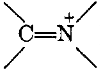
72% and the hexahydrojulolidine to about 22%. None of the other peaks accounted for more than 2% of the total.

The crude bases from three batches were combined (69.8 g.) and redistilled through a Cason column. The fractions boiling at 83–85°/1.5–1.6 mm. were combined (44.5 g.) and converted to the perchlorate salt by adding a 1:1 mixture of ethanol and 70% perchloric acid to an ether solution of the bases. Recrystallization of the perchlorate twice from ethanol yielded 53.6 g. (44% on the basis of 75.3 g. of julolidine) of colorless needles that melted (>280°) with decomposition at a point which depended upon the rate of heating. The infrared spectrum was identical with that of an authentic sample kindly supplied by Prof. F. Bohlmann,[6] ν_{max}^{Nujol} 1680 cm.$^{-1}$ ().

Anal. Calcd. for $C_{12}H_{20}ClNO_4$: C, 51.89; H, 7.26; N, 5.04. Found: C, 52.07; H, 7.21; N, 4.92.

Pure tetrahydrojulolidinium perchlorate was converted to the free base in 83% yield, b.p. 62° (0.04 mm.), n^{25}D 1.5369, $\nu_{max}^{CCl_4}$ 1645 cm.$^{-1}$; ν_{max}^{film} 1655 cm.$^{-1}$.

Anal. Calcd. for $C_{12}H_{19}N$: C, 81.30; H, 10.80; N, 7.90. Found: C, 81.09; H, 11.00; N, 7.97.

1-Methyl-Δ^5-tetrahydrojulolidinium Iodide (X, $X^- = I^-$).—To a solution of 5.0 g. (0.0282 mole) of tetrahydrojulolidine in 25 ml. of acetone was added about 5.7 g. (0.0403 mole) of methyl iodide. After standing about 5 min., beautiful white needles formed. The crystals were removed by filtration and washed with a small amount of acetone and a small amount of ether, m.p. 221–222° dec., yield 7.42 g. (83%), no infrared absorption in the 1600–1700-cm.$^{-1}$ region. A small sample was recrystallized from acetone for analysis.

Anal. Calcd. for $C_{13}H_{22}IN$: C, 48.91; H, 6.95; N, 4.38. Found: C, 48.98; H, 6.95; N, 4.30.

1-Methyl-Δ^5-tetrahydrojulolidinium Perchlorate (X, $X^- = ClO_4^-$).—To a warm acetone solution of 2.0 g. (0.00627 mole) of tetrahydrojulolidinium iodide was added dropwise an acetone solution of silver perchlorate until no more precipitate was formed. The silver iodide was removed by filtration and the filtrate was evaporated to dryness, m.p. 138–140°, yield 1.34 g. (73%). The residue was recrystallized from isopropyl alcohol, irregular prisms, m.p. 140–140.5°. No infrared absorption was present in the 1600–1700-cm.$^{-1}$ region. The ultraviolet spectrum of 1-methyltetrahydrojulolidinium perchlorate was measured in water after a baseline using water in both sample and reference beams was determined in duplicate. The sample run at a concentration of 6.86 × 10^{-5} mole/l. was also made in duplicate. The instrument was calibrated using mercury lines and the corrections were applied to the wave length values. The following values are significant: $\lambda_{\epsilon\,1000}^{H_2O}$ 213.8 mμ; $\epsilon_{\lambda\,200\,m\mu}^{H_2O}$ 7.2 × 10^3. 1,1-Dimethylpyrrolidinium perchlorate served as a saturated model. A solution in water at 6.52 × 10^{-5} mole/l. showed no appreciable absorption in the ultraviolet down to 200 mμ. The calculated apparent molecular weight of the monomer is 145.88. The apparent molecular weight in water was determined as 137.[35,36] The n.m.r. spectrum in methylene chloride showed a singlet signal at τ = 6.69 for the $N^+—C\underline{H}_3$, complex absorption at τ = 6.34 and 6.39 for four ring protons next to nitrogen and broad signals at τ = 7.81 and 8.42 for other ring protons.

Anal. Calcd. for $C_{13}H_{22}ClNO_4$: C, 53.51; H, 7.60; N, 4.80. Found: C, 53.33; H, 7.52; N, 4.84.

The acute toxidity of this compound was found to be LD_{50} = 15.43 ± 0.95 mg./kg. in fasted mice by intravenous route. As in the case of 1,1-dimethyl-Δ^9-octahydroquinolinium perchlorate, mild tonic convulsions which lasted 5 min. were observed immediately after injection on all doses. Deaths were immediate.

