occurring 2-pyrones including yangonin, 4-methoxyparacotoin, and anibine.[8] These synthetic methods will provide a basis for study of the biosynthesis and metabolism of such compounds, since diketo acids, the 2-pyrones and other derivatives, labeled with C^{14} at the carboxyl position, can be readily and inexpensively prepared from carbon-14 labeled carbon dioxide.

Experimental Section[16]

Starting β-Diketones.—Benzoylacetone (**1a**), 2,4-pentanedione (**1b**), and ethyl acetoacetate (**1h**) were obtained from commercial sources. 6,6-Diphenyl-5-hexene-2,4-dione (**1f**) was prepared by the condensation of benzophenone and dipotassio-2,4-pentanedione.[17] Sodioacetoacetaldehyde was prepared by a published procedure.[6] 7-Methyl-2,4-heptanedione (**1d**) and 2-acetyl-1-naphthol (**4**) were gifts of Professor D. E. Pearson. 6-Phenyl-5-hexene-2,4-dione (**1e**) was prepared in low yield by acylation of acetone with cinnamoyl chloride as described by Linn and Hauser.[18]

Preparation of 5-Phenyl-3,5-pentanedionoic Acid (2a) (Table I).—Several methods were investigated for preparation of **2a**. The preferred procedure involved preparation of a suspension of 0.087 mole of sodium amide[19] (from 2.0 g of sodium) in 300 ml of commercial anhydrous liquid ammonia in a 1-l., three-neck flask equipped with a condenser and glass stirrer. Benzoylacetone (4.05 g, 0.025 mole) was added through a powder funnel and the reaction mixture was stirred 30 min. The ammonia was rapidly evaporated on a steam bath as an equal volume of anhydrous ether was added. The ether was allowed to reflux for several minutes to ensure removal of most of the ammonia. Lumps of Dry Ice (about 200 g) were added and the reaction mixture stirred for 45 min. The ethereal suspension was poured into a mixture of ice and 40 ml of 12 *N* hydrochloric acid. The layers were separated and the ether layer was extracted several times with a cold, 5% solution of sodium bicarbonate. The alkaline extract was immediately acidified with hydrochloric acid. The white precipitate was collected and air dried at room temperature to yield 3.6 g (74%) of diketo acid **2a**, mp 93–94.5° (lit[20] mp 94°).

In order to obtain good yields of pure product, it was essential that the bicarbonate solution of diketo acid be rapidly acidified so as to minimize spontaneous decarboxylation. Diketo acid **2a** and the other acids prepared in this study (with the exception of **5**, which was quite stable) were handled and stored at room temperature or below to avoid decarboxylation.

No carboxylation was obtained with sodium hydride in tetrahydrofuran as the base or with methyl magnesium carbonate[21] in toluene as the combined base and carboxylating agent.

Preparation of Dicarbonyl Acids 2b–h and 5 (Table II).—Dicarbonyl acids **2b–h** and **5** were prepared by procedures similar to the above preparation of **2a** employing sodium amide, except for the variations noted below. Acid **1f** was prepared from ethyl dipotassioacetoacetate.

In the case of diketo acid **2b**, the bicarbonate extraction was omitted. The initial ether extract was dried over magnesium sulfate and the solvent was evaporated. Unreacted 2,4-pentanedione was removed under reduced pressure (0.02 mm) at room temperature.

All products were essentially pure as obtained by the above procedures. Acids **2b**, **e**, and **h** were recrystallized from ether–hexane, **2c** from ether–benzene–hexane, and **2f** from benzene–hexane. The compounds were placed in the solvent at room temperature and cooled to −20° to effect crystallization. Suitable conditions could not be found for crystallization of **2d**. The naphthol derivative **5** was somewhat more stable and was recrystallized from hot ethanol.

Preparation of 4-Hydroxy-2-pyrones 3a–f and 9.—Approximately 20 ml of commercial anhydrous liquid hydrogen fluoride was placed in a 50-ml polyethylene bottle containing 1.0 g of diketo acid. The mixture was allowed to stand in the hood for 24–48 hr until the hydrogen fluoride had evaporated. To ensure that all traces of hydrogen fluoride had been removed, the flask was allowed to stand in an evacuated desiccator over sodium hydroxide pellets for several hours. In some cases the residue was also washed with a dilute solution of sodium bicarbonate. The residues of 2-pyrones were usually essentially pure as indicated by melting point and infrared spectra. Analytical samples of **2c** and **2d** were prepared by recrystallization from ether. Ethanol was employed for **2f** and ether–acetic acid for **9**. 2-Pyrone **2e** was analyzed without recrystallization.

