neutralization of the acid solution and crystallized from ethanol. Yellow plates of 3-amino-4'-acetobiphenyl separated, m.p. 154–159° (uncor.). Repeated crystallizations gave a m.p. of 147.4–150.5°.

Anal. Calcd. for $C_{14}H_{13}ON$: C, 79.59; H, 6.20. Found: C, 79.63; H, 6.50.

Preparation of 3-Bromo-4'-acetobiphenyl.—The procedure followed was that for the preparation of *o*-chlorobromobenzene from *o*-chloroaniline.[41] White needles formed from 30–40° petroleum ether, m.p. 39–40°. Mixed with 3-bromo-4'-acetobiphenyl (m.p. 40–41.2°) prepared from acetyl chloride and 3-bromobiphenyl, it melted at 39–40.9°.

Deamination of 3-Amino-4'-acetobiphenyl.—The procedure was the same as that used to deaminate 2-amino-5-bromobiphenyl.[37] The crude product was crystallized from alcohol and then 60–70° ligroin; white needles separated, m.p. 120–121°. A mixed melting point with *p*-phenylacetophenone (crystallized from ligroin, m.p. 121.0–121.6°) melted at 120.5–121.5°.

Measurements.—A quarter-millimole sample of acid (a tenth-millimole sample in the cases of the nitrobiphenylcarboxylic acids) was weighed into a 250-ml. beaker and dissolved in 50 ml. of purified butyl cellosolve,[42] b.p. 166.8–167.2° (uncor.) at 750 mm. Fifty milliliters of a 0.1 *M* aqueous solution of lithium chloride (anhydrous Merck and Co., Inc., Reagent) was then stirred into the cellosolve so that a 50% (by volume) aqueous butyl cellosolve solution (μ = 0.05) was obtained. The lithium chloride was added to the acid solutions to increase the dielectric constant of the medium as well as to decrease any error arising from small changes in ionic strength.[43]

The 0.05 *N* carbonate-free sodium hydroxide used in the titrations was made up in the following way. One hundred milliliters of standard, approximately 0.1 *N* carbonate-free sodium hydroxide was run as quickly as possible into a brown bottle containing 100 ml. of carbonate-free butyl cellosolve. The usual siphon connections were then made to a 5-ml. buret, graduated in 0.01 ml., and the solution was well mixed.[44] The exact normality of the solution did not need to be known since the half-neutralization point of all titrations was determined from the end-point of the titration, not from the amount of acid weighed out.

The apparatus used in the potentiometric titrations consisted of a Leeds and Northrup glass electrode, calomel electrode, Type K potentiometer, 2420-c galvanometer and a Westinghouse thermionic amplifier. The sensitivity of the galvanometer (0.001 volt) limited *p*H readings to ±0.01 unit. The asymmetry correction of the glass electrode was determined with a biphthalate buffer (*p*H 4.008), an acetate buffer (*p*H 4.648) and a phosphate buffer (*p*H 6.857).[45] A manually operated thermostat maintained the temperature of the acid solution at 25 ± 0.5°.

E.m.f. measurements were made at various intervals during a titration, but in the half-neutralization range e.m.f. determinations were made after each 0.1 ml. of base added, while near the equivalence point measurements were taken after each 0.05 ml. of base added. For acids weaker than benzoic acid a stream of nitrogen was blown over the surface of the solutions throughout the final part of the titration. The end-point was reached when ΔE for each 0.05 ml. of base passed through a maximum. The *p*H at half-neutralization was then obtained graphically. The relationship between e.m.f. measurements and *p*H values found to hold for aqueous solutions was assumed to hold for 50% (by volume) aqueous butyl cellosolve solutions.

The *pK* values reported are the *p*H measurements at half-neutralization,[46] and, therefore, are of significance only in a relative sense. Representative results for two acids follow: 4-nitrobiphenyl-4'-carboxylic acid, *pK* = 5.25, 5.24, 5.26, 5.26; 4-methylbiphenyl-4'-carboxylic acid, 5.69, 5.69, 5.70.

Acknowledgment.—We gratefully acknowledge the assistance afforded by a Frederick Gardner Cottrell Grant of the Research Corporation.

