method of Londergan.[30] The 3-hydroxybutanone was from Distillation Products. The only recognizable product obtained from treating 3-hydroxybutanone in the manner described in the previous cases was recovered 2,4-dinitrophenylhydrazine. Treating 5-hydroxypentan-2-one in the same way gave a black viscous fluid which resisted attempts at working up.

The Products A, B, F, and G.—These were shown to be identical by their infrared spectra. These spectra had a strong, sharp carbonyl band in the region 1727–1736 cm.$^{-1}$. The ultraviolet spectra were also identical and had maxima at 263–264 and 349–350 mμ in ethanol; maxima at 267 and 361 mμ and a shoulder at 340 mμ in chloroform.

The Products C, E, D, and H.—These were shown to be identical by their infrared spectra. A strong carbonyl band appeared in the region 1727–1730 cm.$^{-1}$. The ultraviolet spectra had the same characteristics as those of the products A, B, F, and G.

Spectra.—The infrared spectra were obtained in potassium bromide pellets on a Perkin-Elmer Infracord, Model 137. The ultraviolet spectra were obtained with a Beckman Model DK-2 recording spectrophotometer. The n.m.r. spectra were obtained with a Varian Associates Model A-60 spectrometer. The solvents, internal standard, and instrumental conditions are noted on the spectra.

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[30] T. E. Londergan, N. L. Hause, and W. R. Schmidz, *J. Am. Chem. Soc.*, **75**, 4456 (1953).

Hydrogenolyses of Aromatic Halides with Triaryltin Hydrides[1]

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Electronic and solvent effects have been evaluated for the hydrogenolysis of aromatic halides with triaryltin hydrides. For the aromatic halides, electron-releasing groups inhibit the reaction while electron-attracting groups accelerate the reaction. Hydroxylic solvents speed the reaction and triphenylboron accelerates the reaction more in hydroxylic solvents than in hydrocarbon solvents. Consistent with these data is a heterolytic reaction mechanism, but not necessarily SN1 or SN2. New triaryltin hydrides described are tris(*p*-tolyl)tin, tris(*p*-fluorophenyl)tin, and trimesityltin hydrides.

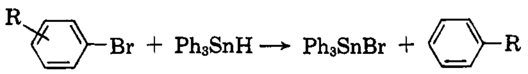
A number of workers have recently reported on the hydrogenolyses of alkyl,[5–9,11] aryl,[5,7,10] and acyl[12] halides with organotin hydrides. It has been shown that triphenyltin hydride is an effective hydrogenolysis agent[5–8,10] and that the ease of replacement of the halogens[7,10] is iodine > bromine > chlorine. It has also been demonstrated that aromatic hydrogenolysis is decreased by electron-donating groups in the *para* position.[10] We would now like to report on a more detailed study of the effect of substituents and solvents on the hydrogenolysis of aromatic halides.

A series of substituted bromobenzenes and halonaphthalenes was treated with triaryltin hydrides and in each case the bromine was removed from the benzene ring or the halogen from the halonaphthalene and triaryltin halide was formed. The extent of hydrogenolysis was determined by titration of the triaryltin halide formed with base (Tables I, II, III, and IV). Tables I and II show that the reaction of triphenyltin hydride and tri(*p*-tolyl)tin hydride with the substituted bromobenzenes at 154° proceeded nearly to completion, affording yields between 85–100%.

At 124°, the reaction showed a greater selectivity, giving lower yields with compounds substituted with electron-donating groups in the *ortho* and *para* positions and increased yields with compounds substituted with electron-withdrawing groups. The increased yields of the *o*- and *m*-anisyl bromides may be due to the inductive effect of the methoxyl group. The increased reactivity of the phenyl substituted bromobenzenes cannot be explained on the basis of their electron-withdrawing ability. On the basis of electron withdrawal of the nitrile group a much higher yield

TABLE I

HYDROGENOLYSES OF AROMATIC HALIDES



R			% Yield		
ortho	*meta*	*para*	Temp., °C.: 94	124	154
CH_3	...	...	10	52	...
...	CH_3	...	9	34	...
...	...	CH_3	10	50	91
OCH_3	...	...	12	60	...
...	OCH_3	...	5	43	...
...	...	OCH_3	7	34	90
H	H	H	12	55	90
F	...	...	14	78	...
...	F	...	17	85	...
...	...	F	9	71	...
Cl	...	...	39	96	...
...	Cl	...	38	88	100.5
...	...	Cl	16	82	97
Ph	...	...	16	94	...
...	Ph	...	8	80	...
...	...	Ph	22	90	95
CF_3	...	...	61	98	...
...	CF_3	...	21	80	...
...	...	CF_3	13	79	...
...	...	CN	20	59	
Iodobenzene			84	90	100

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[1] Supported by the U. S. Army Research Office (Durham), under contract DA-30-069-ORD-2851 and grant DA-ORD-31-124-61-G39. We gratefully acknowledge this generous support.

[2] Taken from portions of the dissertations submitted to the Faculty of the Polytechnic Institute of Brooklyn in partial fulfillment of the requirements for the degree of Doctor of Philosophy (Chemistry) (D. H. L., 1963, and A. S., 1964).

[3] Taken from part of the thesis submitted to the Faculty of the Polytechnic Institute of Brooklyn in partial fulfillment of the requirements for the degree of Bachelor of Science (Chemistry), 1962.

[4] To whom inquiries should be sent.

