

Friedel-Crafts alkylation of benzene with 1,2-diphenyl-2-propanol, 1-chloro-2,3-diphenylpropane and 2-methyl-1-phenyl-2-butanol[†]

Ali A. Khalaf*, Ibrahim M. Awad, T. I. El-Emary and H. A. K. Abd El-Aal

Chemistry Department, Faculty of Science, Assiut University, Assiut, Egypt

E-mail : khalafal@aun.edu.eg

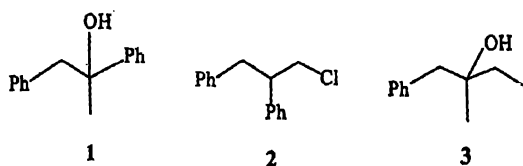
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Abstract : The alkylation of benzene with 1,2-diphenyl-2-propanol (1) using $\text{AlCl}_3/\text{CH}_3\text{NO}_2$ catalyst gave a mixture of 1,2,2- (4) and 1,1,2-triphenylpropane (5) as product alkylates. With 85% H_2SO_4 catalyst, the product consisted of *E*-1,2-diphenylpropene (6) after 2 h of a mixture of 5 and 6 after 18 h. Similar alkylation of benzene with 1-chloro-2,3-diphenylpropane (2) using AlCl_3 catalyst gave a mixture consisting of 4, 5 and 6. Finally, alkylation of benzene with 2-methyl-1-phenyl-2-butanol (3) using $\text{AlCl}_3/\text{CH}_3\text{NO}_2$ gave 2-methyl-1,1-diphenylbutane (10) as sole product alkylate. The identities of the products were confirmed spectroscopically and by comparison with unequivocally prepared samples. Mechanisms are proposed to rationalise the observed results.

Keywords : Friedel-Crafts alkylation, triphenylpropanes, carbocation rearrangement, dimerisation.

Introduction

Friedel-Crafts alkylation chemistry is a lively subject of both theoretical and industrial value¹. For that matter, we present here our findings on the reaction of benzene with three unprecedented mono- and diphenylated alkylating agents, namely, 1,2-diphenyl-2-propanol (1), 1-chloro-2,3-diphenylpropane (2) and 2-methyl-1-phenyl-2-butanol (3).



Results and discussion

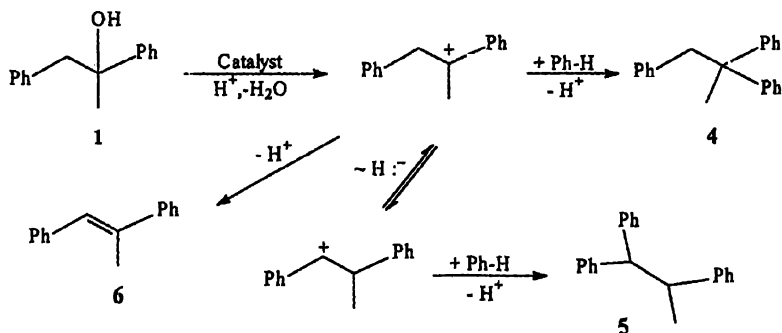
Alkylation of benzene with 1,2-diphenyl-2-propanol (1) : As pointed out in the experimental section, carbinol 1 was obtained in excellent yields via three alternative Grignard routes. With 85% H_2SO_4 catalyst at room temperature, 1 and benzene gave a product consisting of *E*-1,2-diphenylpropene (6) (88% yield) after a reaction time of 2 h and a mixture of 5 (32% yield) and (6) (60% yield)

after a reaction time of 18 h. With $\text{AlCl}_3/\text{CH}_3\text{NO}_2$ catalyst at room temperature for 20 h, the reaction of 1 with benzene gave a complex product mixture (GC/MS) of which two alkylates, namely, 1,2,2-triphenylpropane (4, 4%) and 1,1,2-triphenylpropane (5, 15%) could be identified and the remaining (*ca.* 81%) was composed of high molecular weight unidentified products. The products and their mechanistic modes of formation are formulated in Scheme 1.