(41) J. L. Hartwell, *Org. Syntheses*, **24**, 22 (1944).

(42) See the purification of butyl "carbitol," J. O. Halford, THIS JOURNAL, **53**, 2944 (1931).

(43) L. A. Wooten and L. P. Hammett, *ibid.*, **57**, 2289 (1935).

(44) This solution was assumed to deteriorate after ten days.

(45) D. I. Hitchcock and A. C. Taylor, THIS JOURNAL, **59**, 1812 (1937).

(46) Unit activity coefficients and equality of *p*H with logarithm of reciprocal of hydrogen ion concentration were assumed.

BRYN MAWR, PENNA. RECEIVED SEPTEMBER 26, 1950

[CONTRIBUTION FROM THE KEDZIE CHEMICAL LABORATORY, MICHIGAN STATE COLLEGE]

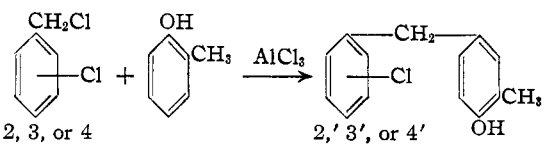
Chloro Substituted Diphenylmethanes, Phenyl Benzyl Ethers and Benzophenones Prepared from Ortho- or Para-cresol[1,2]

BY RALPH C. HUSTON AND KENNETH R. ROBINSON[3]

A series of halogenated phenolic derivatives of diphenylmethane have been prepared in this Laboratory, many of which show interesting bactericidal properties. In order to extend the series, the three monochlorobenzyl chlorides were condensed with *o*- and *p*-cresol. Eight of the nine resulting diphenylmethane derivatives were also prepared by reduction of the corresponding benzophenones which had in turn been prepared from the six possible methylphenyl chlorobenzoates obtainable from *o*- and *p*-cresol. Reduction of 2'-chloro-2-hydroxy-5-methylbenzophenone gave 2-methylxanthene. Six new methylphenyl chlorobenzyl ethers were obtained as by-products.

It has been recently been shown that a series of substituted diphenylmethanes prepared in this Laboratory[4] possess promising specific bactericidal activity. The present investigation was undertaken in order to extend this series to include nine additional chloro substituted diphenylmethanes. The nine corresponding substituted benzophenones and six substituted phenyl benzyl ethers were also prepared.

Two methods of direct alkylation of *o*- and *p*-cresol were used. When the condensation was carried out using an acidic catalyst ($AlCl_3$) *p*-alkylation of *o*-cresol took place.[5]



When the direct alkylation was carried out using basic conditions, in a non-dissociating solvent, carbon alkylation took place exclusively in the ortho position to the hydroxyl group of the phenol. In addition to the carbon alkylation some oxygen alkylation also occurred.

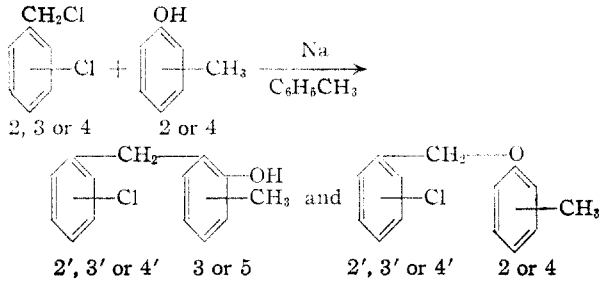
(1) From a thesis submitted by Kenneth R. Robinson to the Graduate School of Michigan State College in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

(2) Presented before the Division of Organic Chemistry at the 118th Meeting of the American Chemical Society, Chicago, Ill., September 3–9, 1950.

(3) Film Division, E. I du Pont de Nemours and Co., Inc., Buffalo, N. Y.

(4) R. C. Huston, *et al.*, THIS JOURNAL, **55**, 2146, 3639 (1933).

(5) R. C. Huston, *ibid.*, **46**, 2775 (1924).

