chromatography (glpc) on a 4-ft THEED on Chromosorb W column at 110° showing the presence of only phenacyl alcohol and ethyl phenacyl ether (approximately 30:70 ratio) and several very minor (unidentified) fractions. No ethyl phenylacetate was present in the neutral fraction.

The sodium hydroxide extracts (from above) were acidified and extracted with ether. The ether solutions were treated with small portions of ethereal diazoethane. The solvent was removed and the residues (approximately 5% by weight) were analyzed by glpc. No ethyl phenylacetate was present. Total recoveries of products averaged 90–95%.

Registry No.—Phenacyl chloride, 532-27-4; phenacyl bromide, 70-11-1; phenacyl iodide, 00-00-0; p-NO_2-phenacyl bromide, 99-81-0; p-Cl-phenacyl bromide, 536-38-9; m-CH_3O-phenacyl bromide, 000-00-0; p-CH_3O-phenacyl bromide, 2632-13-5.

Group Contributions to Phosphorus-31 Chemical Shifts of Tertiary Phosphines

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Additive alkyl group contributions to the phosphorus-31 chemical shifts of tertiary phosphines have been assigned for 12 alkyl groups. These group contributions are shown to be related to the number of carbon substituents on the α and β positions of the alkyl groups. Previously unreported phosphorus-31 chemical shift data are also presented for some tertiary phosphines.

The idea of additive group contributions to the phosphorus-31 chemical shifts in trivalent phosphorus compounds was first suggested in 1956 by Van Wazer[1] and co-workers. This concept has been criticized favorably and unfavorably since then and much of the discussion, ranging from simple empiricism to quantum mechanics, has been concerned with the magnitude and sign of the chemical shifts.[2–9] There seems to be general agreement that bond angles and electronegativities of the groups are major factors in determining the ^{31}P chemical shift, while other factors such as the effect of the lone-pair excitation energy and π bonding are not well agreed upon. One remarkable paper[10] proposes that the ^{31}P chemical shift in trivalent phosphorus compounds depends only on bond angles and "magnetic configuration" of the molecule. This same idea extended to quadruply bonded phosphorus compounds implies that all tetraorganophosphonium salts, *i.e.*, tetraphenyl-, tetramethyl-, and tetrabutylphosphonium, would have the same ^{31}P chemical shift. This is not the case.[11]

Most of the reports have attempted to treat all types of PZ_3 (Z = alkyl, aryl, alkoxy, halogen, hydrogen, etc.) compounds, but we are concerned in this work with only trialkyl-, dialkylphenyl-, and alkyldiphenylphosphines, which have previously been treated by both quantum mechanical[9] and empirical procedures.[3,4]

In previous work it was noted that the order of group contributions is Me > Et > i-Pr > t-Bu in tertiary, secondary, and primary phosphines and in quaternary phosphonium salts (part of the series was also reported for phosphonates[1]). This is the reverse order of the inductive effect as one would have normally expected the t-butyl to shield phosphorus most in this series, and hence result in the highest ^{31}P chemical shift and the largest group contribution. The experimental series might be explained in several ways. (a) Alkyl groups are more electronegative than phosphorus in phosphines and hence t-butyl can accept more electronic change than isopropyl, etc. (it is well known that, if Z is more electronegative than R in RZ compounds, the electronegativity series is Me > Et > i-Pr > t-Bu, and it has also been shown that, if R is more electronegative than Z, the electronegative series is t-Bu > i-Pr > Et > Me).[12] However, the electronegativity argument probably loses its cogency within a series of alkyl groups which all lie very close together in electronegativities and, indeed, very close to the electronegativity of phosphorus itself. Huheey[13] has calculated that the electronegativities of the alkyl groups in this study lie between 2.34 and 2.39 and that a phosphorus hybrid p orbital which contains 13.7% s character (based on the observed bond angle in trimethylphosphine) has an electronegativity of 2.38. If three or four significant figures in electronegativity values can be reliable (which we doubt), then perhaps electronegativity arguments could be applied to this problem. (b) As the alkyl group becomes larger the bond angles increase and the phosphorus bonds gain s character with concomitant increase in the p character of the lone pair. Assuming that a lone pair is more effective in shielding the nucleus than bonding pairs, a lone pair with large s character would be most effective in shielding because of its spherical symmetry about the nucleus. Thus small alkyl groups (small bond angles, large s character of the lone pair, high shielding) should have large group contributions. (c) Hyperconjugation of the α protons could occur, $R_2\ddot{P}CR_2H \leftrightarrow R_2\underset{-}{\ddot{P}} = CR_2\,\underset{+}{H}$, leading to the most shielding of phosphorus by those alkyl groups with the largest number of α protons. This type of hyperconjugation has been sug-

