no change in nmr or tlc from that of the starting mixture. Gas chromatography-mass spectra of the recovered material: *m/e* (rel intensity) major identical with that of the minor component of the 70-mg sample; minor 211 (15), 180 (15), 152 (9), 124 (26), 110 (100), 97 (31).[28]

Application of this technique to the 70-mg sample of **17** + **18** likewise resulted in no change in tlc.

**Acknowledgments.** One of us (G. S.) wishes to express appreciation to Merck and Co. for financial assistance. We are grateful to Merck and Co. for permitting use of their gas chromatography-mass spectral facilities and to Mr. Jack L. Smith for running these spectra.

**Registry No.**—**1a,** 38896-30-9; **1b,** 51552-68-2; **2a** cis epimer 1, 51552-69-3; **2a** cis epimer 2, 51552-70-6; **2a** trans epimer, 51552-71-7; **2b** cis epimer 1, 51552-72-8; **2b** cis epimer 2, 51552-73-9; **2b** trans epimer, 51552-74-0; **3a,** 51552-75-1; **3b,** 19384-65-7; **3c,** 51552-76-2; **3d,** 39011-44-4; **5,** 51552-77-3; **6** isomer 1, 51552-78-4; **6** isomer 2, 51552-79-5; **6** isomer 3, 51552-80-8; **6** isomer 4, 51552-81-9; **7,** 51552-82-0; **8,** 51552-83-1; **9,** 2050-43-3; **10,** 33890-03-8; **11,** 51552-84-2; **13,** 51552-85-3; **14,** 51552-86-4; **15,** 51552-87-5; **17,** 51552-88-6; **18,** 51552-89-7; 4-[(2-carboxyethyl)amino]isophthalic acid, 51552-90-0; quinoline-6-carboxylic acid, 10349-57-2; 1-acetyl-6-carboxy-2,3-dihydro-4(1*H*)-quinolone, 51552-91-1.

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(28) This spectrum should be identical with that of the major component of the 70-mg sample of **17** + **18**. The differences that are observed are presumably because of the presence of the $R_f$ 0.29 unknown reported in the experimental data for **16**.

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# An Improved Synthesis of Indenes. II. Alkyl-Substituted Indenes[1]

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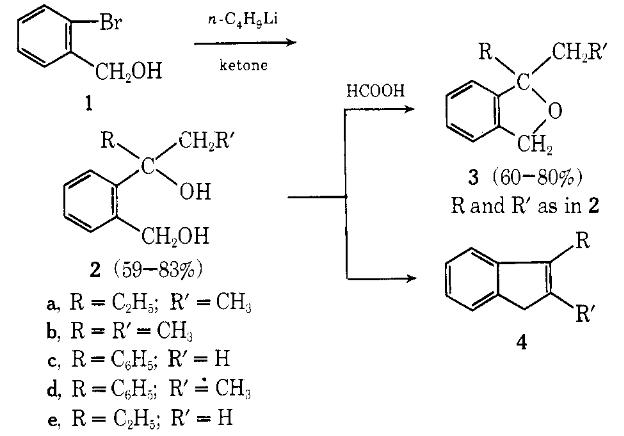
Dialkylphthalans **3**, unlike their spiro analogs, are stable to both hot formic acid and boron trifluoride etherate. Dialkyl indenes **4** (R and R′ alkyl) can, however, be prepared from diols **2** by a new procedure which involves heating the derived monoacetate **6** with hot acetic anhydride-formic acid. While this procedure is not efficient for the preparation of 3-arylindenes, the latter can be prepared in good yield from the corresponding diol **2** or phthalan **3** by use of either sulfuric acid-carbon tetrachloride (0°) or polyphosphoric acid (60°).

While fused indenes can be easily prepared[2] from spirophthalans analogous to **3**, or from the corresponding diols analogous to **2**, our initial attempts to extend this synthesis to simple acyclic indenes **4** were unsuccessful; results consistent with earlier reports.[3] We have now studied a series of acyclic diols **2** and phthalans **3** (Scheme I) and have defined useful conditions for their conversion to indenes **4**.

