

hexane-THF, 11/5/1): bp 92–96 °C (2.7 mm) [lit.^{2d} bp 110–112 °C (5 mm)].

2-tert-Butoxycarbonyl-3-methoxycarbonylmethyl-2-(cis-2-pentenyl)cyclopentanol (13b, R' = t-Bu). A solution of **10b** (R' = t-Bu, 37 mg, 0.11 mmol) and NaBH₄ (6 mg, 0.16 mmol) in MeOH (1.5 mL) was refluxed for 1 h. The mixture was quenched with AcOH (0.1 mL) and concentrated in vacuo. Column chromatography (SiO₂, benzene-AcOEt, 5/1) of the residue gave **13b** (R' = t-Bu, 36 mg, 96%): bp 75–79 °C (0.005 mm); ¹H NMR (CCl₄) δ 0.98 (t, 3, CH₃), 1.20–2.69 (m, 21), 3.60 (s, 3, CH₃O), 3.96 (m, 1, CHO), 5.21–5.54 (m, 2, HC=CH); IR (neat) 3509 (OH), 1721 cm⁻¹ (C=O).

Anal. Calcd for C₁₈H₃₀O₅: C, 66.23; H, 9.26. Found: C, 66.28; H, 9.50.

2-tert-Butoxycarbonyl-3-methoxycarbonylmethyl-2-(cis-2-pentenyl)cyclopentanone (14b, R' = t-Bu). To a solution of **13b** (R' = t-Bu, 15 mg, 0.046 mmol) in CH₂Cl₂ (1 mL) was added dropwise 2 M chromic acid (0.1 mL). The mixture was stirred at 16–20 °C for 12 h and then diluted with AcOEt. Upon the usual workup as described for the oxidation of **15**, there was obtained **14b** (R' = t-Bu, 10 mg, 67%) after column chromatography (SiO₂, benzene-hexane-AcOEt, 10/5/1): bp 79–83 °C (0.01 mm); ¹H NMR (CCl₄) δ 0.97 (t, 3, CH₃), 1.29–2.79 (m, 11), 1.45 (s, 9, CH₃), 3.64 (s, 3, CH₃O), 4.94–5.59 (m, 2, HC=CH); IR (neat) 1738 cm⁻¹ (C=O).

Anal. Calcd for C₁₈H₂₈O₅: C, 66.64; H, 8.70. Found: C, 66.87; H, 8.94.

Methyl dl-Jasmonate (1b) from 14b (R' = t-Bu). A solution of **14b** (R' = t-Bu, 7.2 mg, 0.022 mmol) in benzene (1 mL) containing a catalytic amount of *p*-toluenesulfonic acid was refluxed for 20 min. After the usual workup, the residue was chromatographed (SiO₂, benzene-AcOEt, 10/1) to give **1b** (4.5 mg, 90%); ¹³C NMR (multiplicity, carbon no.) δ 14.1 (q, 12), 20.6 (t, 11), 25.5 (t, 4), 27.2 (t, 8), 37.8 (t, 2 or 5), 38.0 (d, 3), 38.8 (t, 5 or 2), 51.6 (q, 13), 54.0 (d, 7), 124.9 (d, 9), 134.0 (d, 10), 172.5 (s, 1), 218.8 (s, 6); IR and ¹H NMR data were identical with those of an authentic sample.

Registry No.—**1b**, 20073-13-6; **2b**, 63569-04-0; (*E*)-**4**, 32815-00-2; (*Z*)-**4**, 75314-31-5; **5** (R' = Me), 63528-42-7; **5** (R' = t-Bu), 63528-43-8; **7a** (R' = Me), 63528-44-9; **7a** (R' = t-Bu), 63528-45-0; **7b** (R' = Me), 63528-46-1; **8a** (R' = Me), 63528-47-2; **8a** (R' = t-Bu), 63528-48-3; **9a** (R' = Me), 63528-49-4; **9b** (CR' = Me), 63528-50-7; **10a** (R' = Me),

63528-51-8; **10a** (CR' = t-Bu), 63528-52-9; **10b** (R' = Me), 63528-53-0; **10b** (R' = t-Bu), 63528-54-1; **13a** (R' = Me), 63528-55-2; **13b** (R' = Me), 63534-37-2; **13b** (R' = t-Bu), 63528-56-3; **14a** (R' = Me), 55254-74-5; **14b** (R' = Me), 55254-73-4; **14b** (R' = t-Bu), 63528-57-4; **15**, 51388-61-5; AcCH₂CO₂Me, 105-45-3; pentynyl bromide, 16400-32-1; AcCH₂CO₂-*t*-Bu, 1694-31-1.