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[12] J. E. Huheey, *ibid.*, **69**, 3284 (1965).

[13] J. E. Huheey, private communication.

gested[11,14] to occur in phosphonium salts, although one would not expect it to be so important in phosphines where the phosphorus already has a lone pair of electrons. The phosphorus chemical shifts do vary widely (over a range of at least 80 ppm for tertiary phosphines) and hence are very sensitive to electronic effects. The range of chemical shifts in quaternary phosphonium salts is quite a bit smaller (*ca.* 30 ppm), although electrostatically hyperconjugation is more favorable. A possible explanation of these differences between phosphonium salts and phosphines could be the favorable angles, which are closer to right angles, *i.e.*, *ca.* 92 to 100°, in phosphines, and which allow reasonable hyperconjugative π bonding with the phosphorus d orbitals. In phosphonium salts the angles are almost or exactly tetrahedral and not favorable for extensive π bonding.

Experimental Section

Preparations of phosphines were carried out in an atmosphere of prepurified nitrogen. The phosphorus-31 nmr was measured on pure liquids or on methylene chloride solutions of solids in 15-mm sample tubes with 85% phosphoric acid as an external reference in a sealed 3-mm tube inserted concentrically through a serum stopper in the larger tube. Measurements were made at 24.3 Mc/sec on a Varian Associates DP-60 instrument. Calibrations were made by usual side-band techniques. Proton nmr was measured on a Varian Associates A-60A spectrometer. Analyses were performed by Midwest Microlab, Inc., Indianapolis, Ind.

Two methods were employed for the preparations.

A. Lithium Diphenylphosphide Method. Preparation of Neopentyldiphenylphosphine.—Lithium diphenylphosphide was prepared by stirring a suspension of 26.2 g (0.1 mole) of triphenylphosphine, 200 ml of dry tetrahydrofuran (THF), and 1.4 g (0.2 g-atom) of lithium metal at room temperature for 3 hr.[15,16] After this time most of the lithium had dissolved to produce a deep red solution. The solution was heated to reflux temperature and 9.3 g (0.1 mole) of *t*-butyl chloride was added over a 5-min period. After refluxing for 15 min, a solution of 10.7 g (0.1 mole) of neopentyl chloride in 25 ml of THF was added over a 20-min period. The reflux was maintained for 12 hr, during which time the color changed from red to yellow. The solution was cooled to room temperature and 150 ml of saturated, deoxygenated aqueous ammonium chloride was added rapidly. After removal of the aqueous layer, the organic layer was dried over sodium sulfate for 3 hr, then filtered through glass wool, and distilled at atmospheric pressure to remove solvent and then at reduced pressure to yield 13.2 g (52%) of clear distillate, bp 116–122° (0.1 torr). The proton nmr had an aromatic multiplet (relative intensity, 10) and the neopentyl group was identified by a doublet (J_{PCH} = 3.5 cps) at τ 7.85 (relative intensity, 2) and a singlet at 8.98 (relative intensity, 9).

Methylneopentyldiphenylphosphonium Bromide.—Addition of methyl bromide to an ether solution of neopentyldiphenylphosphine gave the phosphonium salt, mp 197–198° (recrystallized from chloroform–ethyl acetate). The hexafluorophosphate, mp 155–156°, was prepared metathetically from ammonium hexafluorophosphate and the bromide in water.