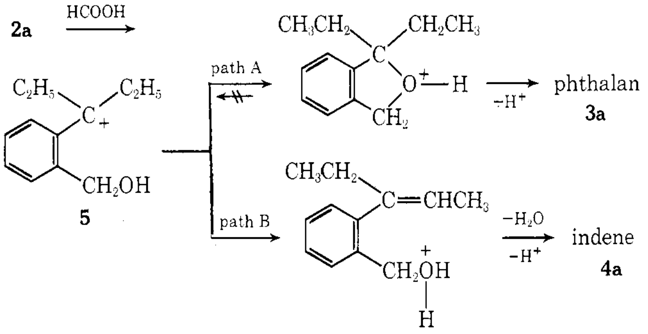
Phthalans **3a** and **3b** (where R = alkyl), in sharp contrast[2] to their cyclic analogs, are quite stable to hot formic acid and to hot boron trifluoride etherate in hot benzene; no evidence of decomposition or indene formation was noted. With stronger acids (PPA, $H_2SO_4$) these phthalans gave small amounts of indenes; however, higher condensation products predominate and the procedure is of no synthetic value.

It was observed that reaction of diol **2a** with hot formic acid gave **3a** and, significantly, some indene **4a** (8%). Since **3a** does not give **4a** under these conditions, it was concluded that diols **2** could be converted to indenes **4** by a process not involving phthalans **3**, and that successful conversion of **3a** to indene depended upon inhibiting path A relative to path B in Scheme II. Replacement of the primary hydroxyl group in **2** by acetoxy, as in **6** (Scheme III), was investigated since $CH_3C{=}O^+$ would be a poorer leaving group than $H^+$ which, consequently, should inhibit path A relative to path B (Scheme II). Satisfactory yields of indenes **4a** (60%) and **4b** (59%) were formed to-

Scheme I

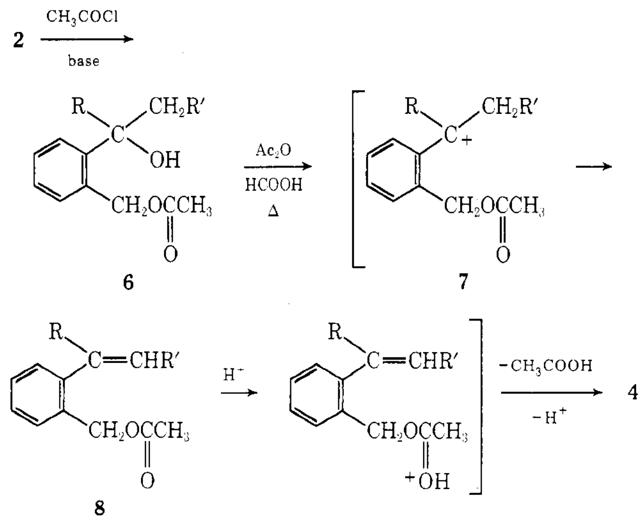


Scheme II



gether with some corresponding phthalan by the procedure outlined in Scheme III; 2,3-dimethylidene (**4b**) formed to the exclusion of 3-ethylidene (**4e**).

Scheme III



The acetate procedure, summarized in Scheme III, was not satisfactory for preparation of the 3-arylindenes. Reaction of the corresponding monoacetate **6c** (R = $C_6H_5$; R′ = H) and the eneacetate **8c** (R = $C_6H_5$; R′ = H) with hot acetic anhydride and formic acid gave principally phthalan **3c** with only low conversion to indene. Presumably, the greater stability of **7** where R = aryl, relative to **7** where R = alkyl, is sufficient to permit reaction of the type shown in path A (Scheme II) to dominate in these cases.

3-Arylindenes of type **4c** and **4d** can be prepared from diols of type **2** or the corresponding phthalans **3** by use of stronger acids. Both Pittman and Miller[4] and Bertoli and Plesch[5] have studied spectral behavior of 3-arylindenes in acid media at low temperature. The result of these studies indicate that 3-arylindenes are stable over long periods of time at low temperature (−50 to 0°) in strong acid. Reaction of diol **2c** and phthalan **3c** in a two-phase system in carbon tetrachloride and sulfuric acid[6] at 0° gave 57 and 59%, respectively, of **4c**; the dialkyl phthalans **3a** and **3b** gave complex mixtures under these conditions.

In certain cases polyphosphoric acid can be employed to give 3-arylindenes. Reaction of phthalan **3c** with PPA at 30° resulted in no reaction; reaction at 60° gave a complex mixture. By contrast, reaction of 3-phenyl-3-ethylphthalan (**3d**) with PPA at 60° gave an 82% yield of 2-methyl-3-phenylindene (**4d**). Polyphosphoric acid at 30° is not effective for conversion of either **3c** or **3d** to indenes; its use at higher temperature is limited to the preparation of indenes which are stable to higher reaction temperatures in such strong acid medium.

## Experimental Section

Diols **2** were prepared essentially as previously described[2] for related compounds.