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Cyclodimerization of Styrene

Arnold R. Taylor,^{1a,b,c} Gary W. Keen,^{1c} and Edmund J. Eisenbraun^{*1b,d}

Department of Chemistry, Oklahoma State University, Stillwater, Oklahoma 74074 and
Research and Development Department, Continental Oil Company, Ponca City, Oklahoma 74601

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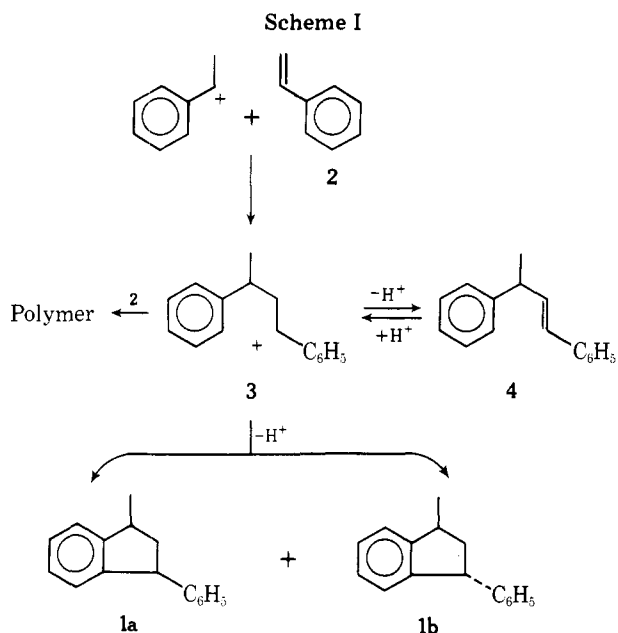
The cyclodimerization of styrene in the presence of sulfuric acid or Amberlyst-15 resin yields a 1:1 mixture of *cis*- and *trans*-1-methyl-3-phenylindan (**1a** and **1b**) via (*E*)-1,3-diphenyl-1-butene (**4**). 1-Methyl-3-phenylindene (**8**) and 3-methyl-1-phenylindene (**9**) were synthesized and converted to **1a** and **1b**. Base-catalyzed equilibration of **1a** and **1b** as well as **8** and **9** gave **1a:1b** (80:20) and **8:9** (30:70), respectively.

cis- and *trans*-1-methyl-3-phenylindan (**1a** and **1b**) can be obtained by cyclodimerization of styrene (**2**) with sulfuric acid,^{2a-g} phosphoric acid,^{3a-d} polyphosphoric acid (PPA),^{3b} alumina-silica,^{3a} perchloric acid,^{3d} chlorosulfonic acid,^{3d} or by passing styrene over hot promoted B₂O₃.⁴ This reaction may proceed through the cation **3**, which can eliminate a proton to form the alkene **4**, cyclize to **1a** and **1b**, or yield polymer, as shown in Scheme I.

The low-temperature dimerization kinetics of **2** to **1a** and **1b** have been reported to be second order, whereas high-temperature kinetics are complex.^{3d} Two isomeric forms of **1** have been reported⁵ and identified⁶ as **1a**, mp 9.5 °C, and **1b**, mp 25.5 °C. It has been reported that **1a:1b** as a 50:45 mixture was converted to a 62:38 ratio by stirring with 10%

AlBr₃⁷ and that **1a** is isomerized to an 82:18 ratio of **1a:1b** with AlCl₃.⁵ The tertiary, twice-benzylic hydrogen of **1** is reported to be more reactive in forming a radical intermediate than the tertiary benzylic hydrogen.⁸

We sought **1a** and **1b** in order to study their stereochemistry and clarify their relative thermodynamic stability. The structure and stability of **1a** and **1b** were studied through equilibration experiments and by preparations from indenenes. Sulfuric acid, ethylaluminum dichloride (EtAlCl₂),⁹ and Amberlyst-15 (A-15),¹⁰ an insoluble sulfonic acid resin, were tested as catalysts for the cyclodimerization reaction. Using A-15 allowed convenient monitoring of this reaction. Samples were periodically withdrawn from the A-15-catalyzed reactions and analyzed by GC.¹¹ The linear dimer **4** appears to be



an initial product and it slowly disappears as **1a:1b** (1:1) form. The reaction conditions were varied, but the combined yield of **1a** and **1b** from the A-15-catalyzed cyclodimerization remained at about 20%.