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[16] Melting points were determined in open capillaries using a silicone oil bath and are corrected. Infrared spectra were determined with a Beckman IR-10 spectrometer using the potassium bromide pellet method for solids, and the neat liquid between sodium chloride plates for liquids. Nmr spectra were determined with a Varian A-60 spectrometer employing approximately 10% solutions in deuteriochloroform. Elemental analyses were performed by Galbraith Laboratories, Inc., Knoxville, Tenn.

[17] R. J. Light and C. R. Hauser, *J. Org. Chem.*, **26**, 1716 (1961).

[18] B. O. Linn and C. R. Hauser, *J. Am. Chem. Soc.*, **78**, 6066 (1956).

[19] J. T. Adams and C. R. Hauser, *ibid.*, **66**, 1220 (1944); R. Levine, J. A. Conroy, J. T. Adams, and C. R. Hauser, *ibid.*, **67**, 1510 (1945).

[20] K. Balenović and D. Sunko, *Monatsh.*, **79**, 1 (1948).

[21] M. Stiles, *J. Am. Chem. Soc.*, **81**, 2598 (1959); M. Stiles and H. L. Finkbeiner, *ibid.*, **81**, 505 (1959).

Relative Reactivities of Certain Disodio-β-diketones toward Alkyl Halides in Liquid Ammonia[1]

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A study was made of the relative reactivities of disodio salts of β-diketones towards alkyl halides in liquid ammonia. The procedure involved treatment of an equal amount of two different disodio-β-diketones with a limited amount of an alkyl halide. The order of relative reactivities observed was isobutyryl, propionyl > acetyl > phenylacetyl. The relationship of these reactivities to basicity is discussed.

It has previously been observed that not only the mode of alkylation of acetylacetone or benzoylacetone is changed, but also the rate of the reaction is greatly increased on converting the common intermediate monocarbanion **1′** to the dicarbanion **1″** before adding the alkyl halide.[2,3] Thus, alkylation of **1′** to form the 3 derivative is too slow to be practical in liquid ammonia (at −33°), whereas that of **1″** to give the 1-derivative is sufficiently rapid in this medium to furnish a useful method of synthesis.[2,4]

$RCO\bar{C}HCOCH_3$
1′

$RCO\bar{C}HCO\bar{C}H_2$
1″

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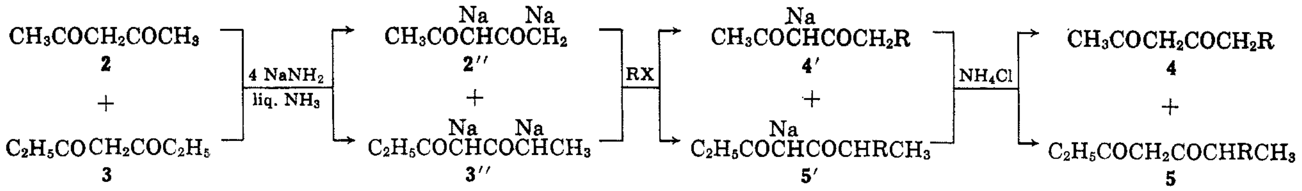
[1] Supported by the National Science Foundation.

[2] C. R. Hauser and T. M. Harris, *J. Am. Chem. Soc.*, **80**, 6360 (1958).

[3] For convenience mono- and dicarbanions are designated prime and double prime, respectively, and only carbanion resonance forms are represented even though other resonance forms may make more important contributions to the structures of the anions.

[4] See (a) K. G. Hampton, T. M. Harris, and C. R. Hauser, *J. Org. Chem.*, **30**, 61 (1965); (b) K. G. Hampton, T. M. Harris, and C. R. Hauser, *ibid.*, **31**, 663 (1966).

