

very slow induced decomposition of cumene hydroperoxide was reported to occur with AIBN.¹⁹

Competitive Reactions.—Competitive autoxidations were carried out with an equimolar mixture of two substituted isopropylbenzenes (each ca. 1.2–0.6 M) in chlorobenzene at 60° in the presence of AIBN (0.26–0.09 M). Aliquots (each 1 ml) were taken out at appropriate intervals of time and reduced with 15% KSH in aqueous methanol. After completion of reduction, the solution was washed with water, extracted with two 4-ml portions of chloroform, and analyzed by means of glpc. The products were analyzed by a Yanagimoto gas chromatograph with a flame ionization detector, Model GCG-550F, employing

(19) J. A. Howard and K. U. Ingold, *Can. J. Chem.*, **46**, 2655 (1968).

a 1.0 m × 2.5 mm column packed with Apiezon Grease (5%) on Chamelite CS of 80–100 mesh using N₂ as a carrier gas at 160–220°. The internal standards for glpc were nitrobenzene for cumene and *m*-diisopropylbenzene for isopropylcumyl hydroperoxide and isopropylcumyl alcohol. The relative rate constants of competitive oxidation were calculated from the equation $k = k_m/k_p = \log [(b - y)/b] / \log [(a - x)/a]$. Here, *a* and *b* are initial concentrations of substrates and *x* and *y* are corresponding concentrations at time *t*.

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Spiro Hydrocarbons and Dibenzo[*c,p*]chrysene from 1-Tetralone

JOHN W. BURNHAM,^{1a} ROBERT G. MELTON,^{1b} AND EDMUND J. EISENBRAUN*

Department of Chemistry, Oklahoma State University, Stillwater, Oklahoma 74074

GARY W. KEEN AND MYNARD C. HAMMING

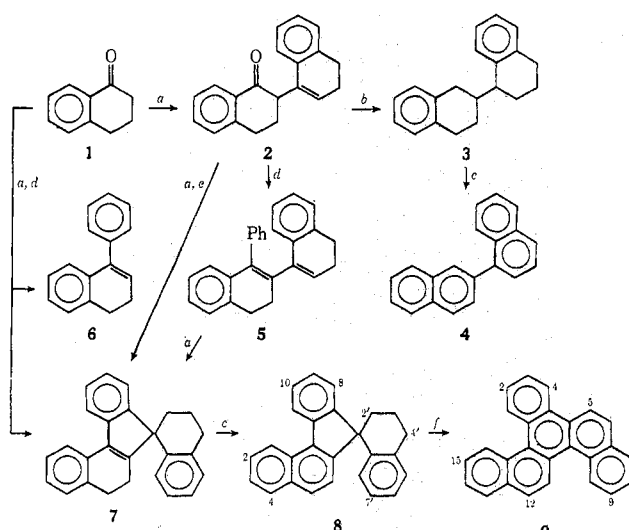
Research and Development Department, Continental Oil Company, Ponca City, Oklahoma 74601

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1-Tetralone (1) and phenylmagnesium bromide yield 3,4-dihydro-1-phenylnaphthalene (6) and 1',2',3',4',5,6-hexahydrospiro[7*H*-benzo[*c*]fluorene-7,1'-naphthalene] (7). The latter is formed in a series of reactions involving the self-condensation of 1 to 2-(3,4-dihydro-1-naphthyl)-3,4-dihydro-1(2*H*)-naphthalenone (2), addition of Grignard reagent, acid-catalyzed dehydration to the diene 5, and its subsequent cyclization to 7. The latter was dehydrogenated with Pd/C to 1',2',3',4'-tetrahydrospiro[7*H*-benzo[*c*]fluorene-7,1'-naphthalene] (8) and then 8 was converted to dibenzo[*c,p*]chrysene (9) by heating in the presence of Pd/C and sulfur.

1-Tetralone (1) serves as a useful starting material in the synthesis of 3,4-dihydro-1-phenylnaphthalene (6) or 1-phenylnaphthalene.² It is of interest that a low yield (2–3%) of 1',2',3',4',5,6-hexahydrospiro[7*H*-benzo[*c*]fluorene-7,1'-naphthalene] (7) is formed during the acid-catalyzed work-up. The latter is more conveniently prepared from either 2 or 5 as shown in Scheme I.