Demethylation of 1-Methyl-Δ^5-tetrahydrojulolidinium Iodide.[30,31]—Anhydrous tetrahydrofuran (75 ml.) freshly distilled from lithium aluminum hydride was heated under reflux with 3.0 g. (0.0792 mole) of lithium aluminum hydride for 1 hr. Then 4.0 g. (0.0125 mole) of finely crystalline 1-methyltetrahydrojulolidinium iodide was added. Gas evolution ceased after 3 hr., when 77% of the theoretical amount of methane had been collected. The heating was stopped, and the mixture was stirred overnight. The reaction mixture was cautiously hydrolyzed with water and sodium hydroxide solution, and the solution was extracted with ether. The dried ether extracts were evaporated to leave 2.0 g. (90%) of crude tetrahydrojulolidine. A perchlorate salt had an infrared spectrum identical with that of tetrahydrojulolidinium perchlorate. The crude tetrahydrojulolidine was distilled (78°/0.3 mm.) and afforded 1.25 g. of pure tetrahydrojulolidine, the infrared spectrum of which was identical with that of tetrahydrojulolidine originally prepared.

Steric Effects in Methyl-substituted 1-Tetralones

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The combined effects of four methyl groups at positions 4, 5, 7, and 8 in the 1-tetralone moiety strongly inhibited addition of the Grignard reagent to the carbonyl group. This was in contrast to corresponding dimethyl- and trimethyl-1-tetralones, having two or three methyl groups at these positions, showing that the steric hindrance is not due to substitution at any one position. Molecular models indicate that the most favorable conformation of 4,5,7,8-tetramethyl-1-tetralone is with the 4-methyl group axial and the alicyclic ring in the boat form, causing considerable shielding of the carbonyl group by the 4-methyl group. The resulting lack of coplanarity of the carbonyl group with the aromatic ring was confirmed by ultraviolet spectral studies. This effect of 4,5-dimethyl crowding appears to be greater than the steric effect of a methyl group at position 2.

While preparing a series of polymethylnaphthalenes, we have had occasion to employ the reaction of methyl-substituted 1-tetralones with the methyl Grignard reagent, followed by dehydration and dehydrogenation of the tertiary alcohols. This generally good method was unsuccessful in one case, because of strikingly low reactivity of 4,5,7,8-tetramethyl-1-tetralone with the Grignard reagent.

Two of the ketones, 5,8-dimethyl-1-tetralone (I)[1] and 5,7,8-trimethyl-1-tetralone (II)[2] were

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[1] E. de B. Barnett and F. G. Sanders, *J. Chem. Soc.*, 434 (1933).

[2] W. Cocker, B. E. Cross, and J. McCormick, *ibid.*, 72 (1952).

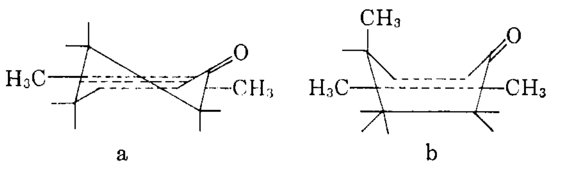
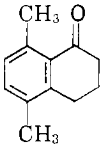


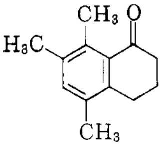
Fig. 1.—Conformation of methyl-substituted 1-tetralones.

synthesized from succinic anhydride and *p*-xylene and with pseudocumene and had previously been treated successfully with methylmagnesium iodide. 4,5,8-Trimethyl-1-tetralone (III), which was synthesized by Mosby[3] from *p*-xylene and γ-valerolactone, has been treated with methyllithium but the Grignard reagent was not tried by that investigator. We have treated these ketones with methylmagnesium iodide, followed by dehydration and dehydrogenation to form the corresponding polymethylnaphthalenes in 70 to 80% yields.[4]

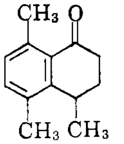
4,5,7,8-Tetramethyl-1-tetralone (IV) was synthesized by Mosby's general method,[3] in which γ-1,2,4,5-trimethylphenylvaleric acid, obtained by the condensation of pseudocumene with γ-valerolactone in the presence of two moles of aluminum chloride, was cyclized with polyphosphoric acid.