Anal. Calcd for $C_{18}H_{24}P_2F_6$: C, 51.93; H, 5.81. Found: C, 51.67; H, 5.93.

B. Grignard Method. Preparation of Diisobutylphenylphosphine.—Isobutylmagnesium bromide was prepared from 21.0 g (0.865 g-atom) of magnesium turnings and 100 g (0.8 mole) of isobutyl bromide in 500 ml of dry THF. The Grignard solution was cooled in a Dry Ice–acetone bath and a solution of 52.5 g (0.29 mole) of dichlorophenylphosphine in 50 ml of THF was added dropwise. The cooling bath was removed and the solution was allowed to warm to room temperature. The solution was hydrolyzed with deoxygenated, saturated ammonium chloride solution and the layers were separated. The organic layer was dried over calcium chloride, then distilled at atmospheric pressure to remove solvent. The residue was distilled at reduced pressure to give *ca.* 50 ml of crude distillate, bp 75–85° (0.5 torr). Redistillation with a 12-in., vacuum-jacketed, silvered Vigreux column gave 33.7 g (52%) of clear distillate, bp 108–109° (3 torr) [lit.[17] 168° (50 torr)].

TABLE I

PREPARATION OF TERTIARY PHOSPHINES

Phosphine	Method	Bp, °C (torr)	Yield, %
Neopentyldiphenyl	A	116–122 (0.1)	52
Isobutyldiphenyl	B	113–115 (0.5)	30
Diisobutylphenyl	B	108–109 (3)	52
sec-Butyldiphenyl	A	141–145 (1.5)	73
Di-*sec*-butylphenyl	B	107–110 (2)	69
Cyclopentyldiphenyl	B	160–163 (2)	48
Dicyclopentylphenyl	B	112–115 (0.7)	63

Preparation of *t*-Butyldiphenylphosphine.—Chlorodiphenylphosphine (33.3 g, 0.15 mole) in 50 ml of THF was added to the cold (−78°) solution of the Grignard reagent made from *t*-butyl chloride (60.8 g, 0.66 mole) and magnesium (29.1 g, 1.2 g-atoms) in 500 ml of THF. Hydrolysis of the mixture with saturated ammonium chloride solution, separation of the organic layer, distillation of the THF at atmospheric pressure, and distillation of the residue at reduced pressure gave 20 g of crude product, bp 120–155° (1 torr).

This distillate had a ^{31}P nmr spectrum consisting of a peak at −17.1 ppm and a symmetrical doublet at +40.2 ppm (J = 216 cps). The ^{31}P chemical shift of *t*-butyldiphenylphosphine predicted by extension of the data in ref 11 is −19.8 ppm.[18] The singlet is therefore assigned to this compound. The doublet is due to diphenylphosphine; chemical shift[19] is +41.1 ppm (J_{PH} = 214 cps[19]). The crude distillate was distilled with a 12-in., silvered, vacuum-jacketed Vigreux column to give two fractions with boiling points of 115–118° (2 torr) [Ph_2PH; lit.[20] bp 150–151° (12 torr)] and 144–146° (2 torr) (t-$BuPh_2P$). The proton nmr spectrum of the high-boiling fraction has an aromatic multiplet (relative area 10) at low field and a methyl doublet (relative area 9) at τ 8.84 and J_{PCCH} = 12.4 cps, which is expected for *t*-butyldiphenylphosphine.

The molar ratio of diphenylphosphine to *t*-butyldiphenylphosphine as estimated from the ^{31}P nmr peak areas was 6:1, corresponding to an over-all yield of about 10% for *t*-butyldiphenylphosphine.