SCHEME I



*Amberlyst-15, toluene, Δ. ^bPd/C, H₂, CH₃CO₂H. ^cPd/C, Δ. ^dC₆H₅·MgBr, H₃O⁺. ^eC₆H₅Li. ^fPd/C, S, Δ.

(1) (a) American Petroleum Institute Graduate Research Assistant, 1969–1973; Continental Oil Company Fellow, 1973; (b) American Petroleum Institute Graduate Research Assistant, 1968–1971.

(2) (a) R. Weiss, "Organic Syntheses," Collect. Vol. III, Wiley, New York, N. Y., 1955, p 729. (b) The distillation residue obtained by the procedure of ref 2a weighed 7 g and yielded 3% of 7 (based on 0.4 mol of 1) after treatment with Amberlyst-15. Cf. Experimental Section for inverse addition of phenylmagnesium bromide and the phenyllithium procedure leading to 7.

Although 2 and 5 may readily be detected by glc as products during the preparation of 6, their direct isolation, particularly that of 2, is difficult. Accordingly, to establish that 2 and 5 may be intermediates to 7, and to obtain a quantity of 7, we prepared 2^{3–5} and 5 and then used these compounds for the synthesis of 7, 8, and 9 as shown in Scheme I. Also to substantiate the 2,1' linkage of 2, we converted it *via* 3 to 1,2'-binaphthyl (4).⁶

The best preparation of 7 (55%) resulted from addition of phenyllithium to 2 (cf. steps *a* and *e* of Scheme I). The latter was completely consumed in this reaction. This preparation of 7 involves acid-catalyzed dehydration and cyclization.^{5,7a} We also noted that the reaction of 2 with phenylmagnesium bromide, regardless of mode of addition, was incomplete, at least 30% of 2 being recovered. By cautious acidification^{7b} of Grignard reaction products, the diene 5 may be isolated. It, in turn, is readily converted to 7 with Amberlyst-15.^{5,7a}

Inverse addition (adding phenylmagnesium bro-

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(4) (a) H. L. Retcofsky, L. Reggel, and R. A. Friedel, *Chem. Ind. (London)*, 617 (1969); (b) M. Orchin, L. Reggel, and R. A. Friedel, *J. Amer. Chem. Soc.*, **71**, 2743 (1949).

(5) We thank Rohm and Haas Co., Philadelphia Pa., for a sample of Amberlyst-15 sulfonic acid resin. Literature describing its use may be obtained from this source.

(6) Correspondence regarding samples of 4, 1-phenylnaphthalene, 6, and 8 should be addressed to A. J. Streiff, American Petroleum Institute, Carnegie-Mellon University, Pittsburgh, Pa. 15213.

(7) (a) Amberlyst-15⁵ in boiling benzene or toluene was effective in causing dehydration and cyclization to 7. (b) Cold hydrochloric acid was used to decompose Grignard reaction products and to cause dehydration to 5. (c) A structure analogous to 7 was proposed earlier for a product obtained from the reaction of *o*-tolylmagnesium bromide and 1: M. Orchin, L. Reggel, and R. A. Friedel, *J. Amer. Chem. Soc.*, **73**, 1449 (1951).

mide to 1) gave 12% of 7 as final product. Presumably the higher concentration of ketone 1 in this mode of addition permits self-condensation to 2 as compared to addition of the phenyl group to 1.

To establish the structure^{7c} of 7, it was dehydrogenated to the spiro hydrocarbon 8 by heating in the presence of Pd/C. The strongest argument for the structure of these spiro hydrocarbons is the anisotropy exhibited by the C-1, C-11, and C-8' protons in the pmr spectra (100 MHz) of both hydrocarbons 7 and 8. Dreiding models of 7 and 8 indicate that the C-1 and C-11 protons should experience deshielding.^{8a} For 7, this strong interaction results in a downfield triplet at δ 7.88 for both protons. For 8, the C-1 and C-11 protons give separated signals (pair of doublets) shifted to δ 8.79 and 8.37, respectively. Strong shielding is observed for the C-8' proton of 7 and 8. These high-field shifts appear as doublets centered at δ 6.49 and 6.25, respectively. Dreiding models of 7 and 8 also show that the C-8' proton is situated above the aromatic rings of the fluorene system and hence should be influenced by aromatic ring currents.^{8b}