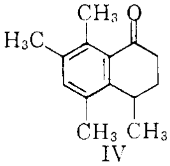
I



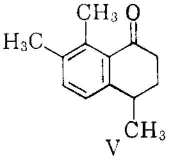
II



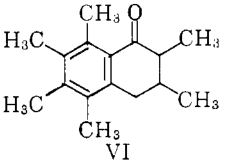
III



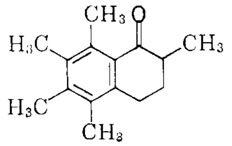
IV



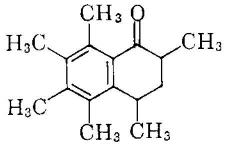
V



VI



VII



VIII

This reactivity of IV was markedly low toward the Grignard reagent. A variety of forcing conditions gave a maximum yield of five percent of impure 1,2,4,5,8-pentamethylnaphthalene. Using methyllithium, compound IV was converted to a 61% yield of this hydrocarbon.

This result shows that the combined effects of four methyl groups at positions 4, 5, 7, and 8 give rise to much more steric hindrance than a combination of any three of them.[5] The effects of groups at positions 7 and 8 are well known, the former being buttressing on the bulk effect of the latter.[6]

Inspection of molecular models, such as Stuart-Briegleb models, shows that the most stable conformation of compound IV is with the alicyclic ring in the boat form and the 4-methyl group axial. The equatorial position for the 4-methyl group in either the chair or the boat form of the alicyclic ring would involve a large amount of interference with the 5-methyl group, while an axial 4-methyl group in the chair form would be subject to considerable interaction with the axial 2-hydrogen atom. In the preferred conformation (Fig. 1b), the methyl group is on the side of the ring toward which the carbonyl group is tilted, and it shields considerably the otherwise open side of the carbonyl group. The conformation of compound III would be similar, its greater reactivity being ascribed to the lack of buttressing of the 8-methyl group.

In the absence of the 5-methyl group, the models —*e.g.*, Fig. 1a—suggest that the 4-methyl group assumes the energetically favored equatorial conformation in the chair form. With one side of the carbonyl group exposed, Grignard addition takes place readily. We hoped to check this point further by studying the reaction of 4,7,8-trimethyl-1-tetralone (V) with methylmagnesium iodide, but unfortunately we were unable to synthesize this ketone.

The above reasoning would imply that the well known effect of *ortho*-methyl groups, causing noncoplanarity of the carbonyl group in phenyl ketones,[7] would be somewhat suppressed by the axial 4-methyl groups in compounds III and IV. It has been shown by Braude and collaborators that the intensity of the K-band of the ultraviolet spectra of this class of compounds provides a good measure of the coplanarity of the carbonyl group.[8] Measurement of the ultraviolet absorption spectra of the four tetralones studied here showed that the two compounds, III and IV, which had methyl groups at positions 4 and 5 as well as 8, had somewhat more intense K-bands than the other 8-methyl substituted tetralones (Table I). This may be considered evidence for the correctness of the assignment of conformation for this series of compounds.

It is also interesting that compound IV shows a significantly higher carbonyl stretching frequency than the other tetralones, as shown in Table I. This is indicative of a decreased CCC angle at the carbonyl group, caused by increased *s* character of the carbonyl bond, as is regularly observed in decrease of ring size in cyclic ketones and benzocyclan-

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[3] W. L. Mosby, *J. Am. Chem. Soc.*, **74**, 2564 (1952).

[4] Likewise, 4,5,7-trimethyl-1-tetralone and 2,3,4-trimethyl-1-tetralone were found to be converted by this sequence to 1,4,5,7- and 1,2,3,4-tetramethylnaphthalenes in 72.3 and 74.1% yields, respectively.

[5] One not studied here, 4,5,7-trimethyl-1-tetralone, would undoubtedly be more reactive than the other three, since it does not possess the 8-methyl group, a major source of steric hindrance.

[6] M. Rieger and F. H. Westheimer, *J. Am. Chem. Soc.*, **72**, 19 (1950).

[7] M. T. O'Shaughnessy and W. H. Rodebush, *ibid.*, **62**, 2906 (1940).

[8] Summarized by E. A. Braude in "Progress in Stereochemistry," W. Klyne, ed., Vol. I, Academic Press, New York, 1954, pp. 144–148.

