Modern Friedel-Crafts chemistry. Part 31.[†] An efficient synthetic approach to mono-, di- and triphenylindane derivatives via direct Friedel-Crafts cyclialkylation of selected phenylated alkanols

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Abstract

Facile procedures for the synthesis of mono-, di- and triphenylindanee derivatives from the alcohols **1-4** are described thus treatment with 85% H_2SO_4, $AlCl_3/CH_3NO_2$, H_3PO_4 and/or PPA under varying conditions produced 1,1-dimethyl-3-phenylindanee **6** from 2-methyl-4,4-diphenyl-2-butanol **1**, 3,3-dimethyl-1,1-diphenylindanee **9** from 2-methyl-4,4,4-triphenyl-2-butanol **2**, 1-methyl-1,3-diphenylindanee **12** from 2,4,4-triphenyl-2-butanol **3** and 1,1,3-triphenylindanee **15** from 1,1,3,3-tetraphenyl-1-propanol **4**. The starting and final products were characterized by elemental, IR, ^{1}H NMR and MS analyses.

Keywords: Friedel-Crafts cyclialkylation, 1,1-dimethyl-3-phenylindane, 3,3-dimethyl-1,1-diphenylindane, 1-methyl-1,3-diphenylindane, 1,1,3-triphenylindane, di-, tri- and tetraphenyl alkanols

Introduction

Intramolecular Friedel-Crafts reactions promoted by Bronsted and Lewis acid catalysts provide good routes for the construction of carbo- and heteropolycyclic systems.[1-3] In recent years, we have directed part of our research efforts to study both the synthetic utilities and the mechanistic aspects of these important reactions.[4-6] These efforts not only illustrated the broad

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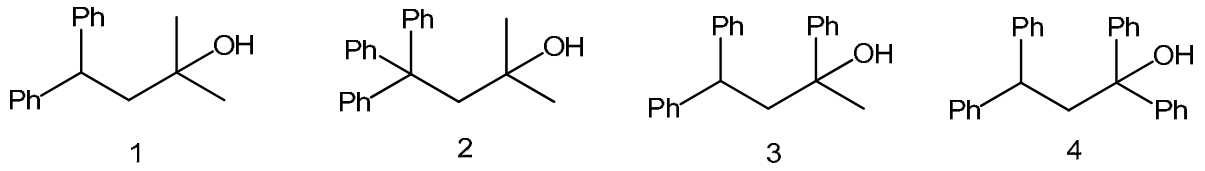
applicability and the facility of this ring closure approach, but also proved its dependence on electronic, steric and ring-strain factors.[7-11]

Herein, we describe the synthesis of mono-, di- and triphenylated indane derivatives *via* intramolecular Friedel-Crafts reactions of alkanols **1**-**4** (Scheme 1).



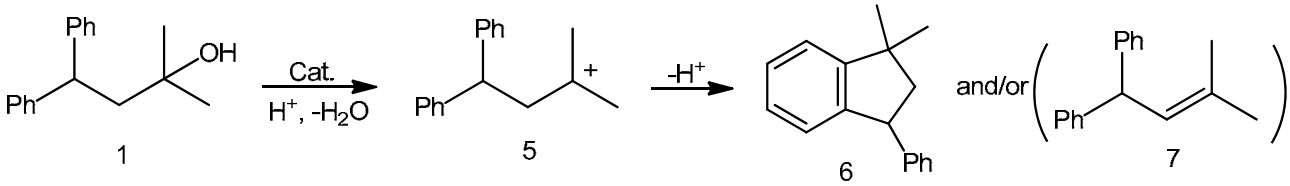
Scheme 1. Starting alkanols **1**-**4**

Results and Discussion

Production of 1,1-dimethyl-3-phenylindane 6

As shown in Table 1 and Scheme 2, reaction of 2-methyl-4,4-diphenyl-2-butanol **1** in the presence of $AlCl_3/CH_3NO_2$, H_3PO_4 or PPA gave 1,1-dimethyl-3-phenylindane **6** as a sole product. In the presence of 85% H_2SO_4 however, the reaction gave 3-methyl-1,1-diphenyl-2-butene **7** after 2 hours and a mixture of **6** and **7** after 12 hours.