Aromatization of 8 to dibenzo[*c,p*]chrysene (9) was accomplished by heating it in the presence of Pd/C and sulfur.^{8c} The structure of 9 is supported by its high-resolution mass spectrum, which shows a molecular ion peak (m/e 328), peaks resulting from loss of 1, 2, and 4 H atoms, and the formation of doubly charged ions m/e 164 (M)²⁺, 163 ($M - 2H$)²⁺, and 162 ($M - 4H$)²⁺. The m/e 326 ion apparently loses CH and C₂H₂ to yield doubly charged ions m/e 156.5 and 150. These fragmentations are characteristic of condensed polynuclear aromatic hydrocarbons.^{9a,b} The pmr spectrum of 9 shows a multiplet of six aromatic protons at δ 9.56–8.19. This corresponds to the bay protons at positions C-4 and C-5 and fjord protons at C-1, -10, -11, and -16. The remaining ten peninsular protons give rise to an upfield multiplet centered at δ 7.93. A similar spectrum was reported for dibenzo[*g,p*]chrysene.^{9c}

Experimental Section¹⁰

Conversion of 1-Tetralone (1) to 2-(3,4-Dihydro-1-naphthyl)-3,4-dihydro-1(2H)-naphthalenone (2).—1-Tetralone (292 g, 2 mol), 30 g of Amberlyst-15,⁵ and 750 ml of dry toluene were combined in a 2-l., one-neck flask equipped with a Dean-Stark trap. The mixture was heated at reflux for 4.5 hr with magnetic stirring until production of water (4 ml) ceased. The reaction mixture was cooled, filtered, and concentrated with a rotary evaporator. The concentrated oil was mixed with 100 ml of ether, and the yellow-white crystals of 2 (37 g) that formed after

refrigeration for 2 days were filtered out. The mother liquor was distilled at 80° (0.1 mm) to give 193 g of recovered 1. A small forerun containing naphthalene was collected. Ether (150 ml) was added to the cooled viscous pot residue which then crystallized on seeding. An additional 45 g of 2 was obtained as brown crystals. The combined yield of crude 2 was 91% based on recovered 1. This mixture was washed with ether and recrystallized from acetone to give 2 as colorless crystals: mp 132.5–135° (lit.^{4b} mp 132.5–134.2°); orange 2,4-dinitrophenylhydrazine mp 249–250° dec (lit.^{4b} mp 247–248°); mass spectrum (70 eV) m/e (rel intensity) 274 (77), 146 (75), 129 (100), 43 (97), 29 (91); pmr (CDCl₃) δ 8.24 (m, 1, isolated ArH at C-8), 7.63–7.24 (m, 7, ArH), 5.88 (t, 1, vinylic), 3.89 (t, 1, C=CCH and adjacent to C=O), 3.21–2.02 (m, 8, -CH₂-); uv as previously recorded.^{3b,4b}

Pd/C-Catalyzed Hydrogenation of 2 to 1,2,3,4,1',2',3',4'-Octahydro-1,2'-binaphthyl (3).—A 100-g (0.36 mol) sample of 2 in 400 ml of acetic acid in the presence of 5 g of 10% Pd/C was hydrogenated at 50 psi and at 65° for 12 hr. The catalyst was filtered out with Dicalite. Water (1.5 l.) was added to the filtrate and the mixture was extracted with ether (2 × 500 ml). The extract was washed with water and 100 ml of 10% sodium hydroxide, dried (MgSO₄), filtered, and distilled to give 89 g (95%) of 3: bp 165° (0.1 mm) [lit.¹¹ bp 175–180° (0.2 mm)]; mass spectrum (70 eV) m/e (rel intensity) 262 (8), 132 (21), 131 (100), 130 (30), 129 (17), 115 (15), 91 (22); pmr (CCl₄) δ 7.32–6.72 (m, 8, ArH), 3.08–1.12 (envelope, 14, ArCH, ArCH₂, and -CH₂-); uv as previously recorded.¹¹