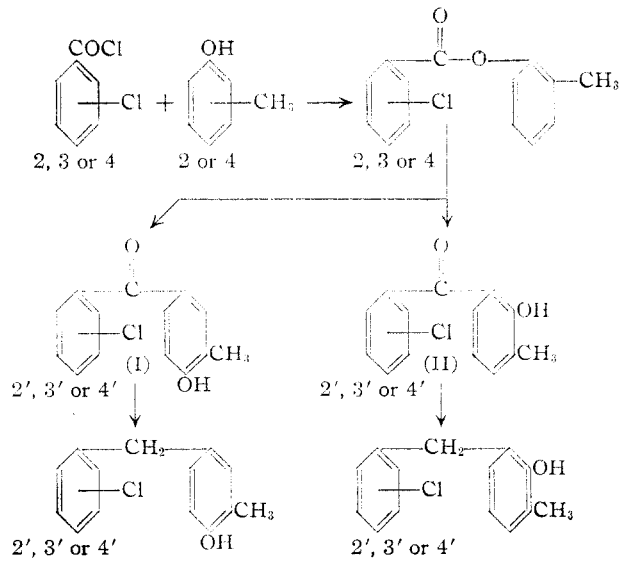


Toluene was first used as the solvent for these basic condensations. However, in many cases considerable amounts of the starting materials were recovered and the higher boiling xylene was employed as suggested by Wheatley.[6] With this solvent only small amounts of starting material were recovered and the yields of the diphenylmethanes and ethers were increased.

The ethers were also prepared in a sodium hydroxide–ethyl alcohol medium using the method described by Lyman and Reid.[7]

The substituted diphenylmethanes were also prepared by a three-step synthesis. In the first step the *o*-, *m*- or *p*-chlorobenzoyl chloride was esterified with *o*- or *p*-cresol in the usual manner. The esters were then allowed to undergo the Fries rearrangement.[8] As was expected the rearrangement of the *o*-cresyl esters gave predominately the *p*-hydroxybenzophenones (I). It was possible to separate the two isomers by taking advantage of the greater solubility of (II) in common solvents. The final step in this series of reactions was the reduction of the substituted benzophenones to the corresponding substituted diphenylmethanes. This was accomplished by means of the Huang-Minlon modification[9] of the Wolff–Kishner reaction.

The entire series of reactions is



The Wolff–Kishner reduction of the 3'-chloro- and 4'-chlorobenzophenones gave the corresponding 3'-chloro- and 4'-chlorodiphenylmethanes in quite satisfactory yields. However, the reductions of the 2'-chlorobenzophenones were less satisfactory. The reduction of 4-hydroxy-3-methyl-2'-chlorobenzophenone and 2-hydroxy-3-methyl-2'-chlorobenzophenone was accomplished although the yields were poor. In the case of 2-hydroxy-5-methyl-2'-chlorobenzophenone only 2-methylxanthene was isolated.

The reaction mixture obtained from the first 2'-chlorobenzophenones mentioned above gave a goodly amount of higher boiling material and it may be that they contained some 2',4-dihydroxy-3-methyldiphenylmethane and 4-methylxanthene, respectively.

The reduction and ring closure of 2-hydroxy-2'-chlorobenzophenones accompanied by loss of hydrogen chloride, either directly or through the intermediate formation of dihydroxy derivatives, is being further studied.

The 2-methylxanthene was also prepared by the catalytic hydrogenation of the known 2-methylxanthone.[10]

Experimental

***m*-Chlorobenzyl chloride** was prepared by the peroxide catalyzed side-chain chlorination of *m*-chlorotoluene using sulfuryl chloride with carbon tetrachloride as the solvent.[11] The yield of material boiling from 93–95° (14 mm.) was 76–79%.

The acidic condensations were carried out by adding 163 g. (1.5 moles) of *o*-cresol and 80.5 g. (0.5 mole) of the chlorobenzyl chloride to 400 ml. of petroleum ether (C.P. b.p. range 30 to 75°). To this mixture was added, with stirring, 33.4 g. (0.25 mole) of anhydrous aluminum chloride over a two-hour period. The stirring was then continued at room temperature (25–35°) for a period of 72 hours.