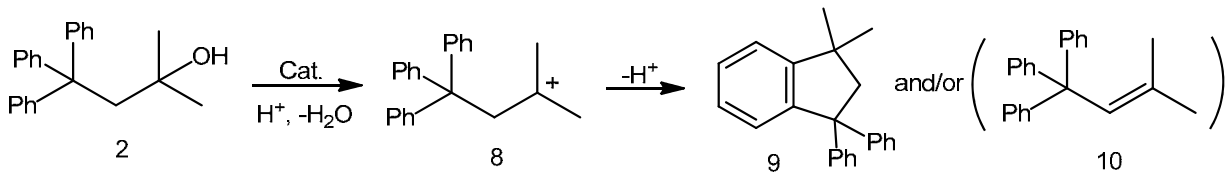
The failure of 85% H_2SO_4 to induce complete closure of **5** is probably due to the greater tendency, under these conditions, to localize the positive charge on the tertiary center, so elimination to 3-methyl-1,1-diphenyl-2-butene **7** is favored.



Scheme 2. Cyclialkylation of 2-methyl-4,4-diphenyl-2-butanol **1**.

Production of 3,3-dimethyl-1,1-diphenylindane 9

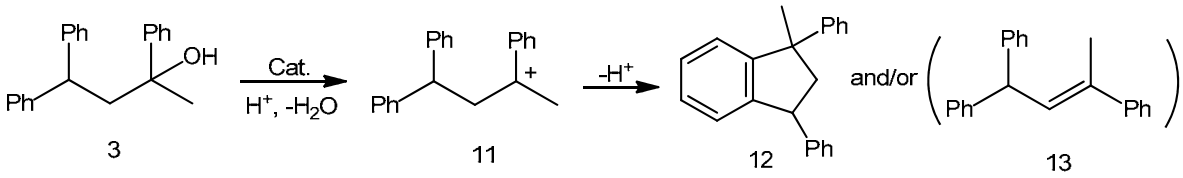
A set of six experiments carefully designed to explore the effect of reaction variables (such as catalyst type, solvent nucleophilicity, time and temperature) on the course of cyclialkylation of 2-methyl-4,4,4-triphenyl-2-butanol **2** were conducted. Indane **9** was obtained as sole product in the presence of $AlCl_3/CH_3NO_2$, H_3PO_4 or PPA catalysts. With 85% H_2SO_4 catalyst, however, varying proportions of **9** and 1,1,1-triphenyl-2-butene **10** were obtained depending on reaction time (Table 1, entries 7-12 and Scheme 3).



Scheme 3. Cyclialkylation of 2-methyl-4,4,4-triphenyl-2-butanol **2**.

Production of 1-methyl-1,3-diphenylindane 12

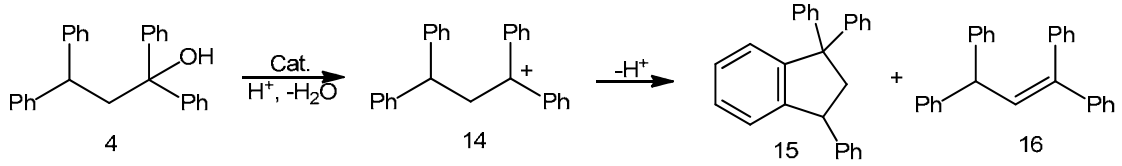
This was obtained as a sole product from 2,4,4-triphenyl-2-butanol **3** with either $AlCl_3/CH_3NO_2$ or H_3PO_4 catalyst. Using 85% H_2SO_4 as catalyst, however, resulted in pure 1,1,3-triphenyl-2-butene **13** after 2 hours of reaction and in a mixture of **12** (38%) and **13** (49%) after 15 hours (Scheme 4 and Table 1, entry 16).



Scheme 4. Cyclialkylation of 2,4,4-triphenyl-2-butanol **3**.