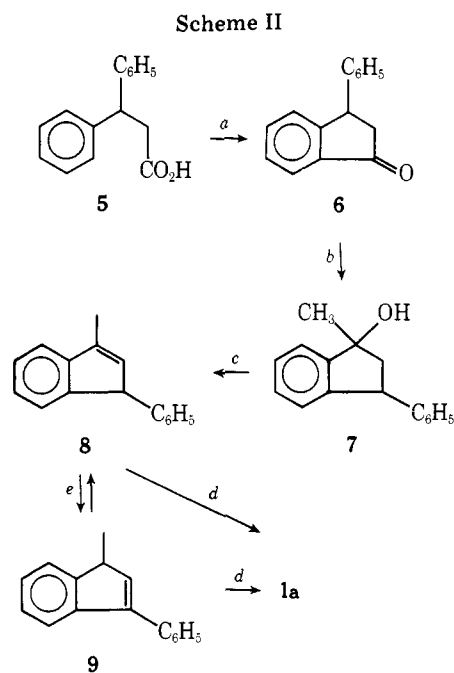
A sulfuric acid catalyzed reaction^{2f} was used for production of the **1a:1b** mixture required for the equilibration studies. The yield was 69–80%; some **4** always remained. A diluted sulfuric acid solution and a lower temperature allowed isolation of **4** in good yields.^{2g} Amberlyst-15 and PPA were used to show that **4** is converted to **1a** and **1b**. A similar conversion of **4** to **1a** and **1b** has been accomplished via a supported H₃PO₄ catalyst at 200 °C.¹² The cyclization of **4** to **1** with A-15 in 80% yield is a more facile reaction than direct conversion of **2** to **1** with A-15 (20% yield). This suggests that the A-15 catalyst becomes coated with polystyrene when **2** is present.

A series of reactions was run to determine whether EtAlCl₂¹³ would be useful in the dimerization of styrene, since the dimerization of α -methylstyrene had been successfully carried out with this catalyst,¹⁴ but EtAlCl₂ was less convenient to handle and gave a product of lower purity. Its use was not studied further.

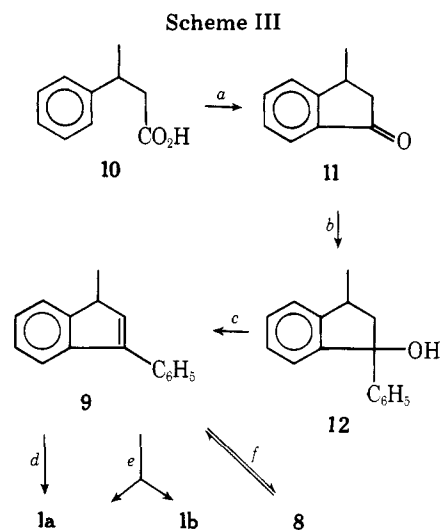
The ¹H NMR signals of **1a** and **1b** could not be confidently assigned from mixtures, so individual samples of **1a** and **1b** were prepared as shown in Scheme II. Isomer **1b** was obtained in approximately 95% purity by recrystallizing a **1a:1b** mixture from petroleum ether, bp 60–68 °C, slowly cooled in dry ice. Dehydration and distillation of **7** yielded 1-methyl-3-phenylindene (**8**) and 3-methyl-1-phenylindene (**9**) in a 30:70 ratio, respectively. Hydrogenation of **8** or a mixture of **8** and **9** over Pd/C catalyst produced only **1a**.^{5,6} When **8** was treated with a 5% KOH solution in methanol, an equilibrated mixture of **8:9** (30:70) was obtained.

3-Methyl-1-phenylindene (**9**) was synthesized as shown in Scheme III to further the equilibration studies and ¹H NMR assignments of **1a**, **1b**, **8**, and **9**. None of **8** was observed in the preparation of **9**. Isomerization of **9** by a 5% solution of KOH in methanol gave the same equilibrated mixture of **8:9** (30:70) as obtained from **8**. The reduction of **8** and **9** by sodium in liquid ammonia gives mixtures of **1a** and **1b**, as shown in Table I.