The structures of compounds 4, 5 and 6 were confirmed by unequivocal synthesis : compound 4 by the coupling of $\text{CH}_3\text{CPh}_2\text{Cl}$ with benzylmagnesium chloride², compound 5 by the reduction of 1,1,2-triphenyl-2-propanol with HI/P in acetic acid³ and compound 6 by the dehydration of 1 by $\text{AcOH}/\text{H}_2\text{SO}_4$ ⁴.

Alkylation of benzene with 1-chloro-2,3-diphenylpropane (2) : 2,3-Diphenyl-1-propanol obtained through a series of standard reactions starting from phenylacetic acid (see Experimental) was converted to chloride 2 by reaction with thionyl chloride in pyridine. The alkylation of benzene with 2 was catalysed by anhydrous AlCl_3 . The product, which was shown to be complex by

[†]Part 28 of the series 'Modern Friedel-Crafts Chemistry'. For preceding paper of the series see Ref. 6.

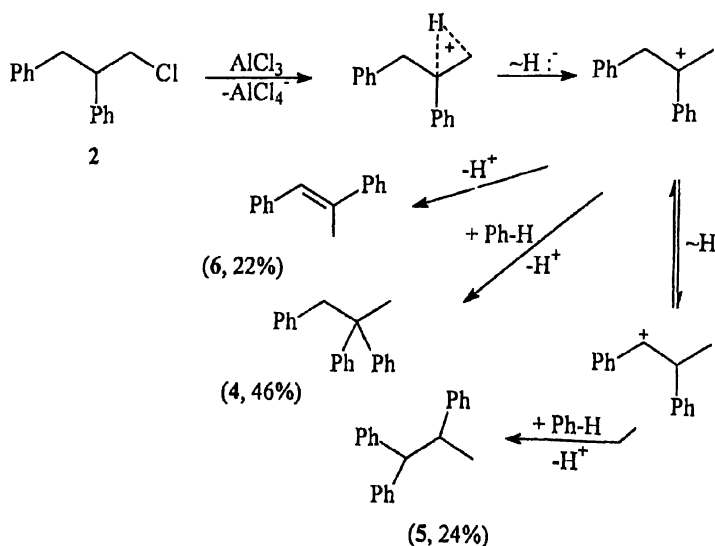


Scheme 1

TLC (thin layer chromatography), was column chromatographed leading to the separation of three components. These by comparison with authentic samples were shown to be identical with compounds 4, 5 and 6 obtained from carbinol 1. The mechanism of formation of these compounds and their percentage yields are depicted in Scheme 2.

Alkylation of benzene with 2-methyl-1-phenyl-2-butanol (3) : Compound 3, obtained by two alternative Grignard routes, was allowed to react with benzene in the presence of $\text{AlCl}_3/\text{CH}_3\text{NO}_2$ catalyst. Analysis of the flash