The reaction was repeated with a steady stream of nitrogen used to sweep all gases into a trap at −78°. The condensed vapors were treated with bromine at various stages of the procedure and the products were analyzed by gas chromatography for 1,2-dibromo-2-methylpropane (by comparison of retention times with an authentic sample of the dibromide). Isobutylene was evolved during the addition of chlorodiphenylphosphine to the Grignard reagent. Isobutylene was not detected during the preparation of the Grignard nor during hydrolysis of the reaction mixture. The preparation of *t*-amyldiphenylphosphine was carried out in the same manner as that for *t*-butyldiphenylphosphine. The crude product contained mostly Ph_2PH as identified from the ^{31}P nmr spectrum. The only other peak in the nmr occurred at −15.4 ppm and has been assigned to *t*-amyldiphenylphosphine, which was not isolated.

Results

The group contributions of benzyl, phenyl, and 12 alkyl groups are given in Table II. The sum of the respective group contributions for the three groups of

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(14) D. H. McDaniel, *Science*, **125**, 545 (1957).

(15) A. M. Aguiar, J. Beisler, and A. Mills, *J. Org. Chem.*, **27**, 1001 (1962).

(16) A. M. Aguiar, J. Giacin, and H. J. Greenberg, *ibid.*, **28**, 3545 (1963).

(17) W. C. Davies, P. L. Pearse, and W. J. Jones, *J. Chem. Soc.*, 1262 (1929).

(18) The σ^P value of the *t*-butyl group can be estimated from the ^{31}P chemical shift of *t*-butyltriphenylphosphonium iodide by use of eq 1 of ref 11, and the σ^P value can be used to calculate the expected chemical shift of *t*-butyldiphenylphosphine by use of eq 1 of ref 3.

(19) K. Moedritzer, L. Maier, and L. C. D. Groenweghe, *J. Chem. Eng. Data*, **7**, 307 (1962).

(20) L. Maier, "Progress in Inorganic Chemistry," Vol. 5, F. A. Cotton, Ed., John Wiley and Sons, Inc., New York, N. Y., 1963, p 57.

TABLE II

GROUP CONTRIBUTIONS TO THE ^{31}P CHEMICAL SHIFTS OF TERTIARY PHOSPHINES

Group	Group contribution	σ^P
Methyl	21	0
Neopentyl	18	3
Isobutyl	15	6
n-Propyl	11	10
n-Butyl	11	10
Ethyl	7	14
Cyclopentyl	0	21
Cyclohexyl	−2	23
sec-Butyl	−3	24
Isopropyl	−6	27
t-Amyl	−21	42
t-Butyl	−23	44
Benzyl	4	17
Phenyl	3	18
n-C_nH_{2n+1}, $n \geq 3$	11	10

TABLE III

^{31}P CHEMICAL SHIFTS (PPM *vs.* 85% H_3PO_4) OF TERTIARY PHOSPHINES

Phosphine	Δ (obsd)	Δ (calcd)	Phosphine	Δ (obsd)	Δ (calcd)
Me_3P	62[a]	63	i-Bu_2PhP	34.2	33
Me_2PhP	46[a]	45	i-$BuPh_2P$	21	21
Me_2EtP	48[a]	49	*sec*-Bu_2PhP	−1.8	−3
$MePh_2P$	26[a]	27	*sec*-$BuPh_2P$	3.2	3
$MeEt_2P$	34[a]	35	t-$BuPh_2P$	−17.1	−17
Et_3P	20.4[a]	21	t-$AmPh_2P$	−15.4	−15
Et_2PhP	16[a]	17	c-Pe_3P	−0.8	0
$EtPh_2P$	12[a]	13	c-Pe_2PhP	−1.6	3
n-Pr_3P	33[a]	33	c-$PePh_2P$	3.9	6
n-Pr_2PhP	27.7	25	*neo*-$PePh_2P$	23.9	24
n-$PrPh_2P$	17.6	17	c-Hx_3P	−7[a]	−6
i-Pr_3P	−19.4[a]	−18	c-Hx_2PhP	−2.5[a]	−1
i-Pr_2PhP	−10[a]	−9	c-$HxPh_2P$	4.4[a]	4
i-$PrPh_2P$	−0.2[a]	0	Bz_3P	10.4	12
n-Bu_3P	32.3[a]	33	Bz_2PhP	12.1	11
n-Bu_2PhP	26.2[a]	25	$BzPh_2P$	10.4	10
n-$BuPh_2P$	17.1[a]	17	Vinyl-Ph_2P	11.7	...
i-Bu_3P	40	45	Allyl-Ph_2P	17.1	...