SCHEME I



In the present investigation a study was made of the relative reactivities (nucleophilicities)[5] of several types of disodio-β-diketones towards certain alkyl halides in liquid ammonia. These disodio salts appeared to be quite soluble in this medium[6] although the solutions tended to be hazy like a Grignard solution in ether. The general procedure involved addition of an equimolar mixture of two β-diketones to 4 molecular equiv of sodium amide in liquid ammonia, followed by slightly less than 1 molecular equiv of an alkyl halide and, finally, by ammonium chloride. Such competitive reactions may be illustrated with acetylacetone (**2**) and dipropionylmethane (**3**) with which *n*-butyl bromide and four other butyl halides were employed; the intermediate disodio salts were **2''** and **3''**, and the products **4** and **5**, respectively (Scheme I).[3]

Similarly, competitive reactions were effected between disodio salts **2''** and **6''**[7] and between disodio salts **3''** and **7''** with butyl bromide, and between disodio salts **8''** and **9''** with ethyl bromide.[3] The relative extent of formation of the two isomeric γ-alkyl derivatives produced in each case was determined by gas chromatographic comparison of the crude reaction product with authentic samples of the compounds. The authentic samples were prepared by alkylation of disodio salts of appropriate β-diketones. The results are summarized in Table I.

$CH_3CO\overset{Na}{C}HCO\overset{Na}{C}HC_6H_5$

6''

$(CH_3)_2CHCO\overset{Na}{C}HCO\overset{Na}{C}(CH_3)_2$

7''

$C_6H_5CO\overset{Na}{C}HCOCH_2Na$

8''

$C_6H_5CO\overset{Na}{C}HCO\overset{Na}{C}HCH_3$

9''

TABLE I

COMPETITIVE REACTIONS OF DISODIO-β-DIKETONES WITH ALKYL HALIDES IN LIQUID AMMONIA

Expt[a] no.	Disodio salts A	Disodio salts B	RX	% Alkylation of: A	% Alkylation of: B	k_A/k_B
1	2''	3''	*n*-C_4H_9Br	20	80	1/6.4
2[b]	2''	3''	*n*-C_4H_9Br	14	86	1/6.4
3	2''	3''	*n*-C_4H_9Cl	23	77	1/5.1
4	2''	3''	*n*-C_4H_9I	25	75	1/4.3
5	2''	3''	*sec*-C_4H_9Br	27	72	1/3.8
6	2''	3''	*i*-C_4H_9Br	18	82	1/7.5
7	2''	6''	*n*-C_4H_9Br	62	38	1/0.52
8	3''	7''	*n*-C_4H_9Br	*ca.* 50	*ca.* 50	1/1
9	8''	9''	C_2H_5Br	32	68	1/2.7

[a] Ratio of halide to total disodio salts was 0.9:2 except in expt 2. [b] The ratio was 0.1:2.

The difference in the relative amounts of alkylation of A and B (Table I) is not so great as the difference in the relative reactivities since in each of the experiments the more reactive disodio salt was depleted more rapidly; this improved the ability of the less reactive dicarbanions to compete for the alkyl halide during the later phase of the reaction. Notice that in expt 2, where 1/9 as much *n*-butyl bromide was used as in expt 1, the difference of the amount of alkylation of A and B increases. The ratio of the reactivities of A and B (k_A/k_B) has been calculated by eq 1 where $(A)_0$ and $(B)_0$ are the initial disodio salt concentrations of A and B, respectively, and (A) and (B) are the final concentrations of A and B, respectively, assuming complete reaction had occurred with the alkyl halide.

$$k_A/k_B = \frac{\log (A)/(A)_0}{\log (B)/(B)_0} \qquad (1)$$

Experiments 1, 2, 7, and 8 in Table I show that the relative reactivities (nucleophilicities) of dicarbanions **2''**, **3''**, **6''**, and **7''** decrease in the order: isobutyryl (**7**) ≃ propionyl (**3**) > acetyl (**2**) > phenylacetyl (**6**). The extent of reaction in these cases was 80% or more.

Experiments 1 and 3–6 in Table I indicate that this order of reactivities (nucleophilicities) was dependent principally on the structure of the disodio-β-diketones and not appreciably on the structure of the alkyl halide, since the values obtained with the five different butyl halides were only ±5% of a 22:78 mixture of products. However, the values might possibly vary more with more complex halides.

A comparison of expt 1 and 9 in Table I shows that, while the ratios of the products were somewhat different in these two cases in which both the β-diketone and halide were varied, the same order of reactivity of the acetyl and propionyl type dicarbanions was observed. This order would presumably hold for many variations of the β-diketone and halide.

It would be of interest to determine the relative basicities of the four types of dianions employed in this paper, but such data are difficult to obtain experimentally because disodio and monosodio salts of diketones are not readily equilibrated with one another.[2,4] Potassio salts, although rapidly equilibrated, are only slightly soluble in ammonia making them unsuitable for comparisions.[6] However, relative basicities might be estimated from knowledge of both the relative ease of dianion formation by amide ion and the relative reactivity of the dianions with ammonia.