Pd/C-Catalyzed Dehydrogenation of 3 to 1,2'-Binaphthyl (4).—A 89-g (0.34 mol) sample of 3 and 5 g of 10% Pd/C were heated together at 300° under nitrogen for 2.5 hr. The cooled mixture was dissolved in benzene and filtered through Dicalite to remove catalyst. An equal portion of petroleum ether^{10f} was added and the solution was decolorized by elution through a 1 × 4 in. column of basic alumina. The solvents were removed by rotary evaporation to give 86 g of crystalline 4. Recrystallization from petroleum ether^{10f} gave 80 g (93%) of 4 free of impurity by glc:^{10a} mp 76–78° (lit.¹¹ mp 76.5–77.5°); mass spectrum (70 eV) m/e (rel intensity) 254 (100), 253 (72), 252 (53), 250 (13), 127 (10), 126 (27); pmr (CDCl₃) δ 8.05–7.18 (m, ArH).

Conversion of 1-Tetralone (1) to 3,4-Dihydro-1-phenylnaphthalene (6) and 1',2',3',4',5,6-Hexahydrospiro[7H-benzo[*c*]fluorene-7,1'-naphthalene] (7).—The preparation of 6 from 584 g (4 mol) of 1 and 4.8 mol of phenylmagnesium bromide was carried out as described^{2a} except that commercial Grignard reagent¹² was substituted and Amberlyst-15⁵ in boiling toluene was used for dehydration. Glc studies^{10a} at 240° of this reaction mixture showed the presence of 1-phenyl-1,2,3,4-tetrahydronaphthalene-6-1-phenylnaphthalene in a ratio of 5:80:15. The hydrocarbon mixture was distilled at 95–99° (0.01 mm) through an 18-in. vacuum-jacketed Vigreux column to give 486 g (67%) of crude 6 and 32 g of distillation pot residue. Redistillation^{10a} gave pure 6: bp 91° (0.01 mm) [lit.¹³ bp 130.5–135.5° (0.3 mm)]; mass spectrum (70 eV) m/e (rel intensity) 206 (100), 205 (29), 202 (18), 191 (38), 128 (25), 91 (22); pmr (CCl₄) δ 7.36–6.97 (m, 9, ArH), 5.96 (t, 1, vinylic), 2.91–2.59 (m, 2, ArCH₂-), 2.44–2.03 (m, 2, allylic); uv max (95% ethanol) 205 m μ (log ϵ 4.39), 220 (4.36), 267 (3.91).

The identity of the glc peaks assigned to 1-phenyl-1,2,3,4-tetrahydronaphthalene and 1-phenylnaphthalene in the reaction product mixture was established by glc^{10a} comparison at 225° with authentic materials. Samples of these hydrocarbons were obtained from 6 by catalytic hydrogenation and catalytic dehydrogenation in the presence of 10% Pd/C catalyst.

The pot residue (32 g) was recrystallized twice from benzene to give colorless crystals of 7: mp 189–190°; mass spectrum (70 eV) m/e (rel intensity) 334 (100), 305 (12), 303 (12), 289 (10), 229 (11), 215 (21); pmr (CDCl₃) δ 7.88 (t, 2, isolated ArH at C-1 and C-11), 7.42–6.72 (m, 9, ArH), 6.49 (d, 1, ArH at C-8'), 3.08–2.56 (m, 4, ArCH₂), 2.50–1.61 (m, 6, ArCH₂CH₂- and ArCH₂-CH₂CH₂-); uv max (95% ethanol) 203 m μ (log ϵ 4.75), 238 (4.46), 266 (3.87), 294 (3.86).

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(9) (a) M. C. Hamming and N. G. Foster, "Interpretation of Mass Spectra of Organic Compounds," Academic Press, New York, N. Y., 1972; (b) R. Engel, D. Halpern, and B.-A. Funk, *Org. Mass Spectrom.*, **7**, 177 (1973); (c) R. H. Martin, N. Defay, F. Geerts-Evrard, and S. Delavarene, *Tetrahedron*, **20**, 1073 (1964).