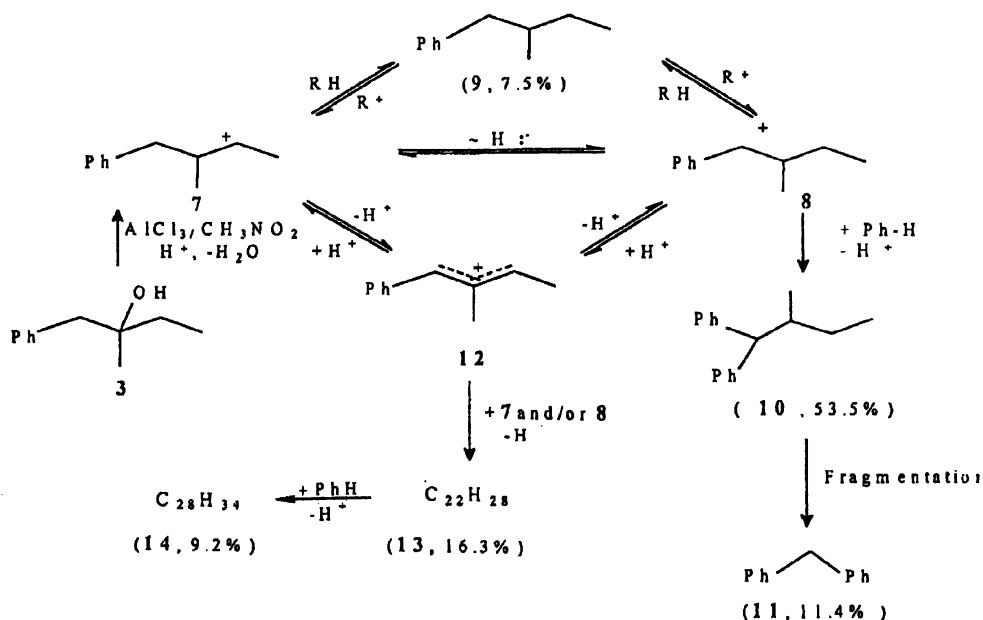
chromatographed crude by GC/MS showed the presence of five components with parent M^+ peaks at m/z 148, 168, 224, 292 and 370 corresponding to molecular formulas $\text{C}_{11}\text{H}_{16}$, $\text{C}_{13}\text{H}_{12}$, $\text{C}_{17}\text{H}_{20}$, $\text{C}_{22}\text{H}_{28}$ and $\text{C}_{28}\text{H}_{34}$, respectively. Based on theoretical considerations and comparison with available experimental data, the first two components were shown to be 2-methyl-1-phenylbutane (9) and diphenylmethane (11) (Scheme 3). The third and major component was shown to be 2-methyl-1,1-diphenylbutane (10) identical in all respects with a sample prepared unequivocally as reported⁵. The last two com-



Scheme 2

ponents are believed on theoretical grounds to be an alkene dimer 13 ($C_{22}H_{28}$) stemming from the reaction of intermediate carbocations 7 and/or 8 with their equilibrating alkene(s) (12). Further reaction of 13 with benzene leads to hydrocarbon 14 ($C_{28}H_{34}$). The exact structures of both 13 and 14 have not yet been disclosed. Notably, such dimerisation and alkylation reactions are very well known¹. The products, their composition percentages and their mechanistic interpretations are depicted in Scheme 3.

chloride (87% yield⁷). (ii) By the reaction of desoxybenzoin with methylmagnesium iodide (90% yield⁸). (iii) By the reaction of phenylacetone with phenylmagnesium bromide (89% yield). Recrystallisation from pet. ether (b.p. 40–60°) gave carbinol 1 in the form of white crystals, m.p. 51–52° (lit.^{7,8} m.p. 49–51°); IR (KBr) ν_{max} : 3420, 3050, 2940, 1590, 1480, 1440, 1370, 1215, 1020, 760, 695 cm^{-1} ; ¹H NMR ($CDCl_3$) δ : 1.6 (3H, s, CH_3), 2.0 (1H, s, OH exchangeable with D_2O), 3.05 (2H, s, CH_2), 7.0–7.6 (10H, q, Ar).



Scheme 3

Experimental

Materials and instruments :

All chemicals are reagent grades purchased from known suppliers and were used as furnished. The purities and identities of the starting materials and final products were determined by IR, ¹H NMR, GC/MS and/or elemental analysis using the instruments previously described⁶.