At the end of this time the red, oily mixture (two layers) was poured, with stirring, into a mixture of 500 g. of ice and 300 ml. of concentrated hydrochloric acid. The acidic mixture was extracted with four 100-ml. portions of ethyl ether. Evaporation of the ethyl ether left an oil which was treated with 250 ml. of Claisen alkali[12] and extracted with four 100-ml. portions of petroleum ether to remove any ethers that may have been formed. There was always a small amount of oil left upon evaporation of the extraction solvent but never enough to purify and identify.

The alkaline fraction was poured over 500 g. of crushed ice and acidified with dilute hydrochloric acid. Finally, the mixture was extracted with four 100-ml. portions of ethyl ether and the ether evaporated. The resulting oil was distilled at reduced pressure and the fractions containing the phenolic product recrystallized from an appropriate solvent. The products thus obtained are listed in Table I.

In the basic condensations 11.5 g. (0.5 mole) of sodium was placed in 400 ml. of dry toluene or xylene and refluxed with stirring until the sodium had formed small pellets. With continued stirring, 54.7 g.

(6) W. B. Wheatley, *et al.*, *ibid.*, **71**, 64 (1949).
(7) J. A. Lyman and E. E. Reid, *ibid.*, **42**, 615 (1920).
(8) R. Adams, "Organic Reactions," Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1942, pp. 342 ff.
(9) Huang-Minlon, THIS JOURNAL, **68**, 2487 (1946).
(10) J. Meisenheimer, R. Hansen and A. Wachterowitz, *J. prakt. Chem.*, [2] **119**, 315 (1928).
(11) M. S. Kharash and H. C. Brown, THIS JOURNAL, **61**, 2142 (1939).
(12) L. Claisen, *Ann.*, **418**, 96 (1919).

TABLE I

DIPHENYLMETHANES

Compound, chlorodiphenylmethane	Reduction, yield, %	Condensation, yield, %	B.p., °C.	Mm.	M.p., °C.	Crystalline form, needles	Recrystallization solvent	Cl,[a] % Calcd.	Found
4-Hydroxy-3-methyl-2'-	[b]	26.8	142–145	0.8	57.8–58.5	Long	Pet. ether	15.24	15.27
4-Hydroxy-3-methyl-3'-	41.0	12.4	143–146	.7	39.9–40.4	Fine	Hexane	15.24	15.28
4-Hydroxy-3-methyl-4'-	44.9	35.3	145–147	.9	74.1–74.8	Fine	Hexane	15.24	15.15
2-Hydroxy-3-methyl-2'-	[b]	9.5[t]	131–133	.7	42.2–42.8	Short	Pet. ether	15.24	15.26
		29.9[x]							
2-Hydroxy-3-methyl-3'-	44.5[c]	19.1[x]	133–136	.7	43.7–44.5	Long	Pet. ether	15.24	15.12
2-Hydroxy-3-methyl-4'-[e]	60.0[c]	33.2[t]	135–137	.7	47.2–47.9	Long	Pet. ether	15.24	15.24
2-Hydroxy-5-methyl-2'-	[d]	30.3[t]	138–140	.7	42.6–43.4	Needles	Pet. ether	15.24	15.12
2-Hydroxy-5-methyl-3'-	78.8	29.8[x]	139–141	.7	53.2–54.0	Short	Pet. ether	15.24	15.13
2-Hydroxy-5-methyl-4'-	42.1	36.9[t]	140–142	.8	51.4–52.1	Needles	Pet. ether	15.24	15.17

[a] All chlorine analyses were made by the sodium peroxide fusion method.[13] [b] Yield was small and not calculated. [c] Yield is based on only one reaction. [d] Only 2-methylxanthene was isolated. [t] Toluene was used as the solvent in this condensation. [x] Xylene was used as the solvent in this condensation. [e] Previously prepared by Klarmann, *et al.*,[14] m.p. 48°.