(10) (a) The glc studies used a Hewlett-Packard Model 5750 instrument with a 0.25 in. × 12 ft column of 80–100 mesh Chromosorb G (acid washed and DMCS treated) treated with 5% silicone rubber UC W-98. (b) A similar 0.25 in. × 5 ft column of 100–120 mesh Chromosorb W (AW and DMCS treated) coated with 5% UC W-98 was used for analysis of the C₂₈ hydrocarbons. (c) Nmr spectra were obtained with a Varian XL100 spectrometer. (d) Mass spectra were obtained with CEC Model 21-103C and 21-110B spectrometers. (e) Ir and uv spectra were obtained with Beckman IR-5A and Cary 14 spectrometers, respectively. (f) The petroleum ether, bp 60–68°, was redistilled before use. (g) A Nester/Faust autoannular spinning band distillation system, Model NFA-200, was used.

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(12) Phenylmagnesium bromide was obtained from Arapahoe Chemicals, Boulder, Colo.

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Anal. Calcd for $C_{26}H_{22}$: C, 93.37; H, 6.63. Found: C, 93.23; H, 6.79.

Inverse Addition of Phenylmagnesium Bromide to 1 to Form 6 and 7.—To a mechanically stirred solution of 44 g (0.3 mol) of 1 in 500 ml of dry ether at 10° was added 200 ml (0.6 mol) of 3 M phenylmagnesium bromide¹² over a 15-min period. Reflux was established after 5 min and the mixture was stirred for 1.5 hr. The reaction mixture was then added to 500 g of ice and 200 ml of concentrated hydrochloric acid. The ether extract was dried ($MgSO_4$) and concentrated to give 63 g of red-brown oil. The oil was dissolved in 350 ml of toluene and stirred at reflux for 1.5 hr with 3 g of Amberlyst-15.⁵ Filtration and steam distillation gave 41 g of volatile hydrocarbon and 20 g of nonsteam-volatile material. The nonvolatiles were dissolved in petroleum ether^{10f} and percolated through a 2 × 3 in. column of basic alumina; concentration of the effluent and crystallization from petroleum ether^{10f} gave 6 g (12%) of 7.

Inverse Addition of Phenylmagnesium Bromide to 2 to Form 1-Phenyl-3,3',4,4'-tetrahydro-2,1'-binaphthyl (5).—To a stirred mixture of 27.4 g (0.1 mol) of 2 in 500 ml of dry ether was added 67 ml (0.2 mol) of a 3 M phenylmagnesium bromide¹² solution during 5 min. There was no apparent temperature change; the mixture was then heated at reflux for 24 hr. The reaction mixture was added to 500 g of ice and 50 ml of concentrated hydrochloric acid. Extraction with benzene (250 ml), drying ($MgSO_4$), and concentration gave 37 g of yellow oil. Petroleum ether^{10f} (150 ml) was added and 10 g of 2 was recovered on cooling and filtering. The filtrate was percolated through two 2 × 3 in. columns of Merck basic alumina to give 16 g of concentrated oil. This oil crystallized from 25 ml of cold acetone after 3 days to give 8.5 g (40%) of 5: mp 95–97°; mass spectrum (70 eV) m/e (rel intensity) 334 (100), 333 (12), 332 (11), 305 (11), 215 (10), 117 (11); pmr (CCl_4) δ 7.26–6.62 (m, 13, ArH), 5.54 (t, 1, vinylic), 3.04–2.76 (m, 2, $ArCH_2$ at C-4), 2.70–2.28 (m, 4, $ArCH_2CH_2$ and $ArCH_2$ at C-3 and C-4', respectively), 2.10–1.81 (m, 2, $ArCH_2CH_2$ at C-3'); uv max (95% ethanol) 205 $m\mu$ (log ϵ 4.65), 267 (3.97).

Anal. Calcd for $C_{26}H_{22}$: C, 93.37; H, 6.63. Found: C, 93.18; H, 6.68.

Amberlyst-15-Catalyzed Cyclization of 5 to 7.—Three grams of 5 was cyclized over 30 min by heating in 150 ml of boiling toluene containing 2 g of Amberlyst-15.⁵ The reaction mixture was cooled, filtered, and concentrated and the crude product was crystallized from 50 ml of petroleum ether^{10f} to give 2.7 g (90%), mp 189–190°, found to be identical with 7 from other experiments.