TABLE I

ULTRAVIOLET[a] AND INFRARED[b] SPECTRAL DATA FOR 1-TETRALONES

Compound	K-Band mμ	K-Band ε × 10^{-3}	B-Band mμ	B-Band ε × 10^{-3}	C=O Str., cm.$^{-1}$
I	254	11.2	307	2.3	1670
II	258	10.6	313	2.4	1670
III	256	11.6	306	2.4	1660
IV	263	11.3	302	2.4	1695
1-Tetralone[c]	248	12.2	294	1.5	1681

[a] In 95% ethanol solutions. [b] KBr discs. [c] Ultraviolet data from "Ultraviolet Spectra of Aromatic Compounds," by R. A. Friedel and M. Orchin, John Wiley & Sons, New York, N. Y., 1951, spectrum 59; infrared data from ref. 9.

ones.[9] Thus, the crowding of both sides of the carbonyl group by methyl groups in compound IV appears to have caused distortion of the alicyclic ring, the carbonyl group probably being pushed outwards.

Steric crowding of the carbonyl group due to the combined effect of methyl groups at positions 4 and 5 appears to be more pronounced in these reactions than the effect of a methyl group at position 2. 2,3,5,6,7,8-Hexamethyl-1-tetralone[10] (VI) and 2,5,6,7,8-pentamethyl-1-tetralone[11] (VII) have been reported to react with the methyl Grignard under forcing conditions. Molecular models show that the most stable conformation of a methyl group at position 2 will be equatorial, which is away from the plane of the pi orbitals.

Carruthers and Gray[11] have reported that 2,4,5,6,7,8-hexamethyl-1-tetralone (VIII) did not react with either methylmagnesium iodide or with methyllithium, even under forcing conditions. They attributed this situation to a "buttressing effect" of the 4-methyl group, presumably buttressing on the series of methyl groups on the benzene ring. It seems more likely that the cause is the effect described here.

Experimental

The infrared spectra were recorded with a Perkin-Elmer "Infracord" using the potassium bromide disk technique. The ultraviolet spectra were determined by means of a Cary Model 11 spectrophotometer.

5,8-Dimethyl-1-tetralone (I) was synthesized from *p*-xylene and succinic anhydride by the procedure of Barnett and Sanders.[1] In a similar fashion, 5,7,8-trimethyl-1-tetralone (II) was synthesized by the procedure of Cocker, Cross, and McCormick.[2] The reaction of *p*-xylene and γ-valerolactone by Mosby's procedure[3] afforded 4,5,8-trimethyl-1-tetralone (III).

4,5,7,8-Tetramethyl-1-tetralone (IV).—A solution of 12 g. of pseudocumene and 10 g. of γ-valerolactone (0.10 mole each), contained in a three-necked round-bottomed flask protected from atmospheric moisture by a calcium chloride drying tube was cooled in an ice bath and efficiently stirred, while 26.7 g. (0.20 mole) of anhydrous aluminum chloride was added in portions through a wide-bore tube. After the addition was completed, the reaction mixture was kept in the ice bath for 3 hr. longer, and then was kept at room temperature for 12 hr. The reaction mixture was processed by pouring into iced dilute hydrochloric acid, extraction with ether, and washing of the ether extracts with dilute hydrochloric acid, water, and 0.3 *M* sodium carbonate. The basic solution obtained was acidified and extracted with ether. After washing, drying, and evaporation of solvent, these extracts yielded 19.8 g. (90%) of a brown liquid, which was the somewhat crude γ-(2,4,5-trimethylphenyl)valeric acid.

A mixture of 19.0 g. of the above acid and 70 g. of polyphosphoric acid was heated carefully in an open vessel with stirring until a temperature of 150° was reached, which was maintained for 5 min. The mixture was decomposed by pouring into 500 ml. of ice water, followed by ether extraction. After the extracts were washed with 10% sodium hydroxide and with water and dried over magnesium sulfate, distillation gave 13.6 g. (77%) of 4,5,7,8-tetramethyl-1-tetralone as a pale yellow liquid, b.p. 156–160° (7.5 mm.), n^{20}_{D} 1.5615. Since gas chromatography showed the presence of a small quantity of one or two contaminants, it was passed through a basic ion exchange column (amberlite, to remove any starting acid), followed by collection from a gas chromatographic column (5% fluorosilicone on chromosorb at 181°; retention time 18.2 min. at 13 ml./min. flow).

Anal. Calcd. for $C_{14}H_{18}O$; C, 83.0%; H, 8.96%. Found: C, 82.98%; H, 8.73%.