Production of 1,1,3-triphenylindane 4

Attempted cyclialkylation of 1,1,3,3-tetraphenyl-1-propanol **4** with 85% H_2SO_4 or $AlCl_3/CH_3NO_2$ catalyst at room temperature failed, giving similar products whose elemental, spectral and chromatographic data confirmed the formation of pure 1,1,3,3-tetraphenylpropene **16** (Scheme 1 and Table 1, entries 17 and 18). More strenuous conditions were then applied. While treatment of **4** with H_3PO_4 for 4 hours at 240-260 °C gave a mixture consisting of **15** (27%) and **16** (68%), treatment with PPA at 230-250 °C for 48 hours gave solely **15** (75% yield) (Table 1, Entry 19). These results can be attributed to two factors: (i) the steric crowding of the two phenyls encountered in closure of tertiary carbocation **14** to **15** and (ii) the doubly benzylic nature of **14** causes extensive delocalization of the positive charge over the two phenyl groups resulting in a lower energy, and hence a less reactive reaction site in the intermediate cation. The relative retardation influence of these steric and electronic factors on the ring closure step is hard to measure, but it is believed that the steric factor plays the major part.



Scheme 5. Cyclialkylation of 1,1,3,3-tetraphenyl-1-propanol **4**.

Table 1. Conditions and results of cyclialkylation reactions of alkanols **1-4**

Entry no.	Reaction conditions					Product Composition (%)
	Catalyst type	Solvent	Temp °C	Time h	Yield %	
	A. 2-Methyl-4,4-diphenyl-2-butanol **1**					
1[a]	85% H_2SO_4	PE[b]	RT	2	83	**7** (100)
2	85% H_2SO_4	PE	RT	12	86	**6** (48), **7** (45)
3[c]	$AlCl_3/CH_3NO_2$	PE	RT	2	90	**6** (100)
4	$AlCl_3/CH_3NO_2$	DCM[d]	RT	1	85	**6** (100)
5	PPA[e]	--	230-250°	5	88	**6** (100)
6[f]	H_3PO_4	--	240-260°	1	80	**6** (100)
	B. 2-Methyl-4,4,4-triphenyl-2-butanol **2**					
7	85% H_2SO_4	PE	RT	6	84	**9** (31), **10** (68)
8	85% H_2SO_4	PE	RT	20	87	**9** (85), **10** (12)
9	H_3PO_4	--	240-260°	1	83	**9** (100)
10	$AlCl_3/CH_3NO_2$	PhH	RT	2	88	**9** (100)
11	$AlCl_3/CH_3NO_2$	PE	RT	2	91	**9** (100)
12	PPA	--	230-250°	2	81	**9** (100)
	C. 2,4,4-Triphenyl-2-butanol **3**					
13	H_3PO_4	--	240-260°	1	87	**12** (100)
14	$AlCl_3/CH_3NO_2$	PE	RT	4	89	**12** (100)
15	85% H_2SO_4	PE	RT	2	85	**13** (100)
16	85% H_2SO_4	PE	RT	15	83	**12** (38), **13** (49)
	D. 1,1,3,3-Tetraphenyl-1-propanol **4**					
17	85% H_2SO_4	PE	RT	10	82	**16** (100)
18	$AlCl_3/CH_3NO_2$	PE	RT	4	86	**16** (100)
19	PPA	--	230-250°	48	75	**15** (100)
20	H_3PO_4	--	240-260°	4	86	**15** (27), **16** (68)

[a]With 85% H_2SO_4 catalyst proportions were: carbinol (0.002 mol), 85% H_2SO_4 (2 ml), solvent (10 ml). [b]Petroleum ether (PE) b.p. 60-80 °C. [c]With $AlCl_3/CH_3NO_2$ catalyst reactant proportions were: carbinol (0.002 mol), $AlCl_3$ (0.0024 mol), CH_3NO_2 (0.024 mol), solvent (10 ml). [d]Dichloromethane. [e]With PPA catalyst reactant proportions were carbinol (0.5 g) and PPA (3 g). [f]With H_3PO_4 catalyst proportions were: carbinol (0.5 g) and H_3PO_4 (4 g).