Synthesis of starting alkylating agents :

1,2-Diphenyl-2-propanol (1) : This was prepared by three alternative Grignard routes following standard procedures except that the complex was decomposed by saturated NH_4Cl solution to avoid possible elimination : (i) By the reaction of acetophenone with benzylmagnesium

1-Chloro-2,3-diphenylpropane (2) : This compound was obtained in a series of five consecutive steps starting with phenylacetic acid following standard procedure⁵. A summary of the steps and of the involved product intermediates is as follows : (i) Perkin-type condensation of phenyl-acetic acid with benzaldehyde in the presence of Ac_2O and Et_3N gave 2,3-diphenylpropenoic acid in the form of white needles from aqueous ethanol (60% yield), m.p. 172° (lit.⁹ m.p. 172–173°); IR (KBr) ν_{max} : 3200, 2500, 1665, 1570, 1485, 1440, 1423, 1075, 920, 740, 690 cm^{-1} ; ¹H NMR ($CDCl_3$) δ : 6.8–7.3 (10H, m, Ar), 7.9 (1H, s, CH), 11.0 (1H, s, COOH). (ii) Reduction of

2,3-diphenylpropenoic acid with Na(Hg)/NaOH gave 2,3-diphenylpropenoic acid in the form of white crystals from ethanol (80% yield), m.p. 89° (lit.¹⁰ m.p. 86, 88–89°); IR (KBr) ν_{\max} : 3300–2500, 1690, 1595, 1485, 1445, 1435, 1410, 1180, 1065, 1020, 738, 790 cm^{-1} ; ¹H NMR (CDCl₃) δ : 2.7–3.4 (2H, m, *J* 9 Hz, CH₂), 3.6–3.8 (1H, t, *J* 9 Hz, CH), 6.9–7.2 (10H, d, Ar), 10.3 (1H, s, COOH). (iii) Esterification of 2,3-diphenylpropanoic acid with methanol gave methyl 2,3-diphenylpropanoate¹¹ as thick pale yellowish oil upon purification by flash chromatography (FC, basic alumina, 8/2 *n*-hexane/AcOEt) (86% yield), n_D^{25} 1.6103; IR (film) ν_{\max} : 3020, 2950, 1725, 1590, 1485, 1430, 1155, 1063, 1020, 740, 690 cm^{-1} ; ¹H NMR (CDCl₃) δ : 2.8–3.5 (2H, m, *J* 9 Hz, CH₂), 3.25 (3H, s, CH₃), 6.95–7.3 (10H, m, Ar). (iv) Reduction of methyl 2,3-diphenylpropanoate with LiAlH₄ gave 2,3-diphenyl-1-propanol¹² as yellowish viscous oil upon purification by FC (basic alumina, *n*-hexane), n_D^{25} 1.5298; IR (film) ν_{\max} : 3550, 3366, 3020, 2910, 1589, 1480, 1440, 1380, 1150, 1050, 1020, 750, 690 cm^{-1} ; ¹H NMR (CDCl₃) δ : 2.4 (1H, s, OH exchangeable with D₂O), 2.8–3.2 (1H, m, *J* 6 Hz, CH), 2.95 (2H, d, *J* 6 Hz, PhCH₂), 3.6 (2H, d, *J* 6 Hz, CH₂OH), 6.9–7.4 (10H, m, Ar). (v) Conversion of 2,3-diphenyl-1-propanol to 1-chloro-2,3-diphenylpropane¹³ with thionyl chloride in pyridine gave the targeted chloride as yellowish liquid (73% yield) upon purification by FC (basic alumina, petroleum ether b.p. 60–80°), n_D^{25} 1.5961; IR (film) ν_{\max} : 3100, 2930, 1580, 1490, 740, 700 cm^{-1} ; ¹H NMR (CDCl₃) δ : 2.7–3.2 (3H, unresolved m, PhCH₂CHPh), 3.6 (2H, d, *J* 6 Hz, CH₂Cl), 6.7–7.3 (10H, s, Ar).

2-Methyl-1-phenyl-2-butanol (3) : This was obtained by two reported alternative Grignard routes following standard procedures with the exception that the complex was hydrolysed with saturated NH₄Cl solution : (i) By the reaction of phenylacetone with ethylmagnesium bromide in 80% yield¹⁴. (ii) By the reaction of 2-butanone with benzylmagnesium chloride in 81% yield¹⁵.