[a] Cited in ref 3.

a tertiary phosphine predicts the phosphorus-31 chemical shift, which can be compared with the experimental values listed in Table III. The relationship between the group contribution and the σ^P values originally reported is given by eq 1.

$$GC = 21 - \sigma^P \qquad (1)$$

The σ^P for benzyl was reported by Maier[21] in a study of primary phosphines and that value is confirmed by agreement with our three phosphines containing the benzyl group. The ^{31}P chemical shifts of the primary phosphines, methyl-, ethyl-, *n*-butyl, isobutyl-, and *t*-butylphosphine,[22] are in the same order as our group contributions, as would be expected from the relationship given by Maier.[21]

Discussion

Two cases immediately arise which cannot be conveniently explained by a and b of possible correlations outlined within the introduction. If the bond angle explanation is the major influence on the chemical shift, then one would expect a bulky group such as neopentyl to have a group contribution close to or between isopropyl and *t*-butyl. Instead of a low group contribution like these two, neopentyl has almost as large a group contribution as methyl, which is at the opposite end of the series!

Secondly, if electronegativity is a major influence, then propyl and ethyl are inverted in the experimental series Me > Pr > Et > *i*-Pr > *t*-Bu. This is true no matter which end of the series is considered the most electronegative. The same difficulty arises for the isopropyl and *sec*-butyl groups, and certainly neopentyl is again far out of line in the experimental series compared to its position in the electronegativity series.

Using concept c we can correctly predict the group contribution for all the alkyl groups listed and hopefully for all the remaining ones. Substitution of one methyl group on the α carbon of the alkyl group decreases the group contribution by roughly 14 ppm (Table IV). This corresponds to a loss of a hyperconjugative hydrogen and a gain of a methyl group. The gain of a methyl group (inductive effect, upfield shift) is not sufficient to overcome the loss of the α hydrogen (downfield shift) and the resulting shift is downfield. Substitution on a β carbon results in only an upfield inductive shift of about 3 ppm (Table IV). Substitution farther out on the alkyl group does not noticeable affect the group contribution. This was mentioned recently by Maier[21] and his value of σ^P for large normal alkyl groups is incorporated in Table II.

TABLE IV

CHANGES OF GROUP CONTRIBUTIONS BY METHYL SUBSTITUTION

Group	α-Me-substituted group	Difference
Me (21)	Et (7)	−14
Et (7)	*i*-Pr (−6)	−13
i-Pr (−6)	*t*-Bu (−23)	−17
n-Pr (11)	*s*-Bu (−3)	−14
s-Bu (−3)	*t*-Am (−21)	−18
Group	β-Me-substituted group	Difference
Et (7)	Pr (11)	+4
Pr (11)	*i*-Bu (15)	+4
i-Bu (15)	neo-Pe (18)	+3
i-Pr (−6)	*s*-Bu (−3)	+3
s-Bu (−3)	*c*-Pe (0)	+3
t-Bu (−23)	*t*-Am (−21)	+2

Thus, the group contribution of an alkyl group to the chemical shift of a tertiary phosphine can be calculated from the equation involving only the number of β and γ carbon atoms of the group.

$$GC = 21 - 14\beta_C + 3\gamma_C \qquad (2)$$

Related to this problem is an interesting plot (Figure 1) of the group contribution of the alkyl group *vs.* the number of α minus the number of β hydrogens of the alkyl group. The following reasoning shows how this plot is related to eq 2. The equation for the line in the graph is

$$\alpha_H - \beta_H = (2/7)GC - 3 \qquad (3)$$

Substitution of eq 2 into 3 gives

$$\alpha_H - \beta_H = 2/7(21 - 14\beta_C + 3\gamma_C) - 3$$

$$\alpha_H - \beta_H = 3 - 4\beta_C + 6/7\gamma_C \qquad (4)$$

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[21] L. Maier, *Helv. Chim. Acta*, **49**, 1718 (1966).