Conclusions

Commenting on the results of Table 1, it is useful to point out the following:
(i) Friedel-Crafts cyclialkylations provide a facile route for the synthesis of mono-, di- and triphenylindane derivatives.
(ii) Intramolecular reactions are much favored over intermolecular ones as indicated by the fact that compound **9** was solely obtained in spite of the presence of nucleophilic benzene as solvent.
(iii) The results of Table 1, especially those with 85% H_2SO_4, reveal that intramolecular ring closure is highly dependent on steric factors. Thus, the ease of ring closure to indanes seems to follow the following order between the employed tertiary alcohols: **1** > **2** > **3** > **4**

Experimental Section

General. Melting points were measured on a digital Gallenkamp capillary melting point apparatus and are uncorrected. Infrared spectra were determined with a Shimadzu 470 infrared spectrophotometer using KBr wafer and thin film techniques (ν cm^{-1}). ^1H NMR spectra were recorded by 90 MHz Varian NMR spectrometer using the appropriate deuterated solvent with TMS as internal standard. Chemical shifts (δ) and *J* values are reported in ppm and Hz, respectively. Elemental analyses were performed on a Perkin-Elmer 2400 Series II analyzer. The mass spectra were performed by JEOL JMS 600 spectrometer at an ionizing potential of 70 ev using the direct inlet system. Reactions were monitored by thin layer chromatography using precoated silica plates (Kieselgel 60, F 254, E. Merck), visualized with UV light. Flash column chromatography (FC) was performed on silica gel (230-400 mesh, E. Merck). All reagents were purchased from Merck, Sigma or Aldrich Chemical Co. and were used without further purification.

Synthesis of starting substrates

2-Methyl-4,4-diphenyl-2-butanol 1. This alcohol was prepared by addition of two equivalents of methylmagnesium iodide to ethyl 3,3-diphenylpropanoate.[12] The reaction mixture was left to stir for overnight then decomposed with sat. aq. NH_4Cl soln and the product was extracted with ether and dried over anhydrous magnesium sulfate. Flash chromatography (FC) of the liquid product [neutral alumina, petroleum ether (PE 60-80°) eluant] gave alcohol **1** in the form of faintly yellowish viscous oil (82%), n_D^{25} 1.5376 (Lit.[13] n_D^{25} 1.5636, b.p. 180-2°/12 mm); IR (Film) ν 3450, 3550, 3046, 2960, 1590, 1520, 1485, 1480, 1378, 1247, 1143, 1081, 760, 695 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.2 (6H, s, $2CH_3$), 2.35 (2H, d, *J* = 7.5 Hz, CH_2), 2.4 (1H, s, OH exchangeable with D_2O), 4.3 (1H, s, *J* = 7.5 Hz, CH), 7.1-7.45 (10H, m, Ar-H).

2-Methyl-4,4,4-triphenyl-2-butanol 2. Addition of two equivalents of methylmagnesium iodide to ethyl 3,3,3-triphenylpropanoate[13] followed by stirring overnight and decomposition with sat. aq. NH_4Cl soln and extraction of the product with ether gave a solid. Crystallization from

petroleum (60-80 °C) gave the product as white needles (86%), m.p. 45 °C; IR (KBr) ν 3563, 3490, 3043, 2975, 1590, 1486, 1440, 1040, 1015, 740, 700 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 0.9 (6H, s, $2CH_3$), 1.67 (1H, s, OH exchangeable with D_2O), 3.05 (2H, s, CH_2), 7.1-7.56 (15H, m, Ar-H). Anal. Calcd. for $C_{23}H_{24}O$ (316): C, 87.34; H, 7.59%; Found: C, 87.15; H, 7.72%

2,4,4-Triphenyl-2-butanol 3. This alcohol was prepared by two alternative routes: (i) By the reaction of β,β-diphenyl-2-propanone[14] with phenylmagnesium bromide and (ii) by the reaction of β,β-diphenylpropiophenone[15] with methylmagnesium iodide. In both routes, the reaction mixtures were heated at reflux for 2 h then left to stir overnight. Decomposition with sat. aq. NH_4Cl soln, extraction with ether and purification by chromatography (basic alumina, petroleum 60-80 °C as eluant) gave **3** as a pale yellow viscous oil (84% and 82% resp.), n_D^{25} 1.5527; IR (film) ν 3450, 3054, 2975, 1600, 1486, 1440, 1370, 1210, 1180, 740, 695 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.25 (3H, s, CH_3), 1.7 (1H, s, OH exchangeable with D_2O), 2.5 (2H, s, *J* = 7.5 Hz, CH_2), 3.74 (1H, t, *J* = 7.5 Hz, CH), 6.9-7.4 (15H, m, Ar-H). Anal. Calcd. for $C_{22}H_{22}O$ (302): C, 87.41; H, 7.28%; Found: C, 87.29; H, 7.06%