**1-(2-Hydroxymethylphenyl)-1-phenylethanol (2c):** 59% yield; mp 109–110° (lit.[7] mp 109–110°) from petroleum ether[8]–acetone; pmr δ 7.9–7.0 (m, aromatic H, 8.9), 5.20 (broad s, OH, 0.97), 4.03 (broad s, $C_6H_5CH_2$ and OH, 3.0), 1.83 (s, $CH_3$, 3.0).

*Anal.* Calcd for $C_{15}H_{16}O_2$: C, 78.92; H, 7.06. Found: C, 78.90; H, 6.99.

**1-(2-Hydroxymethylphenyl)-1-phenylpropanol (2d):** 76% yield; mp 89–90.5° from petroleum ether[8]–acetone; pmr δ 7.8–7.1 (m, aromatic H, 8.9), 4.13 (s, $C_6H_5CH_2$, 1.9), 3.90 (broad s, OH, wt 1.9), 2.26 (q, $CH_2CH_3$, wt 2.1), 0.83 (t, $CH_2CH_3$, wt 2.9).

*Anal.* Calcd for $C_{16}H_{18}O_2$: C, 79.31; H, 7.49. Found: C, 79.56; H, 7.48.

**2-(2-Hydroxymethylphenyl)butan-2-ol (2b):** 80% yield; bp 100–103° (0.07 mm); pmr δ 7.13 (m, aromatic H, 3.9), 4.70 (q, $C_6H_5CH_2$, 1.9), 4.06 (broad s, OH, 1.9), 1.90 (q, $CH_2CH_3$, 2.1), 1.55 (s, $CH_3$, 3.1), 0.78 (t, $CH_2CH_3$, 3.1).

*Anal.* Calcd for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95. Found: C, 73.48; H, 9.06.

**3-(2-Hydroxymethylphenyl)pentan-3-ol (2a):** 84% yield; mp 78–79° (lit.[9] mp 81–82°) from petroleum ether;[8] pmr δ 7.20 (m, aromatic H, 4.0), 4.77 (s, $C_6H_5CH_2$, 1.9), 3.97 (broad s, OH, 1.93), 1.92 (q, $CH_2CH_3$, 4.0), 0.78 (t, $CH_2CH_3$, 6.1).

*Anal.* Calcd for $C_{12}H_{18}O_2$: C, 74.19; H, 9.34. Found: C, 74.17; H, 9.14.

**Phthalans 3** were prepared from **2** (0.28 mol) in boiling formic acid[2] (90%, 100 ml) for 2.5 hr; **3b** was prepared by reaction of **2b** (0.05 mol) with boron trifluoride etherate (0.05 mol) in benzene (150 ml) for 20 hr at room temperature.

**1-Methyl-1-phenylphthalan (3c):** 88% yield; bp 93–95° (0.2 mm) [lit.[7] bp 122–123° (2 mm)]; analytical sample purified by gc[10] (150°); pmr δ 7.83–7.00 (m, aromatic H, 9), 5.13 (s, $C_6H_5CH_2O$, 2.0), 1.83 (s, $CH_3$, 2.9).

*Anal.* Calcd for $C_{15}H_{14}O$: C, 85.68; H, 6.71. Found: C, 85.91; H, 6.57.

**1-Ethyl-1-phenylphthalan (3d):** 66% yield, bp 90–92° (0.12 mm); analytical sample purified by gc[10] (160°); pmr δ 7.83–7.00 (m, aromatic H, 9), 5.13 (s, $C_6H_5CH_2O$, 2.0), 1.83 (s, $CH_3$, 2.9).

*Anal.* Calcd for $C_{16}H_{16}O$: C, 85.68; H, 6.71. Found: C, 85.91; H, 6.57.

There was also obtained a higher boiling fraction [bp 103–115° (0.12 mm), ~⅓ weight of **3d**] that appeared to be (pmr) a mixture of **3d** and 1-(2-formyloxymethylphenyl)-1-phenylprop-1-ene; however, this product was not examined further.

**1-Ethyl-1-methylphthalan (3b):** 73% yield; bp 49–50° (1.4 mm); analytical sample purified by gc[10] (120°); pmr δ 7.31–6.93 (m, aromatic H, 3.9), 5.04 (s, $C_6H_5CH_2O$, 1.9), 1.78 (q, $CH_2CH_3$, 2.0), 1.44 (s, $CH_3$, 3), 0.78 (t, $CH_2CH_3$, 3).

*Anal.* Calcd for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found: C, 81.24; H, 8.49.