The products from both routes were identical and were purified by FC (neutral alumina, benzene) to give pure 3 in the form of viscous liquid, n_D^{25} 1.5175 (lit.¹⁴ n_D^{25} 1.5097, b.p. 110–112° at 14 mm); IR (film) ν_{\max} : 3560, 3410, 3030, 2990, 1590, 1487, 1445, 1375, 1100, 1145, 740, 695 cm^{-1} ; ¹H NMR (CDCl₃) δ : 0.9 (3H, t, *J* 3 Hz, CH₃), 1.0 (3H, s, CH₃), 1.2–1.5 (2H, q, *J* 4.5 Hz, CH₂),

2.5 (1H, s, OH exchangeable with D₂O), 2.65 (2H, s, CH₂), 7.3 (5H, s, Ar).

Synthesis of authentic samples :

1,2,2-Triphenylpropane (4) : This compound was obtained by the coupling of 1-chloro-1,1-diphenylethane¹⁶ (obtained from 1,1-diphenylethanol¹⁷) with benzylmagnesium chloride in benzene following the procedure of Gomberg and Cone¹⁸. The title compound was obtained in the form of white powder from methanol (68% yield), m.p. 116° (lit.² m.p. 116–117°) (Found : C, 92.48; H, 7.25. C₂₁H₂₀ requires : C, 92.64; H, 7.35%); IR (KBr) ν_{\max} : 3040, 2950, 1585, 1500, 1445, 1400, 1360, 1095, 1060, 760, 720 cm^{-1} ; ¹H NMR (CDCl₃) δ : 2.25 (3H, s, CH₃), 3.5 (2H, s, CH₂), 7.10–7.45 (15H, m, Ar); MS *m/z* (%), 271 (M⁺-H, 25.4), 180 (M⁺-PhCH₂,-H, 94.7), 166 (M⁺-PhCH₂,-CH₃, 54), 151 (100), 104 (M⁺-PhCH₂,-Ph, 98), 91 (10.8), 90 (6.4), 77 (24.3).

1,1,2-Triphenylpropane (5) : This compound was prepared in three steps starting with diphenylacetyl chloride¹⁹. (i) Coupling of diphenylacetyl chloride with diphenylcadmium reagent²⁰ in benzene solvent gave 1,2,2-triphenylethanone in the form of white crystals from methanol (62% yield), m.p. 136° (lit.²¹ m.p. 136°); IR (KBr) ν_{\max} : 3080, 2900, 1685, 1580, 1490, 1440, 740, 690 cm^{-1} ; ¹H NMR (CDCl₃) δ : 4.6 (1H, s, Ph₂CH), 7.2–7.9 (15H, m, Ar). (ii) Reaction of 1,2,2-triphenylethanone with methylmagnesium iodide in dry ether and decomposition with saturated NH₄Cl solution gave 1,1,2-triphenyl-2-propanol in the form of white crystals from methanol (63% yield), m.p. 76° (lit.^{8,22} m.p. 76°); IR (KBr) ν_{\max} : 3560, 3050, 2950, 1595, 1480, 1430, 1060, 740, 695 cm^{-1} ; ¹H NMR (CDCl₃) δ : 1.1 (3H, s, CH₃), 2.3 (1H, s, OH exchangeable with D₂O), 4.2 (1H, s, Ph₂CH), 7.1–7.6 (15H, m, Ar). (iii) Reduction of 1,1,2-triphenyl-2-propanol with red phosphorus and HI in glacial acetic acid⁵ gave 1,1,2-triphenylpropane (5) in the form of white crystals from methanol (65% yield), m.p. 74° (lit. m.p. 72–74°³, 76°²³); IR (KBr) ν_{\max} : 3015, 2980, 1587, 1480, 1440, 1065, 1010, 735, 690 cm^{-1} ; ¹H NMR (CDCl₃) δ : 1.3 (3H, d, *J* 12 Hz, CH₃), 3.55–4.05 (1H, m, *J* 9 Hz, CH), 4.3 (1H, d, *J* 15 Hz, Ph₂CH), 7.15–7.8 (15H, m, Ar); MS *m/z* (%) 271 (M⁺-H, 15.9), 194 (M⁺-Ph, 14.6), 166 (Ph₂CH, 86), 165 (M⁺-Ph₂CH,-H, 99.1), 151 (100), 104 (M⁺-C₈H₈, 88.1), 90 (PhCH, 20), 77 (8.5).

