (br s, 1H), 1.76-1.84 (m, 1H), 1.78 (s, 3H), 2.33 (s, 3H), 2.54-2.75 (m, 2H), 5.02 (br s, 1H), 5.04 (br s, 1H), 7.16-7.32 (m, 4H). ^{13}C NMR ($CDCl_3$) δ: 19.4, 20.9, 25.7, 26.9, 28.4, 50.8, 82.3, 112.6, 127.3, 128.1, 136.0, 140.0, 146.4. MS (*m/z*): 216 (M^+ (38)), 201 (10), 183 (7), 171 (18), 159 (35), 145 (79), 132 (100), 117 (30), 105 (36), 91 (35), 77 (15).

Minor *trans* diastereoisomer. IR (neat): 3460 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.39 (s, 3H), 1.53 (s, 3H), 2.09 (br s, 1H), 2.16-2.43 (m, 4H), 2.29 (s, 3H), 4.74 (br s, 1H), 5.07 (br s, 1H), 6.99-7.06 (m, 4H). ^{13}C NMR ($CDCl_3$) δ: 19.6, 20.9, 25.7, 27.1, 32.1, 51.5, 81.2, 110.9, 125.5, 128.3, 136.0, 144.2, 147.5. MS (*m/z*): 216 (M^+

(63)), 201 (13), 183 (6), 173 (11), 159 (14), 145 (100), 132 (75), 117 (25), 105 (23), 91 (24), 77 (10).

1-Isopropenyl-2-methyl-2-(3-metylphenyl)cyclobutanol. (6f)

Inseparable 50:50 mixture of two *cis*:*trans* diastereoisomers. Colourless oil. Yield 70 %. IR (neat): 3450 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.48 (s, 3H), 1.64 (s, 3H), 1.93 (br s, 1H), 2.07-2.17 (m, 2H), 2.39-2.45 (m, 2H), 2.33 (s, 3H), 2.39-2.46 (m, 2H), 2.68 (t, 1H, J = 10.5 Hz), 2.85 (t, 1H, J = 9.6 Hz), 4.81 (br s, 1H), 4.88 (br s, 1H), 6.97-7.23 (m, 4H). MS (*m/z*): The same for the two isomers. 216 (M^+ (21)), 173 (12), 145 (100), 131 (48), 117 (34), 105 (17), 91 (18), 77 (5).

1-Isopropenyl-2-[(3-methylphenoxy)methyl]-2-methylcyclobutanol. (6h)

Global yield 80 %. 75:25 Mixture of two *trans*:*cis* diastereoisomers. *Trans*-isomer. Yellow oil. IR (neat): 3440 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 1.33 (s, 3H), 1.34-1.70 (m, 1H), 1.79 (s, 3H), 1.75-1.91 (m, 2H), 1.94 (s, 1H), 2.30 (s, 3H), 2.45-2.54 (m, 1H), 3.67, 3.72 (AB q, 2H, J = 9.0 Hz), 4.90 (s, 2H), 6.66-6.74 (m, 3H), 7.11-7.17 (m, 1H). ^{13}C NMR ($CDCl_3$) δ: 18.4, 19.1, 21.4, 25.0, 29.4, 46.6, 72.5, 80.5, 111.2, 111.4, 115.3, 121.3, 129.0, 139.3, 146.6, 159.3.MS (*m/z*): 246 (M^+ (2)), 162 (16), 147 (58), 138 (129, 119 (5), 108 (100), 91 (10), 77(8).

Cis-isomer: IR (neat): 3420 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: (data worked out from the NMR spectra of the diastereoisomeric mixture). 1.26 (s, 3H), 1.11-1.43 (m, 1H), 1.87 (s, 3H), 175-1.91 (m, 3H), 1.94 (s, 1H), 2.32 (s, 3H), 2.46-2.58 (m, 1H), 3.87, 4.17 (AB q, 2H, J = 9.0 Hz), 4.90 (s, 2H), 6.66-7.18 (m, 4H). MS (*m/z*): the same for the two isomers.