1,1,3,3-Tetraphenyl-1-propanol 4. This alcohol was prepared by addition of two equivalents of PhMgBr to ethyl 3,3-diphenylpropanoate.[12] The reaction mixture was left to stir overnight, decomposed with sat. aq. NH_4Cl soln and the product was extracted, dried and rotary evaporated as above. Crystallization from petroleum (60-80 °C) gave the product as white needles (79%), m.p. 92 °C (Lit.[16] m.p. 94.5 °C); IR (KBr) ν 3550, 3450, 3060, 2963, 1590, 1488, 1445, 1367, 1250, 1160, 745, 697 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.8 (1H, s, OH exchangeable with D_2O), 2.95 (2H,d, *J* = 6 Hz, CH_2), 3.9 (1H, t, *J* = 6 Hz, CH), 6.85-7.52 (20H, m, Ar-H).

Synthesis of Reference Samples

3-Methyl-1,1-diphenyl-2-butene 7. Dehydration of 2-methyl-4,4-diphenyl-2-butanol (**1**) by $AcOH/H_2SO_4$ as reported[17] and purification by chromatography (neutral alumina, *n*-hexane eluant) gave **7** (82%) as a faintly yellowish oil, R_f 0.41 (petroleum 60-80 °C/AcOEt 9:1, silica gel), n_D^{25} 1.574; IR (Film) ν 3055, 3010, 2980, 1590, 1570, 1480, 1422, 1285, 1070, 1020(s), 970, 750, 693 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.7 (6H, s, $2CH_3$), 4.75 (1H, d, *J* = 7.5 Hz, CH), 5.8 (1H, d, *J* = 7.5 Hz, CH), 7.0-7.4 (10H, m, Ar-H). Anal. Calcd. for $C_{17}H_{18}$ (222): C, 91.89; H, 8.10% Found: C, 91.46; H, 8.32%

3-Methyl-1,1,1-triphenyl-2-butene 10. Dehydration of 2-methyl-4,4,4-triphenyl-2-butanol (**2**) by $AcOH/H_2SO_4$ as directed[17] and purification by chromatography (neutral alumina, petroleum 60-80° eluant) gave **10** (85%) as yellowish oil, R_f 0..43 (petroleum 60-80 °C/AcOEt 9:1, silica gel); n_D^{25} 1.591; IR (Film) ν 3050, 3020, 2980, 1595, 1543, 1490, 1440, 1285, 1145, 1027, 940, 745, 697 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.2 (6H, s, $2CH_3$), 5.52 (1H, s, CH), 7.0-7.3 (10H, m, Ar-H). Anal. Calcd. for $C_{23}H_{22}$ (298): C, 92.61; H, 7.38%; Found: C, 92.12; H, 7.46%

1-Methyl-1,3-diphenylindane 12. This compound was obtained in a series of three consecutive steps starting with 3-phenylindanone.[18]

(i) Addition of phenylmagnesium bromide to 3-phenylindanone[18] and treatment as usual gave 1,3-diphenyl-1-indanol in the form of yellow crystals from methanol (84%) m.p. 78 °C (Lit.[19] m.p. 84 °C); IR (KBr) ν 3550, 3455, 3053, 2990, 1596, 1487, 1443, 1370, 1165, 1065, 10350, 765, 695 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 2.0 (1H, s, OH exchangeable with D_2O)), 2.6-2.83 (2H, m *J* = 7.5 Hz, CH_2), 4.23 (1H, t, *J* = 7.5 Hz, CH), 7.1-7.45 (14H, m, Ar-H).

(ii) Conversion into chloride by reaction with thionyl chloride in pyridine[20], extraction and purification by chromatography (basic alumina, petroleum 40-60 °C eluant) gave the corresponding 1-chloro-1,3-diphenylindane as a reddish viscous oil (78%), n_D^{25} 1.638: IR (Film) ν 3062, 2985, 1595, 1510, 1440, 765, 690 cm^{-1}.^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 2.7-3.05 (2H, m, *J* = 7.5 Hz, CH_2), 4.3 (1H, t, *J* = 7.5 Hz, CH), 7.1-7.32 (14H, m, Ar-H).