E-1,2-Diphenylpropene (**6**) : Dehydration of 1,2-diphenyl-2-propanol (**1**) by heating with glacial AcOH/H₂SO₄ gave **6** in the form of white crystals from acetic acid (89%), m.p. 83° (lit.⁴ m.p. 83–83.5°); IR (KBr) ν_{\max} : 3060, 2900, 970, 750, 695 cm⁻¹; ¹H NMR (CDCl₃) δ : 2.35 (3H, s, CH₃), 7.02 (1H, s, CH), 7.05–7.65 (10H, t, Ar); MS *m/z* (%) 193 (M⁺-H, 99.8), 178 (M⁺-H, -CH₃, 100), 117 (M⁺-Ph, 2.1), 102 (M⁺-CH₃, -Ph, 55.4), 90 (52), 77 (12.2).

2-Methyl-1,1-diphenylbutane (**10**) : This was prepared in two steps following standard procedures⁵ : (i) Reaction of *sec*-butylmagnesium bromide (10% excess) with benzophenone in dry ether gave 2-methyl-1,1-diphenyl-1-butanol as pale yellowish oil after purification by FC (neutral alumina, *n*-hexane) (68% yield), n_D^{25} 1.5483; IR (film) ν_{\max} : 3450, 3030, 2960, 1595, 1480, 1440, 760, 695 cm⁻¹; ¹H NMR (CDCl₃) δ : 0.6–1 (3H, m, CH₃), 1.2 (3H, d, *J* 6 Hz, CH₃), 1.3–1.7 (2H, m, *J* 6 Hz, CH₂); 2 (1H, m, OH exchangeable with D₂O), 6.9–7.5 (10H, m, Ar). (ii) Reduction of 2-methyl-1,1-diphenyl-1-butanol with P/HI in glacial acetic acid gave 2-methyl-1,1-diphenylbutane (**10**) as pale yellowish oil after purification by FC (silica gel, 8/2 *n*-hexane/AcOEt) (76% yield), n_D^{25} 1.5603 (lit.^{5a} n_D^{25} 1.5533); IR (film) ν_{\max} : 3060, 2910, 1590, 1480, 1445, 1375, 1070, 1020, 740, 693 cm⁻¹; ¹H NMR (CDCl₃) δ : 0.8 (3H, t, *J* 3 Hz, CH₃), 1.35 (3H, d, *J* 3 Hz, CH₃), 1.4 (2H, m, CH₂), 2.2 (1H, m, CH), 3.4 (1H, d, *J* 10.5 Hz, Ph₂CH), 6.7–7.1 (10H, d, Ar); MS *m/z* (%) 224 (M⁺, 78.7), 208 (M⁺-H, -CH₃, 4.9), 181 (M⁺-C₃H₇, 13.6), 167 (Ph₂CH, 100), 147 (M⁺-Ph, 6.5), 91 (PhCH₂, 89.6), 77 (Ph, 18.7) (Found : C, 90.96; H, 9.02. C₁₇H₂₀ requires : C, 91.07; H, 8.92%).

Alkylation procedures and results :

The alkylation procedures published in earlier papers for Friedel-Crafts alkylations using alcohols and chlorides in the presence of AlCl₃, AlCl₃/CH₃NO₂ and H₂SO₄ catalysts were essentially followed²⁴.