The first of these factors was previously determined for the four types of disodio salts and the observed order was isobutyryl < propionyl < acetyl < phenylacetyl.[4] The second has not yet been determined with ammonia or other proton donors, but the nucleophilicities toward carbon electrophiles, that have now been determined, should approximate this order. One possible exception to this is that disodio salt **7''** of the isobutyryl diketone

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[5] Although the mechanism has not been established, it is likely that it involves an S_N2 type reaction so that relative reactivities may be equated with relative nucleophilicities.

[6] Since dipotassio salts are generally very insoluble in liquid ammonia, reactivity comparisons with them would have reflected both nucleophilicities and solubilities.

[7] Disodio salt **6''** has recently been shown to be formed from 1-phenyl-2,4-pentanedione and 2 molecular equiv of sodium amide in liquid ammonia without apparent contamination by the isomeric 3,5-disodio salt; see ref 4b.

Table II

Preparation of Authentic Alkylation Products

Alkylation products	Yield, %	Bp, °C (mm)	Formula	Calcd, % C	Calcd, % H	Found, % C	Found, % H	Copper chelate Mp, °C	Copper chelate Formula	Copper chelate Calcd, % C	Copper chelate Calcd, % H	Copper chelate Calcd, % Cu	Copper chelate Found, % C	Copper chelate Found, % H	Copper chelate Found, % Cu
2,4-Nonanedione (**4**)	67–75[a]	100–103 (19)						139–140							
6-Methyl-3,5-decanedione (**5**)	73	119–121 (19)	$C_{11}H_{20}O_2$	71.69	10.94	71.88	10.88	67–69	$C_{22}H_{38}O_4Cu$	61.45	8.91	14.78	61.35	9.19	15.01
6-Methyl-2,4-octanedione	78[a]	95–97 (20)						105–106.5							
7-Methyl-2,4-octanedione	78[a]	97–99 (20)						119.5–120.5							
6,7-Dimethyl-3,5-nonanedione	83	117–119 (20)	$C_{11}H_{20}O_2$	71.69	10.94	71.46	10.96	73.5–75	$C_{22}H_{38}O_4Cu$	61.45	8.91	14.78	61.65	8.94	14.97
6,8-Dimethyl-3,5-nonanedione	78	111–114 (20)	$C_{11}H_{20}O_2$	71.69	10.94	71.58	10.92	. . .							
5-Phenyl-2,4-nonanedione	77[b]	125–128 (0.6)						. . .							
2,6,6-Trimethyl-3,5-decanedione	26[c]	143–146 (36)	$C_{13}H_{24}O_2$	73.53	11.39	73.65	11.59	. . .							
1-Phenyl-1,3-hexanedione	85	125–128 (2.5)[d]						134–135[e]							
1-Phenyl-4-methyl-1,3-hexanedione	84	133–135 (3.5)	$C_{13}H_{16}O_2$	76.44	7.90	76.34	7.64	122–123	$C_{26}H_{30}O_4Cu$	66.44	6.44	13.52	66.71	6.53	13.56

[a] See ref 4a. [b] See ref 4b. [c] Diisobutyrylmethane used in this synthesis was not purified by vpc. [d] Lit.[14] bp 166–171° (20 mm). [e] Copper chelate melting point undepressed on admixture with authentic sample (see ref 14).

may be somewhat more hindered toward attack at carbon than at the much smaller hydrogen of ammonia. Thus, in the sequence of reactivity of disodio salts with ammonia the isobutyryl disodio salt should be more reactive than the propionyl one. On the basis of consideration of both proton reactions, the order of basicity of the disodio salt should be isobutyryl > propionyl > acetyl > phenylacetyl.

It has been observed previously that acidity correlations with reactivities can be made among certain closely related compounds on the basis of only the rates of proton abstraction or deuterium exchange and in certain cases on the basis of only nucleophilicity data.[8] Such correlations based on kinetic data obtained from only one-half of the equilibrium reaction must be made very cautiously and with cognizance of the assumptions that are being made.[8]

It should be mentioned that House and Trost recently obtained evidence with an aliphatic monoketone indicating that methylene anions are more stable than methyl anions.[9] Although there is no assurance that their result with monoanions of a monoketone has any bearing on the basicities of dianions of diketones, nonetheless their result does raise some doubt as to the validity of our conclusion that propionyl type dianions (3′′) are less stable than the acetyl type (2′′).