**1,1-Diethylphthalan (3a):** 60% yield; bp 60–63° (1.4 mm); analytical sample prepared by gc[10] (135°); pmr δ 7.50–6.83 (m, aromatic H, 4), 5.08 (s, $C_6H_5CH_2O$, 1.9), 1.80 (q, $CH_2CH_3$, 4), 0.73 (t, $CH_2CH_3$, 5.9).

*Anal.* Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.93; H, 8.97.

Distillation of the pot residue gave (1) a mixture (pmr) of **3a** and indene **4a** [~$\frac{1}{10}$ weight of **6a**, bp 63–70° (1.4 mm)] and (2)

3-ethyl-2-methylindene [**4a**, 8.4% yield, bp 70–73° (1.4 mm)].

**Monoacetates 6a and 6b** were prepared from the corresponding diols **2** in ether by using acetyl chloride (2 equiv) and *N,N*-dimethylaniline to trap hydrogen chloride.[11]

**2-(2-Acetoxymethylphenyl)butan-2-ol (6b):** 88% yield; bp 90–93° (0.11 mm); analytical sample purified by liquid chromatography;[12] pmr $\delta$ 7.5–7.1 (m, aromatic H, 4.0), 5.54 (s, $C_6H_5CH_2O$, 2.0), 2.46 (broad s, OH, 1.0), 2.09 (s, $O_2CCH_3$, 2.9), 1.92 (q, $CH_2CH_3$), 1.62 (s, $CH_3$, total weight at 1.92 and 1.62 = 5), and 0.83 (t, $CH_2CH_3$, 3.0); ir $\nu_{OH}$ 3500, $\nu_{C=O}$ 1745 cm$^{-1}$.

*Anal.* Calcd for $C_{13}H_{18}O_3$: C, 70.24; H, 8.16. Found: C, 70.23; H, 7.96.

**3-(2-Acetoxymethylphenyl)pentan-3-ol (6a):** 90% yield; bp 97–100° (0.07 mm); analytical sample prepared by liquid chromatography;[12] pmr $\delta$ 7.6–7.2 (m, aromatic H, 3.8), 5.50 (s, $C_6H_5CH_2O$, 2.0), 2.52 (broad s, OH, 1.0), 2.08 (s, $O_2CCH_3$) and 1.92 (q, $CH_2CH_3$, combined weight = 7), and 0.79 (t, $CH_2CH_3$, 5.9); ir $\nu_{OH}$ 3500, $\nu_{C=O}$ 1740 cm$^{-1}$.

*Anal.* Calcd for $C_{14}H_{20}O_3$: C, 71.16; H, 8.53. Found: C, 71.33; H, 8.43.

**1-(2-Acetoxymethylphenyl)-1-phenylethanol (6c):** 92% yield; mp 86–87° from petroleum ether;[13] pmr $\delta$ 7.9–7.0 (aromatic H, 9), 4.85 (AB quartet, $C_6H_5CH_2O$, 1.8), 3.35 (s, OH, 1.2), and 1.90 (s, $CH_3$ and $-O_2CCH_3$, 6.3); ir $\nu_{OH}$ 3500, $\nu_{C=O}$ 1740 cm$^{-1}$.

*Anal.* Calcd for $C_{17}H_{18}O_3$: C, 75.53; H, 6.71. Found: C, 75.38; H, 6.59.

**$\alpha$-(2-Acetoxymethylphenyl)styrene (8c)** was prepared from **2c** with acetyl chloride (solvent) as described for **6c**. Excess acetyl chloride was removed by distillation prior to extraction and removal of salt and excess *N,N*-dimethylaniline. The crude product was distilled to give essentially pure **8c:** 91% yield; bp 113–118° (0.08 mm); analytical sample prepared by gc[10] (160°); pmr $\delta$ 7.3–7.05 (aromatic H, 9), 5.85 (d, =CH–, 1), 5.27 (d, =CH–, 1) 4.98 (s, $C_6H_5CH_2O$, 1.9), and 1.88 (s, $O_2CCH_3$, 3.0); ir $\nu_{=CH_2}$ 915, $\nu_{C=O}$ 1740 cm$^{-1}$.

*Anal.* Calcd for $C_{17}H_{16}O_2$: C, 80.92; H, 6.39. Found: C, 80.67; H, 6.24.