(1R,2R)-2-[(4S)-2,2-Dimethyl-1,3-dioxolan-4yl]-1-isopropenyl-cyclobutanol. (6l)

Colourless oil. Yield 98 %. $[\alpha]_D^{24}$ = +50.29 (c 4.01, $CHCl_3$). IR (neat): 3440 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.35 (s, 3H), 1.41 (s, 3H), 1.59-1.68 (m,1H), 1.72-1.78 (m, 1H), 1.80 (s, 3H), 2.02-2.09 (m, 1H), 2.20-2.27 (m, 1H), 2.58 (q, 1H, J = 8.1 Hz), 3.61 (dd, 1H, J = 6.9 Hz and J = 8.1 Hz), 4.07 (dd, 1H, J = 6.3 Hz and J = 8.1 Hz), 4.44 (dt, 1H, J = 6.9 Hz and J = 8.7 Hz), 4.84 (br s, 1H), 5.04 (br s, 1H). ^{13} C NMR ($CDCl_3$) δ: 16.4, 17.6, 25.6, 26.9, 31.1, 45.2, 67.8, 76.2, 78.4, 109.2, 109.7, 148.8. MS (*m/z*): 197 (M^+- 15 (4)), 154 (12), 137 (11), 109 (33), 95 (18), 84 (37), 69 (95), 59 (44), 43 (100).

General procedure for the preparation of cyclopentanones 7a-l.

A solution of the isopropenyl cyclobutanols **6a-h** (0.68 mmol) and PTSA (170 mg, 0.68 mmol,) in dry benzene (10 mL) was stirred at reflux for 30 min. The resulting reaction mixture was washed with saturated aqueous $NaHCO_3$ and brine. The organic layer was dried on sodium sulfate and concentrated in vacuo. The residue was chromatographed on silica gel with light petroleum / diethyl ether (10:1 to 5:1) as eluent to give pure cyclopentanones **7**.

2,2,3-Trimethyl-3-(2-phenylethyl)cyclopentanone. (7a)

Yellow oil. Yield 71%. IR (neat): 1725 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 0.92 (s, 3H), 0.95 (s, 3H), 0.98 (s, 3H), 1.57-1.70 (m, 2H), 1.73-1.95 (m, 2H), 2.25-2.34 (m, 2H), 2.56-2.67 (m, 2H), 7.17-7.32 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 18.6, 19.8, 21.2 29.7, 30.8, 33.8, 38.9, 43.5, 52.6, 125.8, 128.2, 128.4, 142.7, 223.5. MS (*m/z*): 230 (M^+ (66)), 215 (2), 197 (3), 187 (2), 169 (2), 157 (2), 144 (100), 139 (13), 129 (55), 125 (16), 117(8), 105 (10), 91 (66), 83 (31), 70 (18), 55 (53), 43 (26), 41 (38).

2,2-Dimethyl-3-(2-phenylethyl)cyclopentanone. (7b)

Colourless oil**.** Yield 98%. IR (neat): 1770 cm^{-1} ^1H NMR ($CDCl_3$) δ: 0.83 (s, 3H), 1.02 (s, 3H), 1.49-1.61 (m, 2H), 1.74-1.85 (m, 2H), 2.11-2.18 (m, 2H), 2.36-2.44 (m, 1H), 2.54-2.64 (m, 1H), 2.74 (m, 1H), 7.17-7.32 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 17.9, 22.6, 24.9, 31.7, 34.0, 36.2, 46.9, 47.9, 125.8, 128.2, 128.4, 142.2, 223.6. MS (*m/z*): 216 (M^+ (90)), 198(6), 172 (7),131 (15), 107 (53), 91 (100), 77 (10).

2,2-Dimethyl-3-[1-methyl-2-(phenysulfanyl)ethyl]cyclopentanone. (7c)

Inseparable 50:50 mixture of two diastereoisomers. Colourless oil. Yield 98 %. IR (neat): 1780 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 0.85 (d, 3H, J = 10.2 Hz), 1.13 (d, 3H, J = 15 Hz), 1.06-2.42 (m, 12H), 1.25 (s, 6H), 2.78-2.93 (m, 2H), 3.06-3.21 (m, 2H), 7.14-7.36 (m, 10H). First diastereoisomer: MS (*m/z*): 262 (M^+ 83), 247 (4), 150 (11), 135 (10), 123 (86), 110 (100), 97 (20), 83 (32), 69 (53). Second diastereoisomer: MS (*m/z*): 262 (M^+ 72), 247 (4), 150 (11), 137 (10), 123 (62), 110 (100), 97 (20), 83 (24), 69 (36).