Experimental Section[10]

Starting Materials.—Acetylacetone and benzoylacetone were commercially available. 1-Phenyl-2,4-pentanedione was prepared by phenylation of disodioacetylacetone with diphenyliodonium chloride.[11] Dipropionylmethane was prepared by condensation of ethyl propionate and butanone by means of sodium amide.[12] 1-Phenyl-1,3-pentanedione was prepared by condensation of ethyl propionate and acetophenone by means of sodium amide.[13] Diisobutyrylmethane was prepared by condensation of phenyl isobutyrate and methyl isopropyl ketone by means of lithium amide.[14] Vpc of the product indicated the presence of impurities. The material was purified satisfactorily by preparative gas chromatography.

Preparation of Authentic Samples of Alkylation Products.—Each of the disodio salts (2′′, **3**′′ and 6′′–9′′) that was employed in the reactivity studies was also prepared singly in liquid ammonia and alkylated with the appropriate alkyl bromide. A typical preparation involved the addition of 1 molecular equiv of diketone in 20–30 ml of ether to 2 molecular equiv of sodium amide[15] in 400–600 ml of liquid ammonia. The reactions were carried out with 0.05–0.10 mole of diketone. After 20 min, the alkyl bromide in 30 ml of ether was added dropwise over 10 min. After 30 min, 250 ml of ether was added and the ammonia was evaporated on the steam bath. The ethereal suspension was cooled in ice and acidified with a mixture of ice and concentrated

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[8] See D. J. Cram, "Fundamentals of Carbanion Chemistry," Academic Press Inc., New York, N. Y., 1965, Chapter 1; J. O. Edwards and R. G. Pearson, *J. Am. Chem. Soc.*, **84**, 16 (1962), and references cited in both.

[9] H. O. House and B. M. Trost, *J. Org. Chem.*, **30**, 1341 (1965).

[10] Melting points were taken on a Mel-Temp capillary melting point apparatus and are uncorrected. Analyses are by Dr. Ing. A. Schoeller Mikro-Labor, Kronach, West Germany, and by Galbraith Laboratories, Knoxville, Tenn. Vapor phase chromatograms were obtained on an F and M Model 500 gas chromatograph equipped with thermal conductivity detectors using 2-ft silicone gum rubber, 5-ft Apiezon L, and 5-ft Carbowax 20M columns. An Aerograph Autoprep Model A-700 with a 20-ft Carbowax 20M column was used for the preparative separation of diisobutyrylmethane.

[11] K. G. Hampton, T. M. Harris, and C. R. Hauser, *J. Org. Chem.*, **29**, 3511 (1964).

[12] J. T. Adams and C. R. Hauser, *J. Am. Chem. Soc.*, **66**, 1220 (1944).

[13] R. Levine, J. A. Conroy, J. T. Adams, and C. R. Hauser, *ibid.*, **67**, 1510 (1945).

[14] H. D. Murdock and D. C. Nonhebel, *J. Chem. Soc.*, 2153 (1962).

[15] See C. R. Hauser, F. W. Swamer, and J. T. Adams, *Org. Reactions*, **8**, 122 (1954).

hydrochloric acid. The ether layer was separated and the aqueous layer was extracted three times with ether. The combined ethereal solution was dried with anhydrous magnesium sulfate and filtered. The ether was evaporated and the residue was distilled. The yields and boiling points of products are shown in Table II. Five of the diketones were new compounds; their copper chelates were also prepared. The analyses of the diketones and copper chelates are given in Table II.

Nucleophilicity Comparison between Disodio Salts (Table I).—In expt 1 a solution of 10 g (0.10 mole) of acetylacetone and 12.8 g (0.10 mole) of dipropionylmethane in 30 ml of tetrahydrofuran was added to a stirred suspension of 0.40 mole of sodium amide in 600 ml of liquid ammonia to form a solution[6] of disodio salts **2''** and **3''** (0.1 mole of each). After 30 min, 100 ml of tetrahydrofuran was added; then 12.4 g (0.09 mole) of butyl bromide was added over 10–12 min. After 30 min, 6 g of ammonium chloride was added, and the reaction mixture was worked up as described above. Vpc of the crude product showed peaks for acetylacetone, dipropionylmethane, 2,4-nonanedione, and 6-methyl-3,5-decanedione. The mole ratio of 2,4-nonanedione to 6-methyl-3,5-decanedione was 80:20. The products were identified by comparison of retention times with those of authentic samples. The product ratios were obtained by integration of peak areas. The average was taken of several chromatograms.