TABLE II

METHYLPHENYL CHLOROBENZYL ETHERS

Ether	Condensation, yield, %	NaOH–EtOH yield,[a] %	B.p., °C.	Mm.	M.p., °C.	Crystalline form	Recrystallization solvent	Cl, % Calcd.	Found
2-Methylphenyl 2-chlorobenzyl	3.1[t]	71.5	118–119	0.8	37.9–38.8	Granular	Methyl alcohol	15.24	15.06
	14.9[x]								
2-Methylphenyl 3-chlorobenzyl	21.0[x]	87.6	125–127	1.0	[b]	.....	.....	15.24	15.22
2-Methylphenyl 4-chlorobenzyl	12.5[t]	84.3	125–126	0.8	61.2–61.7	Flat needles	Pet. ether	15.24	15.24
4-Methylphenyl 2-chlorobenzyl	8.6[t]	76.8	120–123	0.8	[c]	.....	.....	15.24	15.17
4-Methylphenyl 3-chlorobenzyl	10.8[x]	67.8	128–129	0.9	53.8–54.6	Plates	Pet. ether	15.24	15.01
4-Methylphenyl 4-chlorobenzyl	4.9[t]	88.0	127–130	0.9	91.7–92.6	Granular	Hexane	15.24	15.08

[a] These yields are based on only one reaction. [t] Toluene was used as the solvent in this condensation. [x] Xylene was used as the solvent in this condensation. [b] $n^{20}D$ 1.5791. [c] $n^{20}D$ 1.5791.

(0.5 mole) of *o*- or *p*-cresol, dissolved in 100 ml. of the solvent, was slowly added and this mixture allowed to stir and reflux for one-half hour. At this point 80.5 g. (0.5 mole) of the chlorobenzyl chloride was added over a period of one-half hour. Refluxing and stirring of this mixture were then continued for 36 hours.

After cooling, the reaction mixture was acidified with dilute hydrochloric acid and ice. The organic layer was separated and the aqueous layer extracted with two 100-ml. portions of toluene or xylene. The oil that was left after stripping off the solvent was taken up in 250 ml. of Claisen alkali and the ethers and phenolic products separated. The latter were purified in the manner described previously and are listed in Table I.

The methylphenyl chlorobenzyl ethers extracted from the basic solution were purified by vacuum distillation and finally by crystallization if they could be induced to solidify. These ethers are listed in Table II.

The sodium hydroxide–ethyl alcohol method of preparation of the methylphenyl chlorobenzyl ethers used was essentially the same as that described by Lyman and Reid.[8] Ten grams of NaOH was dissolved in 500 ml. of 95% ethyl alcohol and brought to reflux temperature. To this solution there was added 32.4 g. (0.30 mole) of *o*- or *p*-cresol and then 40.3 g. (0.25 mole) of chlorobenzyl chloride. Stirring and refluxing were continued for three hours. At the end of this time 450 ml. of water was added to the mixture and this then placed in refrigerator. If the oil solidified it was removed and recrystallized from a suitable solvent. If the oil could not be induced to crystallize it was separated from the water–alcohol layer and purified by distillation. The physical constants of these ethers are listed in Table II.

The methylphenyl chlorobenzoates were readily prepared by adding 175 g. (1.0 mole) of chlorobenzoyl chloride to 135 g. (1.25 moles) of *o*- or *p*-cresol and heating to 95° for 2.5–5 hours. After cooling, the mixture was taken up in benzene or ethyl ether and the solution washed successively with 2 *N* NaOH solution and water.

The solvent was then allowed to evaporate and the oil or solid taken up in an appropriate solvent. If the ester was a solid at room temperature it was recrystallized. The physical constants of these esters are listed in Table III.

The Fries rearrangement of the esters was carried out in the same manner as that described by Adams.[8] The rearrangement was accomplished without the aid of a solvent and the aluminum chloride complex was decomposed with ice and concentrated hydrochloric acid. After distilling the resulting solid at reduced pressure (15 mm.) it was purified by recrystallization. In the cases of the *p*-cresyl esters there was always a small amount of the corresponding chlorobenzoic acid present. These acids were readily removed by a sodium bicarbonate wash before recrystallization.

The solvents used to effect a satisfactory separation of the two isomers produced in the rearrangement of the *o*-cresyl esters are given in Table IV. This table also includes the physical constants of the hydroxy-methyl-chloro'-benzophenones.