No isomerization was observed on treatment of **1a** with methanolic hydrochloric acid or sodium ethoxide at room temperature, but treatment with sodium amide produced **1a:1b** (80:20). Isomer **1b** showed similar behavior. Models of **1a** and **1b** show that the methyl and phenyl groups of **1a** may



a PPA, 90 °C. *b* CH₃MgBr, ether. *c* Toluene, Δ . *d* Pd/C, H₂, *e* 5% KOH in CH₃OH.



a PPA, Δ . *b* C₆H₅MgBr. *c* Δ . *d* H₂, Pd/C. *e* Na, NH₃. *f* 5% KOH in CH₃OH.

lie on the equatorial plane of the five-membered ring, whereas in **1b** only the methyl or the phenyl can occupy an equatorial position at one time. This suggests that **1a** has the greater thermodynamic stability.

In summary, the major intermediate (**4**) in the acid-catalyzed cyclodimerization of styrene is accessible by adjusting the temperature and/or the reaction time. Though A-15 is a superior catalyst for the cyclodimerization of α -methylstyrene,¹⁵ only a low yield of cyclodimerization products is realized with styrene. However, sulfuric acid^{2f} is effective in the dimerization of styrene to **1a** and **1b** (1:1). The base-catalyzed equilibrium ratio of indenes **8** and **9** (30:70) and indans **1a** and **1b** (80:20) were determined.

Experimental Section

Cyclodimerization of Styrene (2) by A-15. Several runs were made varying the reactant, catalyst, solvent ratios, and the length of the run. These ratios and times are given in Table II. The reaction mixtures were refluxed under a nitrogen atmosphere, filtered through Dicalite, and distilled to give a maximum yield of a mixture of 20% of **1a** and **1b**, bp 115–117 °C (1 mm), in a 1:1 ratio.

Table I. Reduction of Isomeric Methylphenylindenes with Sodium in Liquid Ammonia

	Sample, g	Sodium, g	% 1a	% 1b
8	1.5	0.7	77	23
8	1.0	0.4	79	21
9	1.0	0.4	82	18
9	1.0	1.0	84	16

Table II

2, mol	A-15, g	Cyclohexane, mL	Time, h
1.0	25	2.5	8
1.0	25	2.0	12
0.2	5	0.5	24
6.6	25	1.5	18
0.9	80	1.0	12

Table III

Time, h	% 1a,1b	% 4
4.0	13.5	86.5
8.5	59.4	40.6
16.0	86.2	13.8
24.0	99.0	1.0

The various runs were sampled and analyzed by gas chromatography¹¹ as shown in Table III. The product showed: IR (neat) 3025, 1600, 1495, 1455, 750 cm^{-1} ; mass spectrum (70 eV) m/e (rel intensity) 208 (M^+ , 100), 193 (70), 179 (31), 178 (30), 130 (41), 115 (49).

Cyclodimerization of 2 to 1 by H_2SO_4 . Freshly distilled 2 (400 g, 3.8 mol) was stirred into 1 L of a 62% solution of H_2SO_4 and treated as described.^{2f} Distillation yielded 285 g (82%) of 1a:1b (1:1), bp 114–116 °C (1 mm).

Dimerization of 2 to (E)-1,3-Diphenyl-1-butene (4). This dimerization was carried out as described^{3a} to give a 77% yield of 4: bp 123–125 °C (1 mm); IR (neat) 2780, 1440, 1005, 957, 737 cm^{-1} ; mass spectrum (70 eV) m/e (rel intensity) 208 (M^+ , 90), 193 (93), 178 (28), 130 (22), 115 (100), 91 (57); $^1\text{H NMR}$ (DCCl_3) δ 7.3 (m, 10, ArH), 6.4 (s, 2, $\text{HC}=\text{CH}$), 3.62 (m, 1, ArCH), 1.44 (d, 3, CH_3). A computer-controlled peak and intensity search of the Cyphernetics Mass Spectral file (27 000 spectra) identified the spectrum as that of 4. This spectrum was then directly compared with that of our reaction product, and they were found to be identical.

Cyclization of 4 to 1. By PPA.^{3a} A 16-g sample of 4 was stirred into 20 g of PPA preheated to 150 °C, and the mixture was allowed to stir at this temperature under a nitrogen atmosphere for 3 h. An 80% yield of 1a:1b (1:1)¹¹ was obtained.