Experiments 3–6 and 8 were conducted similarly. In expt 2 only 0.01 mole of butyl bromide was employed. Experiments 7 and 9 were conducted on one-half the scale of expt 1.

Reaction of 5-Chloro-2-hydroxybenzophenone and Phosphorus Pentachloride. Structural Studies[1]

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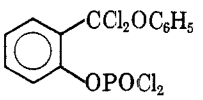
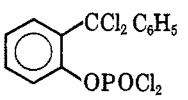
The product from the reaction of 5-chloro-2-hydroxybenzophenone and phosphorus pentachloride has been shown to have the phosphorodichloridate structure VI on the basis of P^{31} nmr and infrared spectra and chemical evidence. 5-Chloro-2-hydroxybenzophenone reacts with phosphorus oxychloride to form the phosphorodichloridate ketone (V). Reaction of V with phosphorus pentachloride converts it into VI, which was obtained from the above reaction, the reaction with V being the normal reaction for conversion of a keto group into a dichloromethylene group. The analogous phosphorodichloridate ester (IV) did not undergo a reaction with phosphorus pentachloride; thus the possibility of an exchange reaction in the phosphorodichloridate group was obviated.

VI reacts with 2 equiv of phenol in the presence of *t*-amine (or sodium phenolate) to form Xa as evidenced by P^{31} nmr, infrared spectra, and basic hydrolysis of the dichloromethylene to a carbonyl group. The dichloromethylene chlorines could not be replaced by phenoxide groups under various conditions attempted. VI was hydrolyzed in homogeneous aqueous dioxane solution to 5-chloro-2-hydroxybenzophenone; the crystalline VI is fairly stable to hydrolysis. VI does not react with sulfur dioxide under various conditions attempted.

The products from the reactions of phenyl salicylate and 2-hydroxybenzophenone with phosphorus pentachloride have been designated as *ortho*-substituted phenyl phosphorodichloridates (I and II, respectively) rather than alternative heterocyclic structures on the basis of P^{31} nmr and chemical studies.[2] Since most of



I



II

the chemical studies aimed at structure elucidation were with the product from phenyl salicylate, further work on the 2-hydroxybenzophenone product (II) was desirable since the chemical evidence rested mainly on analogy with the results on the phenyl salicylate product. Furthermore, it was of interest to compare the behavior of the phenolic ketone product with that from phenyl salicylate. Since 5-chloro-2-hydroxybenzophenone (III) was readily obtainable commercially,[3] it was selected in place of the less available 2-hydroxybenzophenone.[4]

Results and Discussion

The reaction of phosphorus pentachloride with 5-chloro-2-hydroxybenzophenone (III) was entirely analogous to the reaction with 2-hydroxybenzophenone. The colorless crystals obtained gave the correct elemental analysis and molecular weight for the expected product. Active chlorine (nonaromatic) as determined by basic hydrolysis followed by potentiometric titration of an acidified solution with silver nitrate showed that the expected 4 equiv of chloride ion were present. Although the crystals were stable to water, 5-chloro-2-hydroxybenzophenone was obtained by hydrolysis of an aqueous dioxane solution, showing that the basic carbon skeleton of the compound was unchanged from that of the starting compound. The infrared spectrum of the product showed no absorption bands in the carbonyl or hydroxyl stretching regions. The pertinent infrared absorption bands were close to those observed previously for the corresponding product (II) from 2-hydroxybenzophenone[2]: P═O stretching at 7.70 μ; P–O–C (aromatic) (C–O portion) at 8.38 μ; P^{+5}–O–C (aromatic) (P—O portion) at 10.50 μ. The value for the P^{31} nmr chemical shift was the same as that found previously[2] for the analogous product (II) from 2-hydroxybenzophenone, a value of 0.0 ppm.

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(1) Based on M.S. Thesis of L. Y. C. M., Baylor University, 1964.

(2) A. G. Pinkus, P. G. Waldrep, and S. Y. Ma, *J. Heterocyclic Chem.*, **2**, 357 (1965).

(3) The authors express appreciation to the Dow Chemical Co. for gifts of generous samples.

(4) 2-Hydroxybenzophenone is obtained in low yield from the Fries rearrangement of phenyl benzoate[5] or by the Friedel-Crafts benzoylation of phenol.

(5) N. M. Cullinane, N. M. E. Morgan, and C. A. J. Plummer, *Rec. Trav. Chim.*, **56**, 627 (1937).