It is interesting to note that all of the six *o*-hydroxybenzophenones were yellow whereas the three *p*-hydroxybenzophenones were white.

The product obtained when 4-methylphenyl 4-chlorobenzoate was allowed to undergo the Fries rearrangement was yellow and had a rather sharp melting point. However, the amount of chlorine found was lower than the theoretical despite repeated recrystallizations and distillations. Reduction by the Wolff–Kishner reaction gave 2-hydroxy-5-methyl-4'-chlorodiphenylmethane and therefore it must have contained some of the desired 2-hydroxy-5-methyl-4'-chlorobenzophenone. The results of the chlorine analyses are listed in Table IV.

The reduction of the benzophenones to the corresponding diphenylmethanes was accomplished by the Huang-Minlon modification[10] of the Wolff–Kishner reaction. To 500 ml. of diethylene glycol were added 123.4 g. (0.5 mole) of the hydroxy - methyl - chloro' - benzophenone, 55.0 g. (1.38 moles) of sodium hydroxide and 51.5 g. (0.87 mole) of 85 per cent. hydrazine hydrate. The mixture was stirred and heated to reflux temperature (140°). After 1.5 hours the reflux condenser was removed and the temperature allowed to rise to 195°. The condenser was then replaced and refluxing and stirring continued for 4 hours.

(13) J. F. Lemp and H. J. Broderson, THIS JOURNAL, **39**, 2069 (1917).

(14) E. Klarmann, L. W. Gates and V. A. Shternov, *ibid.*, **54**, 3315 (1932).

TABLE III

METHYLPHENYL CHLOROBENZOATES

Compound, chlorobenzoate	Yield, %	M.p., °C.	Crystalline form	Recrystallization solvent	Cl, % Calcd.	Found
2-Methylphenyl 2-	90.8	138–140[a,b]	.....	.....	14.37	14.17
2-Methylphenyl 3-	80.4	53.2–54.2	Granular	EtOH (95%)	14.37	14.41
2-Methylphenyl 4-	86.3	44.7–45.4	Granular	EtOH (95%)	14.37	14.37
4-Methylphenyl 2-[c]	79.0	68.8–69.8	Flat needles	MeOH	14.37	14.49
4-Methylphenyl 3-	90.2	75.1–76.0	Needles	MeOH	14.37	14.37
4-Methylphenyl 4-	90.8	97.4–98.3	Large	MeOH or ligroin (90–120°)	14.37	14.34

[a] Boiling point, at 10 mm. [b] $n^{20}D$ 1.5787. [c] Previously reported by Auwers,[15] m.p. 68–71°.

TABLE IV

BENZOPHENONES

Compound, chlorobenzophenone	Yield, %	M.p., °C.	Crystalline form	Recrystallization solvent	Cl, % Calcd.	Found
4-Hydroxy-3-methyl-2'-[a]	35.3	167.9–168.6	White granular	EtOH	14.37	14.43
4-Hydroxy-3-methyl-3'-[b]	55.7	151.2–151.8	White powder	Toluene	14.37	14.31
4-Hydroxy-3-methyl-4'-[c]	52.1	210.5–211.5	White needles	EtOH (95%)	14.37	14.33
2-Hydroxy-3-methyl-2'-[a]	11.8	72.3–72.8	Yellow rhombic	MeOH	14.37	14.38
2-Hydroxy-3-methyl-3'-[b]	17.3	69.5–70.3	Yellow prisms	MeOH	14.37	14.23
2-Hydroxy-3-methyl-4'-[c]	12.6	61.5–62.0	Yellow needles	MeOH	14.37	14.29
2-Hydroxy-5-methyl-2'-[d]	71.3	76.3–77.2	Yellow rhombic	Pet. ether	14.37	14.26
2-Hydroxy-5-methyl-3'-	45.1	70.5–71.5	Yellow granular	Pet. ether	14.37	14.31
2-Hydroxy-5-methyl-4'-	50.9	66.9–67.6	Yellow prisms	Pet. ether or EtOH (95%)	14.37	13.48[e]

[a] Two isomers separated by the use of benzene. [b] Two isomers separated by the use of toluene. [c] Two isomers separated by the use of ethyl ether. [d] Previously reported by Rosenmund and Schurr,[16] m.p. 78°. [e] See Experimental.