[22] H. R. Hays and T. J. Logan, Abstracts of Papers, 152nd National Meeting of the American Chemical Society, New York, N. Y., 1966, p O-81; H. R. Hays, private communication.

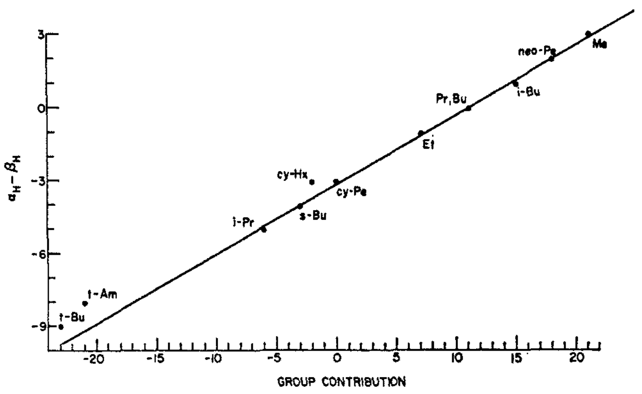


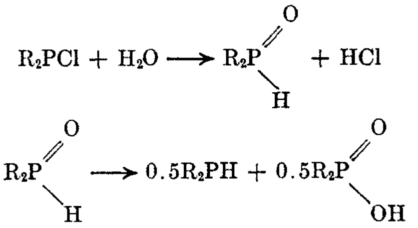
Figure 1.—The number of $\alpha - \beta$ hydrogens of an alkyl group *vs.* the group contribution of the alkyl group to the ^{31}P chemical shift of a tertiary phosphine.

The actual relationship between ($\alpha_H - \beta_H$) and β and γ carbons of an alkyl group is

$$\alpha_H - \beta_H = 3 - 4\beta_C + \gamma_C$$

which agrees substantially with eq 4.

The formation of diphenylphosphine (see the Experimental Section) in the preparation of *t*-butyldiphenylphosphine is also of interest. A similar product was observed in a recent report[23] on the preparation of various *t*-butylphosphines and it was suggested that the secondary phosphine arises by disproportionation of the phosphinous acid formed on hydrolysis of unreacted phosphorus halide, *viz.*



$$R_2PCl + H_2O \longrightarrow R_2P(=O)H + HCl$$

$$R_2P(=O)H \longrightarrow 0.5R_2PH + 0.5R_2P(=O)OH$$

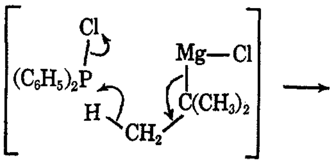
or possibly by hydrolysis of a magnesium phosphide which forms by halogen-metal exchange.

$$R_2PCl + t\text{-BuMgX} \longrightarrow R_2PMgX + t\text{-BuCl}$$

$$R_2PMgX + H_2O \longrightarrow R_2PH + 0.5MgX_2 + 0.5Mg(OH)_2$$

Our results show that isobutylene is given off in the reaction only during the addition of chlorodiphenylphosphine to the *t*-butyl Grignard reagent and not during the preparation of the Grignard reagent nor during the hydrolysis of the reaction mixture. Hence, it is strongly suggested that the secondary phosphine arises by partial reduction of the chlorophosphine with *t*-butyl Grignard reagent.