**2,3-Dimethylindene (4b).** A solution of monoacetate **6b** (1.00 g, 4.49 mmol) in acetic anhydride was stirred at 120° for 4 hr. Formic acid (10 ml, 90%) was added cautiously and the resulting mixture was heated at the reflux temperature for an additional 4 hr. The cooled mixture was added to water (70 ml) and the resulting mixture was stirred for 1.5 hr. The mixture was extracted with petroleum ether[12] (4 × 25 ml) and the combined extracts were washed with saturated aqueous sodium bicarbonate (25 ml) and then dried ($MgSO_4$) and concentrated to give 0.75 g of crude indene (the nmr spectrum showed **4b**, a small amount of phthalan **3b**, and no **4e**). Indene **4b** was purified by column chromatography (Alcoa F-20, 60 g, eluted with petroleum ether);[13] concentration of the initial fractions gave pure (by nmr) indene **4b** (0.38 g, 59% yield). The analytical sample of **4b** was obtained by preparative gas chromatography[10] (130°); pmr $\delta$ 7.57–6.96 (m, aromatic H, 4.1), 3.15 (s, $C_6H_5CH_2$, 2.0), and 2.01 (s, $CH_3$, 5.9).

*Anal.* Calcd for $C_{11}H_{12}$: C, 91.61; H, 8.39. Found: C, 91.58; H, 8.37.

**3-Ethyl-2-methylindene (4a)** was prepared from **6a** as described above for **4b:** 60% yield; pmr $\delta$ 7.77–6.88 (m, aromatic H, 4.2), 3.12 (s, $C_6H_5CH_2$, 2.0), 2.46 (q, $CH_2CH_3$, 2), 1.96 (s, $CH_3$, 2.9), and 1.11 (t, $CH_2CH_3$, 2.9).

*Anal.* Calcd for $C_{12}H_{14}$: C, 91.08; H, 8.92. Found: C, 91.14; H, 9.07.

**3-Phenylindene (4c).** A solution of 1-methyl-1-phenylphthalan (**3c**, 1.00 g, 4.7 mmol) in carbon tetrachloride (15 ml) was added slowly to a cold (0°), stirred mixture of concentrated sulfuric acid (3.68 g) in carbon tetrachloride (10 ml) and the resulting mixture was stirred at 0° for 45 min. A solution of sodium hydroxide (2.95 g) in water (10 ml) was added to neutralize the acid and after 30 min of stirring the organic layer was separated and washed with saturated aqueous ammonium chloride. The crude product (0.95 g) obtained from the dried ($Na_2SO_4$) carbon tetrachloride was 3-phenylindene contaminated with a small amount of unchanged **3c**. The indene was purified by column chromatography (Alcoa F-20, 50 g, petroleum ether[12] as eluent). Concentration of early fractions gave pure 3-phenylindene (**4c**) 0.52 g, 57% yield): pmr $\delta$ 7.6–7.0 (m, aromatic H, 9), 6.48 (t, =$CHCH_2$, 1), and 3.12 (d, $C_6H_5CH_2$, 2).

*Anal.* Calcd for $C_{15}H_{12}$: C, 93.71; H, 6.29. Found: C, 93.59; H, 6.45.

When diol **2c** was used instead of phthalan **3c**, the yield of **4c** was 59%.

**2-Methyl-3-phenylindene (4d).** A mixture of 1-ethyl-1-phenylphthalan (**3d**, 3.50 g, 0.156 mmol) and polyphosphoric acid (5.3 g) was stirred at room temperature for 10 min and was then maintained at 60° for 45 min. The cooled mixture was stirred with water (150 ml) and extracted with chloroform (4 × 35 ml). The combined extract was washed with saturated aqueous sodium bicarbonate (50 ml), dried ($Na_2SO_4$), and concentrated to give 3.55 g of a yellow oil. Distillation of the oil gave **4d** (2.65 g, 82% yield); bp 101–103° (0.12 mm); mp 56–56.5° from acetone (lit.[14] mp 57.5°); pmr $\delta$ 7.63–7.05 (m, aromatic H, 9.2), 3.35 (s, $C_6H_5CH_2$, 1.8), and 2.05 (s, $CH_3$, 2.9).

*Anal.* Calcd for $C_{16}H_{14}$: C, 93.16; H, 6.84. Found: C, 93.04; H, 7.00.

**Registry No.**—**2a**, 51293-49-3; **2b**, 51293-50-6; **2c**, 25770-18-7; **2d**, 51293-51-7; **3a**, 51293-52-8; **3b**, 42502-57-8; **3c**, 51293-53-9; **3d**, 51293-54-0; **4a**, 51293-55-1; **4b**, 4773-82-4; **4c**, 1961-97-3; **4d**, 35099-60-6; **6a**, 51364-44-4; **6b**, 51293-56-2; **6c**, 51293-57-3; **8c**, 51293-58-4.

## References and Notes

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