The ultraviolet spectrum was determined on the analytical sample. The optical density, in a 1.0-cm. cell, of a 95% alcoholic solution of 0.0370 g. in 10.0 ml. was 2.07 at 263 mμ and 0.360 at 302 mμ.

The 2,4-dinitrophenylhydrazone formed minute red leaflets, m.p. 277–278°.

Anal. Calcd. for $C_{20}H_{22}O_4N_4$: N, 14.65. Found: N, 14.60.

Addition of Methylmagnesium Iodide.—Treatment of 5,8-dimethyl-1-tetralone (I) with methylmagnesium iodide according to the procedure of Barnett and Sanders[1] gave, after distillation, a yellow oil which was dehydrogenated with 5% palladium on charcoal at 270–300° for 1 hr. Removal of the catalyst resulted in a 79% yield of 1,4,5-trimethylnaphthalene after one crystallization from methanol. A pure sample of the hydrocarbon melted 61.5–62.0° (lit.,[1,3,12] m.p. 63°, 59.6–60.6°, 61.5–62.0°).

In a similar manner, 5,7,8-trimethyl-1-tetralone (II) gave a 75% yield of 1,2,4,8-tetramethylnaphthalene. A pure sample melted 65.0–65.3° (lit.,[2] m.p. 56–57°). The picrate formed orange needles, m.p. 148–149° (lit.,[2] m.p. 149–149.5°).

In a similar manner, 4,5,8-trimethyl-1-tetralone (III) gave 70% of 1,4,5-8-tetramethylnaphthalene, m.p. 131–132° (lit.,[3] m.p. 132–133°). The picrate formed orange needles, m.p. 155–156° (lit.,[3] m.p. 154.6–155.4°).

In the only reaction in which 4,5,7,8-tetramethyl-1-tetralone (IV) could be caused to react with methylmagnesium iodide, a 1:5 molar ratio of ketone to Grignard reagent was stirred at room temperature during 4 hr. The ether was removed by distillation and the volume was maintained by the gradual addition of dry benzene. When the temperature of the vapors reached 80°, the mixture was refluxed with stirring for 24 hr. Decomposition of the reaction mixture with dilute hydrochloric acid, extraction of the organic material with ether, distillation of the organic material under reduced pressure, and dehydrogenation of the distillate over 5% palladium on charcoal at 300–320° during 2 hr. gave a yellow oil. Upon treatment with a few drops of methanol, 5% of colorless leaflets of 1,2,4,5,8-pentamethylnaphthalene, m.p. 58–60°, was obtained.

Addition of Methyllithium.—A 250-ml. three-necked, round-bottomed flask equipped with a stirrer, separatory

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[9] W. M. Schubert and W. A. Sweeney, *J. Am. Chem. Soc.*, **77**, 4172 (1955).

[10] B. J. Abadir, J. W. Cook, and D. T. Gibson, *J. Chem. Soc.*, 8 (1953).

[11] W. Carruthers and J. D. Gray, *ibid.*, 1280 (1958).

[12] R. F. Evans, J. C. Smith, and F. B. Strauss, *J. Inst. Petroleum*, **40**, 7 (1954).

funnel, and a reflux condenser which was protected with a calcium chloride drying tube was swept out with dry nitrogen. To the flask was added 1.5 g. (0.216 g.-atom) of clean lithium wire and 50 ml. of dry ether. To the stirred, cooled mixture was added dropwise, over 30 min., a solution of 15.5 g. (0.11 mole) of methyl iodide in 50 ml. of dry ether. After 1 hr., only traces lithium remained, and to the cold solution was added, dropwise, a solution of 5.0 g. (0.025 mole) of 4,5,7,8-tetramethyl-1-tetralone in 50 ml. of dry ether. Stirring was continued for 1 hr. and the solution was allowed to stand overnight at room temperature. The mixture was decomposed with cold, dilute hydrochloric acid and the layers were separated. The aqueous layer was extracted with ether and the ether extracts were combined and washed with dilute hydrochloric acid, dilute sodium bicarbonate, and with water. The extracts were dried over anhydrous magnesium sulfate, the ether was removed by distillation, and the residue was vacuum distilled at 162–176°/18 mm. The distillate showed a fairly strong infrared carbonyl band indicating that reaction was not complete.