By A-15. A 40-g sample of 4 was dissolved in 200 mL of cyclohexane, and 20 g of A-15 was added. The mixture was heated at reflux temperature under a nitrogen atmosphere for 4 h to give 1a:1b (1:1)¹¹ in 80% yield.

Cyclodimerization of 2 by Ethylaluminum Dichloride. EtAlCl_2 (0.02 mol, 2.77 g) was dissolved in benzene and slowly added to a stirred benzene solution (600 mL) containing 20 g of 2 (0.2 mol) under a nitrogen atmosphere.^{13,14} A red color developed immediately. Samples were removed periodically and analyzed by GC to determine the concentration of 1a and 1b.¹¹ The solution was stirred for 1 h at room temperature and 4 h at reflux temperature. The excess EtAlCl_2 was destroyed by adding 4 mL of methanol. The solution was washed with water and HCl, dried (MgSO_4), and filtered. The yield was low (24%), and other compounds were formed. The reaction was repeated as above using 0.2 mol of EtAlCl_2 (60% yield of product mixture).

Recrystallization of 1b. A 119-g sample of 1a:1b was recrystallized as described⁵ to give 1b in 95% purity: mp 25 °C (lit.⁵ 25.5 °C); IR (film) 747 cm^{-1} ; mass spectrum (70 eV) m/e (rel intensity) 208 (M^+ , 100), 193 (71), 130 (61), 115 (54), 91 (30); $^1\text{H NMR}$ δ 7.1 (m, 9, ArH), 4.3 (t, 1, Ar_2CH), 3.3 (m, 1, ArCH), 2.1 (q, 2, CH_2), 1.3 (d, 3, CH_3).

Synthesis of 1-Methyl-3-phenylindene (8). The benzene for this experiment was purified by refluxing it in the presence of AlCl_3 for 20 h, cooling, filtering through Na_2CO_3 , and distilling. Cinnamic acid (8.1 mol, 1203 g), 15.6 kg of benzene, and aluminum chloride (1824 g) were combined and treated as described.⁵ Instead of cyclizing

the 3,3-diphenylpropionic acid (5) via the acid chloride, 3 mol (675 g) of 5 was added to 6700 g of PPA that had been preheated to 90 °C. The mixture became yellow, and after 1.5 h of stirring, it was cooled to 70 °C, poured into ice water, and extracted with ether. The combined ether layers were washed with NaOH solution to remove acid, and then with water. The NaOH solution was later acidified to yield 223 g (33%) of 5. The ether layer was dried (MgSO_4), filtered, and distilled to yield a residual orange solid. After two recrystallizations from methanol 298 g of yellow crystals of 3-phenylindanone (6) was obtained (48% conversion, 72% yield), mp 74.5–75.5 °C (lit.⁵ 76.5–77.5 °C).

A 270-g (1.3-mol) sample of 6 dissolved in 900 mL of ether was added to 238 g of CH_3MgBr in 1500 mL of ether over a 20-min period. A green color developed which slowly faded to light yellow. The reaction mixture was heated at reflux for 2.5 h and then cooled. A 33% solution of NH_4Cl (300 mL) was added slowly to decompose the Grignard complex, and a 20% solution of HCl (200 mL) was used to dissolve salts. The product mixture was poured into ice water containing HCl and then extracted with ether (2 \times 1.5 L). The combined ether layers were washed with water, Na_2CO_3 , and water, then dried over MgSO_4 and filtered. Upon stripping the ether, some dehydration of 7 to 8 occurred. This dehydration was completed through use of refluxing toluene and a Dean–Stark trap.

Upon distillation, dehydration of the product mixture occurred to give 216 g of 8:9 (3:1). Several recrystallizations gave pure 8: mp 59–61 °C (lit.⁵ mp 63–64 °C); IR (KBr) 1600, 1070, 875, 845, 787, 765, 753, 695 cm^{-1} ; mass spectrum (70 eV) m/e (rel intensity) 206 (M^+ , 100), 205 (17), 191 (56), 189 (15), 165 (11); $^1\text{H NMR}$ (DCCl_3) δ 7.2 (m, 9, ArH), 6.24 (d, 1, $=\text{CH}$), 4.52 (m, 1, ArCH), 1.2 (s, 3, CH_3).