$$(C_6H_5)_2PCl + (CH_3)_3CMgCl \longrightarrow$$



$$(C_6H_5)_2PH + CH_2{=}C(CH_3)_2 + MgCl_2$$

Registry No.—Neo-PePh$_2$P, 7660-85-7; *i*-BuPh$_2$P, 5952-47-6; *i*-Bu$_2$PhP, 7650-78-4; *sec*-BuPh$_2$P, 7650-79-5; *sec*-Bu$_2$PhP, 7650-80-8; *c*-PePh$_2$P, 7650-81-9; *c*-Pe$_2$PhP, 7650-82-0; *n*-PrPhP, 7650-83-1; *n*-PrPh$_2$P, 7650-84-2; *i*-Bu$_3$P, 4125-25-1; *t*-BuPh$_2$P, 6002-34-2; *t*-AmPh$_2$P, 7650-87-5; *c*-Pe$_3$P, 7650-88-6; Bz$_3$P, 7650-89-7; Bz$_2$PhP, 7650-90-0; BzPh$_2$P, 7650-91-1; vinyl Ph$_2$P, 2155-96-6; allyl Ph$_2$P, 2741-38-0.

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The Synthesis of Substituted 1,8-Diphenylnaphthalenes[1]

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1-(3-Substituted phenyl)naphthalene **10–12** and 1,8-bis(3-substituted phenyl)naphthalene derivatives **5**, **14**, and **15** have been prepared from the appropriate tetralone and octalone precursors. Since only a single stereoisomer of each of the 1,8-diarylnaphthalenes was isolated, no definitive information was obtained concerning the barrier to rotation about the arylnaphthalene bonds. Nitration of either 1-phenylnaphthalene (**20**) or 1,8-diphenylnaphthalene (**6**) yielded the corresponding 4-nitro derivatives (**21** and **22**). Carbonation of the anion radicals derived from these two hydrocarbons (**6** and **20**) yielded the diacids (**25a**, **26a**, and **27a**) accompanied by the starting hydrocarbons (**6** and **20**).

To continue our study of the 1,8-diphenylnaphthalene system,[2,3] we wished to find synthetic routes to derivatives in which the phenyl rings carried substituents. It is our plan to use such derivatives to test the hypotheses[2b] that a substantial energy barrier exists to free rotation of the phenyl rings and that substituents in one phenyl ring may transmit their electronic influence to the second phenyl ring. Since the two

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(1) This work has been supported by research grants from the National Institutes of Health (Grant No. GM-08761) and the National Science Foundation (Grant No. GP-5685).

(2) (a) H. O. House and H. W. Thompson, *J. Org. Chem.*, **28**, 360 (1963); (b) H. O. House, R. W. Magin, and H. W. Thompson, *ibid.*, **28**, 2403 (1963); (c) H. O. House and R. W. Bashe, II, *ibid.*, **30**, 2942 (1965).

(3) For other studies and discussion of this system and relatives, see (a) A. S. Bailey, G. A. Dale, A. J. Shuttleworth, and D. P. Weizmann, *J. Chem. Soc.*, 5110 (1964); (b) E. D. Bergmann, S. Blumberg, P. Bracha, and S. Epstein, *Tetrahedron*, **20**, 195 (1964); (c) C. Dufraisse and Y. Lepage, *Compt. Rend.*, **258**, 1507, 5447 (1964); (d) Y. Lepage and O. Pouchot, *Bull. Soc. Chim. France*, 2342 (1965); (e) S. C. Dickerman, D. deSouza, and P. Wolf, *J. Org. Chem.*, **30**, 1981 (1965); (f) H. H. Jaffé and O. Chalvet, *J. Am. Chem. Soc.*, **85**, 1561 (1963); (g) E. Harnik, F. H. Herbstein, G. M. J. Schmidt, and F. L. Hirshfeld, *J. Chem. Soc.*, 3288 (1954); (h) M. Rabinovitz, I. Agranat, and E. D. Bergmann, *Tetrahedron Letters*, 4133 (1965); (i) Z. A. Akopyan, R. L. Aboyan, and Yu. T. Struchkov, *Zh. Struk. Khim.*, **3**, 602 (1962); (j) V. Balasubramaniyan, *Chem. Rev.*, **66**, 567 (1966).