TABLE V

p-CHLOROBENZOATE DERIVATIVES

Compound, chlorodiphenylmethane	M.p., °C.	Crystal structure	Cl, % Calcd.	Found	
4-Hydroxy-3-methyl-2'-	77.1–77.9	Rhombic	19.10	18.90	19.04
4-Hydroxy-3-methyl-3'-	62.2–62.9	Very fine ndls.	19.10	18.90	19.09
4-Hydroxy-3-methyl-4'-	50.9–51.6	Needles	19.10	18.90	18.91
2-Hydroxy-3-methyl-2'-	55.5–56.5	Rhombic	19.10	18.84	18.91
2-Hydroxy-3-methyl-3'-	85.5–86.5	Small plates	19.10	19.11	18.91
2-Hydroxy-3-methyl-4'-	81.6–82.3	Needles	19.10	19.08	19.18
2-Hydroxy-5-methyl-2'-	73.2–74.1	Granular	19.10	18.93	18.96
2-Hydroxy-5-methyl-3'-	84.0–85.0	Needles	19.10	18.95	18.93
4-Hydroxy-5-methyl-4'-	78.5–79.1	Needles	19.10	18.99	19.02

After cooling, the viscous mixture was added to 750 ml. of water and acidified with dilute hydrochloric acid. The resulting oil was removed by extracting with three 250-ml. portions of ethyl ether. After evaporating the ether on a steam-bath the oil was distilled at reduced pressure (2–3 mm.) and the distillate recrystallized from an appropriate solvent.

This method of reduction was successfully employed for eight of the nine diphenylmethanes and the products obtained were shown by mixed melting points to be the same as those obtained by the direct methods. The results of these reductions are listed in Table I.

2-Methylxanthene was the only compound isolated from the reduction mixture of 2-hydroxy-5-methyl-2'-chlorobenzophenone. The solid distillate was recrystallized from methyl alcohol; yield 35–40%.

A second method of preparation of this xanthene involved preparing the known 2-methylxanthone[10] by refluxing a mixture of 100 ml. of diethyleneglycol, 24.68 g. (0.1 mole) of 2-hydroxy-5-methyl-2'-chlorobenzophenone and 11.0 g. (0.28 mole) of sodium hydroxide for 5.5 hours. The product was isolated in the same manner as described for the Wolff–Kishner reductions above. After recrystallizing from methyl alcohol the product had a m.p.[10] of 123–125°; yield 78.0%.

The reduction of 2-methylxanthone to 2-methylxanthene was accomplished by catalytic hydrogenation. The procedure was similar to that used by Ipatieff[17] for reducing xanthone itself to xanthene. Five grams of 2-methylxanthone was placed in a rocking bomb along with 50 ml. of absolute ethyl alcohol. To this was added 0.5 g. of a copper–chromium oxide catalyst prepared in the manner described by Adkins.[18] The initial pressure of hydrogen was 80 atmospheres. After heating the bomb to 185° it was rocked for two hours. Upon cooling, the contents of the bomb were taken up in acetone and filtered. Evaporation of the solvents left a solid which was recrystallized from methyl alcohol, m.p. 96.4–97.4°. The compound thus prepared was identical with that prepared by the Wolff–Kishner reduction of 2-hydroxy-5-methyl-2'-chlorobenzophenone. *Anal.* Calcd. for $C_{14}H_{12}O$: C, 85.68; H, 6.16. Found: C, 85.79; H, 6.27.

EAST LANSING, MICH. RECEIVED DECEMBER 4, 1950

(15) K. V. Auwers and W. Mauss, *Ann.*, **464**, 293 (1928).

(16) K. W. Rosenmund and W. Schnurr, *Ann.*, **460**, 56 (1928).

(17) V. Ipatieff and N. Orlov, *Compt. rend.*, **183**, 973 (1926).

(18) H. Adkins, "Reactions of Hydrogen," The University of Wisconsin Press, Madison, Wisconsin, 1937, pp. 13–14.