2,2-Dimethyl-3-(4-methylphenyl)cyclopentanone. (7d)

Yellow oil. Yield 95 %. IR (neat): 1770 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.08 (s, 3H), 1.16 (s, 3H), 1.80-2.10 (m, 2H), 2.24-2.42 (m, 2H), 2.32 (s, 3H), 3.37 (dd, 1H, J = 10.9 Hz, J = 9 Hz,), 7.07-7.25 (m, 5H). ^{13}C NMR ($CDCl_3$) δ:. 21.0, 23.7, 24.9, 28.1, 36.6, 45.2, 54.6, 127.9, 129.3, 135.9, 136.3, 221.5. MS (*m/z*): 202 (M^+

(100)), 187 (9), 169 (13), 159 (27), 145 (19), 131 (77), 118 (45), 105 (24), 91 (29), 77 (11).

2,2,3-Trimethyl-3-[(3-methylphenoxy)methyl]cyclopentanone. (7h)

Yellow oil. Yield 80 %. IR (neat): 1770 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 0.96 (s, 3H), 1.02 (s, 3H), 1.06 (s 3H), 1.86.1.89 (m, 1H), 1.99-2.04 (m, 1H), 2.28 (s, 3H), 2.35.2.47 (m, 2H), 3.72, 3.78 (ABq, 2H, J = 9.0 Hz), 6.62-6.76 (m, 3H), 7.13 (t, 1H, J = 7.5 Hz).^{13}C NMR ($CDCl_3$) δ: 14.1, 17.8, 19.2, 22.7, 29.4, 31.9, 44.5, 50.5, 74.4, 110.9, 115.2, 121.7, 129.1, 139.5, 158.6, 222.2.

MS (*m/z*): 246 (M^+ (17)), 139 (4), 121(5), 108 (100), 83 (10), 69 (23), 55 (40).

(3S)-3[(4S)-2,2-dimethyl-1,3-dioxolan-4yl]-2,2-dimethylcyclopentanone. (7l)

Colourless oil. Yield 84 % . $[\alpha]_D^{24}$ = -5.36 (c 1.119, $CHCl_3$). IR (neat): 1740 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 0.99 (s, 3H), 1.19 (s, 3H), 1.35 (s, 3H), 1.40 (s, 3H), 1.46-1.54 (m, 1H), 1.77-1.85 (m, 1H), 1.92-2.02 (m, 1H), 2.14-2.27 (m, 1H), 2.37-2.46 (m, 1H), 3.61-3.68 (m, 1H), 4.03-4.11(m,2H). ^{13}C NMR ($CDCl_3$) δ: 18.3, 21.5, 24.5, 25.8, 26.9, 36.3, 48.0, 50.6, 68.6, 76.9, 109.1, 222.9. MS (*m/z*): 212 (M^+ (2)), 197 (77), 155 (11), 137 (52), 109 (26), 101 (27), 95 (28), 83 (19), 69 (26), 55 (25), 43 (100).

1-[(1Z) and (1E)-1-methyl-1-propenyl]-2-(2-phenylethyl)cyclobutanols. (8b)

Yellow oil. Yield 62 %. Inseparable mixture of two geometric isomers.(70:30) IR (neat): 3440 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.60 (s, 3H), 1.61 (s, 3H), 1.63-1.65 (m, 6H), 1.69-2.69 (m, 10H), 5.23 (q, 1H, J = 6.3 Hz), 5.43 (q, 1H, J = 6.0 Hz), 7.14-7.29 (m, 5H). Major isomer: MS (*m/z*): 230 (M^+ (3)), 215 (10), 202 (4), 185 (3), 167 (3), 126 (8), 111 855), 98 (56), 91 (28), 83 (100). Minor isomer: MS (*m/z*): 230 (M^+ (3)), 215 (209, 197 (2), 185 (1), 126 (24), 111 (67), 98 (74), 91 (60), 83 (100).

2-bromo-6-phenyl-1-hexene : Copper (I) iodide (247 mg, 1.3 mmol) in dry THF (20 mL) was treated at 0°C with 2,3-dibromopropene (5 g, 26 mmol) and stirred for 1h. Then, 3-phenylmagnesium chloride [prepared as usual from magnesium turning (624 mg, 26 mmol) and 3-phenyl-1-chloropropane (4 g, 26 mmol)] in dry THF (30 mL)] was added dropwise at 0°C. The reaction mixture was then stirred for 2 h and then quenched with saturated aqueous NH_4Cl and extracted with diethyl ether. The organic layer was dried on sodium sulfate and concentrated under vacuum to afford an oil which was purified by flash cromatography with pentane. Colourless oil. Yield 95 %. ^1H NMR ($CDCl_3$) δ: 1.58–1.65 (m, 2H),