Alkylation of benzene with 1,2-diphenyl-2-propanol (**1**) :

(i) Using AlCl₃/CH₃NO₂ catalyst : Reaction of **1** (0.636 g, 0.003 mol) with benzene (2.34 g, 0.03 mol) in the presence of AlCl₃/CH₃NO₂ catalyst [formed from AlCl₃ (0.48 g, 0.0036 mol) and CH₃NO₂ (1.31 g, 0.021 mol)] for 20 h at room temperature gave 0.54 g (84.9 wt%) of crude reddish viscous oil. Purification by FC (neutral

alumina, *n*-hexane) gave 0.48 g (75.46 wt%) of clean yellowish viscous oil, n_D^{25} 1.6340. This was shown by GC/MS to contain 1,2,2-triphenylpropane (**4**, 4.1%) and 1,1,2-triphenylpropane (**5**, 14.83%) which were identical in all respects with authentic samples. The rest (81.1%) consisted of high molecular weight unidentified compounds.

(ii) Using 85% H₂SO₄ catalyst : Reaction of **1** (0.848 g, 0.004 mol) with benzene (3.12 g, 0.04 mol) and 85% H₂SO₄ (1 ml) for 2 h at room temperature gave 0.68 g (87.62 wt%) of crude white solid. Crystallization from methanol gave pure *E*-1,2-diphenylpropene (**6**, 72% yield) in the form of white crystals, m.p. 82° (lit.⁴ m.p. 83–83.5°). This compound was identical in all respects with the prepared authentic sample.

Repetition of the above reaction and extending the time for 18 h gave 0.76 g (89.62 wt% yield) of crude white semi-solid which upon separation by preparative TLC (silica gel, 9.5/0.5 *n*-hexane/AcOEt), gave *E*-1,2-diphenylpropene (**6**, 60% yield) and 1,1,2-triphenylpropane (**5**, 32% yield). Both products were identical in all respects with the prepared authentic samples.

Alkylation of benzene with 1-chloro-2,3-diphenylpropane (2) in the presence of AlCl₃ catalyst : Reaction of a mixture of **2** (0.92 g, 0.004 mol), benzene (3.1 g, 0.04 mol) and AlCl₃ (0.053 g, 0.0004 mol) for 3 h at room temperature followed by decomposition, extraction, drying, and evaporation of solvents gave 0.8 g (86.76 wt% yield) of crude solid product. This crude was column chromatographed on neutral alumina giving three fractions : Fraction (a) with pet. ether (b.p. 60–80°) as eluant consisting of *E*-1,2-diphenylpropene (**6**, 22%), (b) with pet. ether (b.p. 60–80°)/AcOEt as mixed eluant consisting of 1,1,2-triphenylpropane (**4**, 46%) and (c) with the same eluant consisting of 1,1,2-triphenylpropane (**5**, 24%). All three products were identical in all respects with the prepared authentic samples.

Alkylation of benzene with 2-methyl-1-phenyl-2-butanol (3) in the presence of AlCl₃/CH₃NO₂ catalyst : Reaction of **3** (0.656 g, 0.004 mol) with benzene (3.12 g, 0.04 mol) and AlCl₃/CH₃NO₂ formed from AlCl₃ (0.64 g, 0.0048 mol) and CH₃NO₂ (2.92 g, 0.048 mol) for 2 h at room temperature gave 0.594 g (90.54 wt.% yield) of crude reddish viscous oil. Purification by FC (neutral

alumina, *n*-hexane) gave a clear yellow viscous oil which on GC/MS analysis showed five major components with M^+ peaks at *m/z* 148, 168, 224, 292 and 370 corresponding to molecular formulas $C_{11}H_{16}$, $C_{13}H_{12}$, $C_{17}H_{20}$, $C_{22}H_{28}$ and $C_{28}H_{34}$, respectively. Comparison with available and prepared authentic samples showed the first three products to be 2-methyl-1-phenylbutane (**9**, 7.53%), diphenylmethane (**11**, 11.4%) and 2-methyl-1,1-diphenylbutane (**10**, 53.5%). The last two products are believed to be a dimer of **12** numbered **13** and its phenylated product, numbered **14** (Scheme 3). A pure identical sample of **10** (0.21 g) was obtained when the above product mixture was subjected to further separation by preparative TLC.

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