Hydrogenation of 8 to 1a. A 51.3-g sample of 8 was dissolved in 95% ethanol containing 10% (by weight) of 5% Pd/C catalyst. This mixture was hydrogenated at 25 psi with shaking until the pressure drop ceased (15–20 min). The suspension was filtered (Dicalite), concentrated, and distilled using a Kugelrohr apparatus to give 1a (39.5 g): IR (film) 731 cm^{-1} ; mass spectrum (70 eV) m/e (rel intensity) 208 (M^+ , 100), 193 (70), 178 (30), 130 (41), 115 (49); $^1\text{H NMR}$ (DCCl_3) δ 7.1 (m, 9, ArH), 4.1 (q, 1, Ar_2CH), 3.1 (m, 1, ArCH), 2.6 (m, 1, *trans*-HCH to Ph), 1.6 (m, 1, *cis*-HCH to Ph), 1.2 (d, 3, CH_3). A 70:30 mixture of 8 and 9 also gave only 1a when hydrogenated as above.

Synthesis of 3-Methyl-1-phenylindene (9). A 30-g sample of 3-phenylbutanoic acid (10) was added to 700 g of PPA preheated to 90 °C under a nitrogen atmosphere. A workup as described above and distillation gave 25 g (83%) of 3-methyl-1-indanone (11). A 30-g sample of 11 (25 g from the previous reaction plus 5 g prepared earlier) was dissolved in 50 mL of ether and then added slowly to 56 g of phenylmagnesium bromide in 500 mL of ether. After a workup similar to that described above, including dehydration by distilling, 32.5 g (92%) of 9 was obtained: mp 35–36 °C (lit.¹⁶ 36–37 °C); IR (KBr) 2850, 1340, 910, 820, 690 cm^{-1} ; $^1\text{H NMR}$ (DCCl_3) δ 7.40 (m, 9, ArH), 6.48 (d, 1, $=\text{CH}$), 3.56 (m, 1, ArCH), 1.38 (d, 3, CH_3).

General Procedure for Equilibration. Erlenmeyer flasks (125-mL) with a side arm were used in all the equilibration experiments. The top opening contained a one-hole neoprene stopper fitted with glass tubing, and nitrogen was passed through the side arm and out the stopper to a bubbler. A Teflon-enclosed magnetic stirring bar was used for agitation. The flasks were filled one-eighth with solvent and the other components were then added. Four flasks were used simultaneously and all were sampled periodically, using a pipet. Each sample was worked up in a 1-dram vial by adding water, then benzene, and shaking. The aqueous layer was removed by pipetting and the organic layer was washed three times with water. After the last aqueous layer was removed, a small amount of Na_2SO_4 was added to dry the benzene solution. GC studies were then carried out.¹¹

Equilibration of 8 by 5% KOH. A 0.2-g sample of 8 was added to a stirred solution of 4.2 g of KOH dissolved in 25 g of methanol. After 30 min, a ratio of 30:70 (8:9) was observed. This ratio remained constant during 2 days of observation.

Equilibration of 9 by 5% KOH. A 0.2-g sample of 9 was added to 4.2 g of KOH dissolved in 25 g of methanol, and the resulting solution was stirred for 3.5 h. A ratio of 30:70 (8:9) was observed.

General Procedure for Reduction of 8 or 9 with Sodium in Liquid Ammonia.^{17a,b} A 5 \times 15 cm cylindrical Pyrex reaction vessel containing a polyethylene-enclosed magnetic stirring bar, F openings for ammonia, a pressure-equalizing dropping funnel, a cold-finger reflux condenser, and a soda-lime guard tube was used to carry out the reduction.^{17b} The apparatus was dried by heating and passing nitrogen through it, and then ammonia was allowed to flow through the vessel for 10 min. Dry ice and acetone were added to the condenser, and ammonia was condensed. Sodium was added to the vessel via Gooch tubing. Once the sodium had disappeared, a dropping funnel

containing the compound (8 or 9) dissolved in ether was attached, and the solution was slowly added with stirring. After the mixture had been stirred from 1 to 1.5 h, NH_4Cl crystals were cautiously added until the blue color disappeared. Ammonia was allowed to evaporate, and the residue was poured into water and extracted by ether. The ether layer was dried (MgSO_4), filtered, and concentrated to give an oil, which was analyzed by GC. The data from duplicate runs on 8 and 9 are given in Table I.