(iii) Coupling with dimethylcadmium[21] in benzene gave 1-methyl-1,3-diphenylindane (**12**) in the form of a thick pale yellowish oil (73%) upon purification by flash chromatography (basic alumina, *n*-hexane eluant), R_f 0.29 (silica gel, petroleum 60-80 °C/AcOEt 9:1 eluant); n_D^{25} l.6103; IR (Film) ν 3070, 2983, 1680, 1594, 1589, 1455, 1055, 1025, 760, 695 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.7 (3H, s, CH_3), 3.5-4.0 (2H, apparent m, *J* = 7.5 Hz, CH_2), 4.25 (1H, m, *J* = 7.5 Hz, CH), 7.0-7.52 (14H, m, Ar-H). Anal. Calcd. for $C_{22}H_{20}$ (284): C, 92.95; H, 7.08%; Found: C, 92.99; H, 7.26%

1,1,3-Triphenyl-2-butene 13. Dehydration of 2,4,4-triphenyl-2-butanol (**3**) by $AcOH/H_2SO_4$ following reported procedure[17] and purification by chromatography (neutral alumina, petroleum 40-60 °C eluant) gave **13** (79%) as a yellowish oil, R_f 0.4 (silica gel, petroleum 60-80 °C/AcOEt 9:1 eluant); n_D^{25} 1.638; IR (Film) ν 3050, 3014, 2985, 1638, 1595, 1487, 1446, 1370, 765 , 696 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.7 (3H, s, CH_3), 4.65 (1H, d, *J* = 7.5 Hz, CH), 5.3 (1H, d, *J* = 7.5 Hz, CH), 7.2-7.5 (15H, m, Ar-H). Anal. Calcd. for $C_{22}H_{20}$ (284): C, 92.95; H, 7.08%; Found: C, 92.87; H, 7.16%

1,1,3-Triphenylindane 15. An authentic sample of this compound was obtained in four steps starting from 3,3,3-triphenylpropanoic acid.[22]

(i) Reaction of 3,3,3-triphenylpropanoic acid with thionyl chloride in dry benzene gave 3,3,3-triphenylpropanoyl chloride[22] in the form of white crystals from petroleum (60-80 °C) (71%), m.p. 123 °C (Lit.[22] m.p.127 °C) IR (KBr); ν 3065, 3100(s), 2930(m), 1800(s), 760(s) 690(s) cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 3.76 (2H, s, CH_2), 7.1-7.5 (15H, m, Ar-H).

(ii) Cycliacylation of 3,3,3-triphenylpropanoyl chloride with $AlCl_3$ catalyst in carbon disulfide solvent gave 76% of crude product which upon crystallization from ethyl alcohol gave 69% of pure 3,3-diphenylindanone as white crystals m.p. 126 °C (Lit.[22] m.p. 130-1 °C); IR (KBr) ν 3045, 2920, 1750, 1595, 1580, 1484, 1450, 1010, 750, 690 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 3.52 (2H, s, CH_2), 7.2-7.8 (14H, m, Ar-H).

(iii) Reaction of 3,3-diphenylindanone with phenylmagnesium bromide in dry ether and decomposition with sat. aq. NH_4Cl soln gave 1,3,3-triphenyl-1-indanol in the form of yellowish viscous oil upon purification by chromatography (basic alumina, petroleum 60-80 °C/PhH eluant), n_D^{25} 1.5957; IR (Film) ν 3450, 3040, 2985, 1595, 1520, 1487, 1440, 1345, 1110, 745,

692 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.9 (1H, s, OH exchangeable with D_2O), 2.9-3.1(2H, apparent m, *J* = 7.5 Hz, CH_2), 7.1-7.5 (19H, m, Ar-H).