The mixture was dehydrogenated with 5% palladium on charcoal at 280–310° for 3 hr. Benzene was added to the cooled mixture and the solution was filtered to remove the catalyst, the solvent was removed by distillation, and the residual material was vacuum distilled. The distillate was crystallized from absolute alcohol to give 3.0 g. (61%) of colorless leaflets of 1,2,4,5,8-pentamethylnaphthalene, m.p. 58–59° (lit.,[13] m.p. 60–61°). The infrared spectrum shows strong absorption at 11.65 and 12.35μ, which is characteristic of a 1,2,4,5,8-structure.[14]

The picrate formed small dark red needles, m.p. 156–156.5° (lit.,[10] m.p. 156–157°).

Acknowledgment.—This research was supported by a grant from the Petroleum Research Fund administered by the American Chemical Society. Grateful acknowledgment is hereby made to the donors of said fund.

(13) L. Ruzicka, H. Schinz, and P. H. Müller, *Helv. Chim. Acta*, **27**, 195 (1944).

(14) Unpublished data in our laboratory.

Friedel–Crafts Reaction of Ferrocene with Phosphorus Trichloride. Formation of Triferrocenylphosphine[1–3]

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Received September 11, 1961

The preparation of the first known compounds with phosphorus bonded to ferrocene carbon is described. The reaction of ferrocene with phosphorus trichloride in the presence of anhydrous aluminum chloride under conditions favoring monosubstitution gave rise to three products: ferrocenylphosphonous dichloride, diferrocenylphosphinous chloride, and triferrocenylphosphine. Formation of these compounds was demonstrated when the following derivatives were isolated: ferrocenylphosphinic acid; diferrocenylphosphinic acid and its hydrate; triferrocenylphosphine oxide, several of its hydrates, and a hydrogen chloride complex; and triferrocenylmethylphosphonium iodide. A tertiary phosphine was thus observed among the products of a Friedel–Crafts reaction for the first time. Infrared absorption bands were assigned to the ferrocenyl-phosphorus group. The possible existence of an interaction between phosphoryl and the ferrocene residues in the anhydrous phosphine oxide is discussed. Spectral evidence for ring hydration and ring hydrochlorination is presented.

Although ferrocene has been reported to undergo a wide variety of substitution reactions that are characteristically aromatic, including Friedel–Crafts acylation and alkylation,[5,6] no investigation of its behavior towards phosphorus trichloride in a Friedel–Crafts reaction has heretofore been described. Indeed, no report of an attempt to introduce, by any other method, a phosphorus-containing function onto a cyclopentadiene ring of ferrocene has been located.[7] Formation, *via* a Friedel–Crafts reaction, of compounds with phosphorus bonded to ferrocene carbon is reported here in detail for the first time.

For more than a half century, aromatic hydrocarbons have been known to interact with phosphorus trichloride in a Friedel–Crafts reaction to give arylphosphonous dichlorides (Ia) and diarylphosphinous chlorides (IIa).[8]

$$\mathrm{ArH} \xrightarrow[(\mathrm{AlCl_3})]{\mathrm{PCl_3}} \underset{\mathrm{Ia}}{\mathrm{ArPCl_2}} + \underset{\mathrm{IIa}}{(\mathrm{Ar})_2\mathrm{PCl}}$$

In general, the reaction of an aromatic compound with phosphorus trichloride catalyzed by anhydrous aluminum chloride gives up to 80% yields of Ia in two to eight hours of refluxing.[9] With reaction periods of the order of thirty hours, 30 to 50% yields of IIa are possible. Increasing alkyl substitution of the benzene ring leads to increased yields of com-

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(1) Supported by the U. S. Army Research Office.

(2) Abstracted from a dissertation submitted to the Temple University Graduate Board in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

(3) Presented in part before the Division of Organic Chemistry, 3rd Delaware Valley Regional Meeting of the American Chemical Society, Philadelphia, Pennsylvania, February 25, 1960.

(4) (a) Frankford Arsenal; (b) Temple University.

(5) For a review, see P. L. Pauson, *Quart. Rev.* (London), **9**, 408 (1955).

(6) For a recent bibliography, see M. D. Rausch, E. O. Fischer, and H. Grubert, *J. Am. Chem. Soc.*, **82**, 76 (1960).

(7) Ferrocenylphosphonic acid has been synthesized in these laboratories from lithioferrocene.

(8) A. Michaelis, *Ann.*, **293**, 193 (1896); *ibid.*, **293**, 261 (1896); *ibid.*, **294**, 1 (1897).

(9) For a review, see G. M. Kosolapoff, "Organophosphorus Compounds," John Wiley & Sons, Inc., New York, N. Y., 1950, pp. 43–46.