Conversion of 2 to 7 Using Phenyllithium.—Phenyllithium (0.4 mol) was prepared as described¹⁴ from 63 g of bromobenzene and 3 g of Li. To the stirred reagent was added, during 40 min at 25–30°, 27.4 g (0.1 mol) of 2 dissolved in 300 ml of dry benzene. The mixture was heated at reflux for 10 hr. During this period the temperature rose from the boiling point of ether to that of benzene. The reaction mixture was cooled and added to ice and 300 ml of 10% HCl. Extraction with ether gave 34 g of concen-

trated oil, ir (neat) 3460 cm^{-1} (OH). The oil was dehydrated and cyclized with 3 g of Amberlyst-15⁵ in 300 ml toluene heated at reflux temperature for 1 hr. Two milliliters of water was collected. The filtered and concentrated product was dissolved in 200 ml of toluene-petroleum ether (1:1) and the mixture was passed through a 1.5 × 3 in. column of basic Merck alumina. Removal of the solvent and crystallization from 75 ml of toluene gave 18 g (55%) of colorless 7, mp 188–190°. This sample was found to be identical with other samples of 7.

Pd/C Catalyzed Dehydrogenation of 7 to 1',2',3',4'-Tetrahydrospiro[7H-benzo[c]fluorene-7,1'-naphthalene] (8).—A 18.3-g sample of 7 and 3 g of 10% Pd/C were heated together at 310° (bath temperature) for 20 min under a blanket of N_2 . The cooled product mixture was dissolved in chloroform and filtered through Dicalite, the chloroform removed by rotary evaporation, and 50 ml of petroleum ether was added to the oil. Refrigeration and filtration gave 16.9 g (92%) of 8 as white plates: mp 157–159°; mass spectrum (70 eV) m/e (rel intensity) 332 (100), 304 (17), 303 (52), 302 (6), 300 (8), 151 (13); pmr ($CDCl_3$) δ 8.79 (d, 1, isolated ArH at C-1), 8.37 (d, 1, isolated ArH at C-11), 7.98–6.95 (m, 10, ArH), 6.76 (t, 1, isolated ArH at C-7'), 6.25 (d, 1, isolated ArH at C-8'), 3.24–3.01 (m, 2, $ArCH_2$), 2.41–1.95 (m, 4, $ArCH_2CH_2CH_2$); uv max (95% ethanol) 204 $m\mu$ (log ϵ 4.70), 237 (4.69), 252 (4.49 sh), 306 (3.97 sh), 317 (4.10), 326 (4.05), 342 (4.16).

Anal. Calcd for $C_{26}H_{20}$: C, 93.94; H, 6.06. Found: C, 93.79; H, 6.14.

The Pd/C and Sulfur Dehydrogenation of 8 to Dibenzo[c,p]-chrysene (9).—A 2-g sample of 8 was heated under nitrogen at 325° in the presence of 0.75 g of 10% Pd/C and 0.75 g of sulfur for 10 min. The mixture was cooled, dissolved in benzene, and filtered through Dicalite to give a green solution. This solution was diluted with an equal volume of petroleum ether^{10f} and passed through a 1.5 × 2.5 in. column of Merck acidic alumina. Concentration and trituration with petroleum ether^{10f} gave 1.2 g of yellow 9: mp 200–202° dec; mass spectrum (70 eV) m/e (rel intensity) 328 (100), 327 (33), 326 (40), 324 (15) [accurate mass values (± 0.003 of theoretical) were obtained for the doubly charged ions 164 (7), 163 (14), and 162 (15)]; pmr ($CDCl_3$) δ 9.56–8.19 (m, 6, ArH), 8.19–7.66 (m, 10, ArH); uv max (95% ethanol) 213 $m\mu$ (log ϵ 4.64), 276 (4.84), 295 (4.71), 305 (4.79), 334 (4.09), 350 (3.87).

Anal. Calcd for $C_{26}H_{16}$: C, 95.09; H, 4.91. Found: C, 95.03; H, 4.91.

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