(iv) Reduction of 1,3,3-triphenyl-1-indanol with P/HI in glacial acetic acid as reported[23] gave 1,1,3-triphenylindane **15** as a yellowish viscous oil upon purification by chromatography (silica gel, benzene eluant) (68%), R_f 0.27 (silica gel, PE 60-80 °C/AcOEt 7.8:2.2 eluant); n_D^{25} 1.574; IR (Film) ν 3060, 2983, 1660, 1596, 1580, 1483, 1440, 1065, 1020, 760, 695 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 2.7 & 3.2 (2H, both apparent m, *J* = 7.5 Hz, CH_2), 4.3 (1H, t, CH), 7.0-7.45 (19H, m, Ar-H). Anal. Calcd. for $C_{27}H_{22}$ (346): C, 93.64; H, 6.35%; Found: C, 93.84; H, 6.11%

1,1,3,3-Tetraphenylpropene 16. Dehydration of 1,1,3,3-tetraphenyl-1-propanol **4** by $KHSO_4$ as reported[24] gave **16** as white crystals from methanol (77%), m.p. 122 °C (Lit.[25] m.p. 125-6 °C), R_f 0.29 (ptroleum 60-80 °C/AcOEt 7.8:2.2, silica gel); IR (KBr) ν 3065, 3030, 2980, 1650, 1595, 1480, 1440, 1274, 1180, 1025, 910, 750, 698 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 4.76 (1H, d, *J* = 9 Hz, CH), 6.5 (1H, d, *J* = 10.5 Hz, CH), 7.2-7.5 (20H, m, Ar-H).

Cyclialkylation procedures

The procedures described earlier[10] for cyclialkylation of arylalkanols with 85% H_2SO_4, H_3PO_4, $AlCl_3/CH_3NO_2$ and PPA were essentially followed. The conditions and results are depicted in Table 1.

Product separation and identification

3-Methyl-1,1-diphenyl-2-butene 7. This product was identical in all respects with the prepared authentic sample.

1,1-Dimethyl-3-phenylindane 6. Viscous oil, R_f 0.35 (silica gel, PE 60-80 °C/AcOEt 9:1 eluant); IR (Film) ν 3050, 2980, 1600, 1540, 1589, 1495, 1450, 1025, 743, 697cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.25 (6H, d, *J* = 15 Hz, $2CH_3$), 1.8 & 2.3 (2H, both apparent m, *J* = 9 Hz, CH_2), 4.3 (1H, t, *J* = 9 Hz, CH), 6.8-7.4 (9H, m, Ar-H). MS (EI, 70 ev) m/z (%), 222 (M^+, 49), 207 (M^+-CH_3, 100), 192 (M^+- $2CH_3$, 4.5), 178 (12.3), 167 (2.4), 166 (2.6), 91 (16.1), 77 (3.3). Anal. Calcd. For $C_{17}H_{18}$ (222): C, 91.89; H, 8.10%; Found: C, 91.46; H, 8.32%

3,3-Dimethyl-1,1-diphenylindane 9. Yellow viscous oil, R_f 0.37 (silica gel, petroleum 60-80 °C/AcOEt 8:2 eluant); IR (Film) ν 3055, 2992, 1595, 1540, 1495, 1440, 1030, 740, 695 cm^{-1}. ^1H NMR (90 MHz, $CDCl_3$, ppm), δ = 1.25 (6H, s, $2CH_3$), 2.9 (2H, s, CH_2), 7.2-7.45 (14H, m, Ar-H). MS (EI, 70 ev) m/z (%), 298 (M^+, 79.1), 283 (M^+-CH_3, 59), 268 (M^+-$2CH_3$, 3.9), 221 (M^+-Ph, 100), 206 (M^+-Ph-CH_3, 11.9), 205 (M^+-Ph-CH_3-H, 30.7), 191(18.9), 178(13), 165 (27), 154 (2.9), 142 (14.5), 124 (12.1), 95 (18.5), 77 (3.4). Anal. Calcd. For $C_{23}H_{22}$ (298): C, 92.61; H, 7.38%; Found: C, 92.12; H, 7.46%

3-Methyl-1,1,1-triphenyl-2-butene 10. This product was identical in all respects with the prepared authentic sample.

1-Methyl-1,3-diphenylindane 12. This product was identical in all respects with the prepared authentic sample.

1,1,3-Triphenyl-2-butene 13. This product was identical in all respects with the prepared authentic sample.
1,1,3-Triphenylindane 15. This product was identical in all respects with the prepared authentic sample.
1,1,3,3-Tetraphenylpropene 16. This product was identical in all respects with the prepared authentic sample.

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