202-2.10 (m, 2H), 2.58-2.63 (m, 2H), 2.76 (t, 2H, *J* = 7.8 Hz), 3.50 (t, 1H, *J* = 6.3 Hz), 5.37 (s, 1H), 5.52 (s, 1H), 7.15-7.30 (m, 5H). ^{13}C NMR ($CDCl_3$) δ: 27.3, 29.5, 34.0, 41.2, 116.4, 125.7, 128.3, 128.3, 134.5, 142.2. MS (*m/z*): 240 (M^+ (10)), 238 (10), 159 (100),143 (23), 12935), 117 (98), 105 (65), 91 (96), 81 (60), 65 (82), 51 (30).

***Cis*-2-decyl-1-[1-(4-phenylbutyl)vinyl]cyclobutanol**. **(9i)**

To a stirred solution of 2-bromo-6-phenyl-1-hexene (908 mg, 3.8 mmol) in THF (20 mL), at -78°C was added tert-butyllithium (4.5 mL, 7.6 mmol, 1.7 M in hexane). After 2h, the cyclobutanone **3i**[3] (800 mg, 3.8 mmol) in 5 mL of THF was added and the resulting solution was stirred for 3h at -78°C and then at room temperature for 10 h. The reaction mixture was quenched with saturated aqueous NH_4Cl and extracted with diethyl ether. The organic layer was dried on sodium sulfate, concentrated under vacuum and the crude cyclobutanol **9i** was purified by chromatography on silica gel (eluent light petroleum / diethyl ether 10:1). Yield 76% .Colourless oil. IR (neat): 3430 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 0.85 (t, 3H, *J* = 5.1 Hz), 1.12-1.70 (m, 26H), 1.81-1.87 (m, 2H), 2.08-2.16 (m, 2H), 2.63 (t, 2H, *J* = 7.2 Hz), 4.78 (s, 1H), 4.98 (s, 1H), 7.13-7.29 (m, 5H).^{13}C NMR ($CDCl_3$) δ: 14.1, 21.7, 22.6, 22.7, 27.1, 28.0, 29.3, 29.4, 29.7, 29.9, 30.5, 31.4, 31.9, 32.1, 35.9, 43.0, 80.2, 107.8, 125.6, 128.2, 128.4, 142.6, 153.6. MS (*m/z*): 370 (M^+ (2)), 342 (2), 279 (3), 251 (4), 238 (8), 187 (4), 169 (6), 144 (13), 130 (16), 117 (22), 104 (9), 91 (100).

***Cis*-2-Decyl-1-vinylcyclobutanol**. **(10i)**

Same procedure used for compounds **6**.

Colourless oil. Yield 81 %. IR (neat): 3410 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 1.75-2.25 (m, 26H), 2.41 (br s, 1H), 4.99 (dd, 1H, *J* = 10.8 Hz, *J* = 1.2 Hz), 5.18 (dd, 1H, *J* = 1.2 Hz, *J* = 17.2 Hz), 6.04 (dd, 1H, *J* = 10.8 Hz, *J* = 17.2 Hz).^{13}C NMR ($CDCl_3$) δ: 14.0, 21.0, 22.6, 27.1, 29.3, 29.0, 29.3, 29.6, 29.8, 31.8, 32.9, 44.8, 46.3, 72.6, 110.7, 144.1. MS (*m/z*): 238 (M^+ (1)), 209 (5), 182 (3), 167 (4), 139 (11), 112 (13), 97 (15), 70 (100), 55 (55).

3-Decyl-2-methyl-2-(4-phenylbutyl)cyclopentanone. (11i)

Colourless oil. Yield 84 % . IR (neat): 1740 cm^{-1}. Spectral data of inseparable 60:40 mixture of two *cis*:*trans* isomers. ^{1}H NMR ($CDCl_3$) δ: 0.81 (s, 3H), 0.88 (t, 3H, *J* = 5.3 Hz), 1.13-1.70 (m, 22H), 1.92-2.41 (m, 4H), 2.54-2.61 (m, 2H), 7.13-7.28 (m, 5H).

Major isomer. MS (*m/z*): 370 (M^+ (1)), 355 (4), 279 (3), 261 (2), 238 (31), 211 (5), 131 (6), 117 (20), 97 (100), 91 (96).

Minor isomer. MS (*m/z*): 370 (M^+ (1)), 355 (8), 279 (3), 238 (28), 211 (5), 131 (6), 117 (20), 97 (97), 91 (100).

References

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