Reduction of 8. Sodium (0.7 g) was dissolved in 50 mL of ammonia and a blue color developed immediately. A 1.5-g sample of 8 dissolved in 15 mL of dry ethyl ether was then added via a dropping funnel. No color change occurred in the solution. After stirring 1.5 h, the reaction mixture was worked up to give 1.3 g of oil. Gas chromatography analysis showed **1a:1b** (77:23).¹¹

The reduction of 1 g of 8 was repeated using 0.4 g of sodium to give **1a:1b** (79:21).¹¹

Reduction of 9. A 1-g sample of 9 and 0.4 g of sodium treated as above gave **1a:1b** (82:18). This was repeated using 1 g of sodium, which resulted in **1a:1b** (84:16).¹¹

Equilibration of 1a by NaNH_2 . The apparatus described in the reduction procedure was used. A 1-g sample of 1a dissolved in 15 mL of dry ether was slowly added to a solution containing 0.3 g of sodium dissolved in 50 mL of liquid ammonia, which contained a crystal of FeSO_4 . When GC studies showed no composition change, the solution was worked up as described in the reduction procedure to yield 1 g of an oil. GC analysis showed that this oil contained **1a:1b** (82:18).¹¹

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Registry No.—**1a**, 14568-75-3; **1b**, 14568-76-4; **2**, 100-42-5; **4**, 7302-01-4; **5**, 606-83-7; **6**, 16618-72-7; **8**, 22360-63-0; **9**, 22360-6-9; **10**, 4593-90-2; **11**, 6072-57-7; cinnamic acid, 621-82-9.

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Preparation and Properties of RMgH and RMg_2H_3 Compounds

E. C. Ashby* and A. B. Goel

School of Chemistry, Georgia Institute of Technology, Atlanta, Georgia 30332

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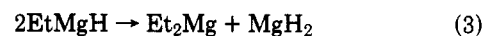
A series of alkyl- and arylmagnesium hydrides, RMgH (where R = Me, Et, *i*-Pr, Cp, and Ph), has been prepared by the reaction of R_2Mg compounds with an active form of magnesium hydride slurry in THF. These products could also be prepared by the reaction of RLi compounds with hydridomagnesium halide (HMgX , where X = Cl and Br), as demonstrated by the preparation of methylmagnesium hydride by the reaction of CH_3Li with HMgCl in THF. Preparation of compounds of the type RMg_2H_3 (where R = Me and Ph) has also been carried out. Contrary to earlier reports, the RMgH compounds have been found to be soluble and quite stable in THF at room temperature. A band in the region 1250-1300 cm^{-1} in the infrared spectrum of these compounds has been assigned to Mg-H stretching. This band is shifted to 940 cm^{-1} in the deuterio analogues, RMgD . Molecular weight studies of methyl- and ethylmagnesium hydride show these compounds to be dimeric in dilute solution.

The existence of compounds of the type RMgH has been the subject of interest and speculation for many years, as these compounds are analogues of Grignard reagents. Rice and co-workers¹ in 1956 reported the formation of PhMgH by the reaction of PhMgBr with LiAlH_4 in 4:1 ratio in ether. Later we showed² that the products of this reaction were not the same as reported by Rice. In a communication in 1962, Bauer³ reported the preparation of $\text{C}_2\text{H}_5\text{MgH}$ by the reaction of silane with $(\text{C}_2\text{H}_5)_2\text{Mg}$ in ether (eq 1).



However, he provided no characterization of the product. Sometime later, Coates and Heslop⁴ reported evidence for the

formation of $\text{C}_2\text{H}_5\text{MgH}$ as an intermediate in the reaction of $(\text{C}_2\text{H}_5)_2\text{Mg}$ and $\text{NaB}(\text{C}_2\text{H}_5)_3\text{H}$ (eq 2); however, they reported that the compound was stable only at -78°C and dissociated at -20°C to give MgH_2 and $(\text{C}_2\text{H}_5)_2\text{Mg}$ (eq 3).



Our earlier attempts⁵ to prepare $\text{C}_2\text{H}_5\text{MgH}$ by the reaction of LiAlH_4 with $(\text{C}_2\text{H}_5)_2\text{Mg}$ in ether in 1:4 ratio and by the reaction of MgH_2 with $(\text{C}_2\text{H}_5)_2\text{Mg}$ in ether were not successful in that MgH_2 was recovered in both cases. In this communi-