[5] J. G. Noltes and G. J. M. van der Kerk, *Chem. Ind.*, **9**, 294 (1959).

[6] E. J. Kupchik and R. E. Connolly, *J. Org. Chem.*, **26**, 4747 (1961).

[7] H. G. Kuivila, L. W. Menapace, and C. R. Warner, *J. Am. Chem. Soc.*, **84**, 3584 (1962).

[8] D. H. Lorenz and E. I. Becker, *J. Org. Chem.*, **27**, 3370 (1962).

[9] E. J. Kupchik and R. J. Kiesel, *Chem. Ind.*, No. **37**, 1654 (1962).

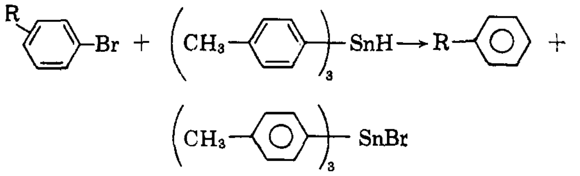
[10] L. A. Rothman and E. I. Becker, *J. Org. Chem.*, **24**, 294 (1959); **25**, 2203 (1960).

[11] E. J. Kupchik and T. Lanigan, *ibid.*, **27**, 3661 (1962).

[12] H. G. Kuivila, *ibid.*, **25**, 284 (1960), and earlier references cited therein.

TABLE II

HYDROGENOLYSES OF AROMATIC HALIDES



R			% Yield	
ortho	*meta*	*para*	Temp., °C.: 124	154
CH_3	...	...	36	85
...	CH_3	...	41	100
...	...	CH_3	39	99
OCH_3	...	...	46	98
...	OCH_3	...	37	97
...	...	OCH_3	28	96
H	H	H	39	92
F	...	...	67	87
...	F	...	78	99
...	...	F	48	100
Cl	...	...	86	97
...	Cl	...	84	100
...	...	Cl	57	100
Ph	...	...	89	100
...	Ph	...	80	90
...	...	Ph	76	99
CF_3	...	...	92	106
...	CF_3	...	76	99
...	...	CF_3	73	88
Iodobenzene			100	100

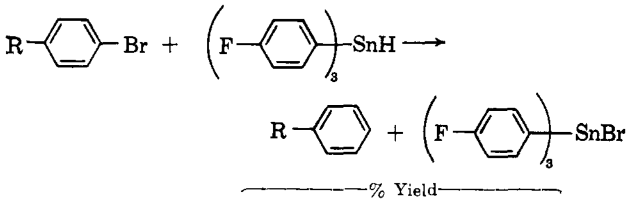
was expected for *p*-bromobenzonitrile. However, it was found that nitriles catalyze the decomposition of triphenyltin hydride at elevated temperatures. The greater than 100% yield from *o*-trifluoromethylbromobenzene may be due to attack of the trifluoromethyl group by triaryltin hydrides. Attempted isolation of triphenyltin fluoride from this reaction as well as from the reaction with benzotrifluoride itself was unsuccessful.

A comparison of yields at 94° (Table I) shows the reaction to be much less complete than at the two higher temperatures. At this low temperature only iodobenzene and the compounds substituted with strong electron-withdrawing groups were significantly hydrogenolysed.

The yields in the hydrogenolyses with tris(*p*-fluorophenyl)tin hydride (Table III) were less dependent on the aromatic halide. Both electron-withdrawing and electron-donating substituents increased the extent of hydrogenolysis. Even at 154° the hydrogenolysis reactions were not completed. This behavior may be due to the relative thermal instability of this hydride.

TABLE III

HYDROGENOLYSIS OF AROMATIC HALIDES

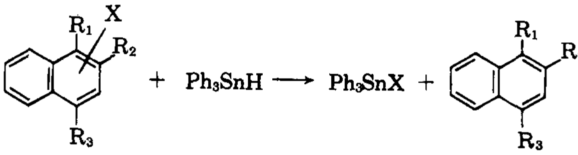


R *para*	% Yield Temp., °C.: 124	154
CH_3	20	79
OCH_3	22	82
H	16	67
F	32	73
Ph	38	96
Cl	33	89
CF_3	47	89

The hydrogenolysis of naphthalene halides (Table IV) again demonstrates that the ease of removal of the halogens is in the order I > Br > Cl. It is a well known fact that the 1-position is the preferred position for electrophilic attack in the naphthalene series.[13] Conversely, the 2-position is the preferred position for nucleophilic attack. It was, therefore, expected that 2-bromonaphthalene should be hydrogenolyzed much faster than 1-bromonaphthalene at 94°. However, 1-iodonaphthalene was found to react faster than 2-iodonaphthalene at 94°. This may be explained by the fact that the carbon–iodine bond in the 1-position is a weaker one than that of the 2-position, due to the steric interaction of the iodine atom with the hydrogen atom in the 8-position. The preferred hydrogenolysis of the 1-position thus relieves the steric strain. It is also noteworthy that the selectivity of nucleophilic attack decreases with increasing temperature. The increased reactivity of 4-bromo-1-methylnaphthalene *vs.* 1-bromo-2-methylnaphthalene probably results from the steric hindrance provided by the *ortho* methyl group to attack by the hydride ion and, perhaps also, from the electron-donating inductive effect provided by the *ortho* methyl group.

TABLE IV

HYDROGENOLYSIS OF AROMATIC HALIDES



	% Yield		
Ph_3SnH/ArX, 1.5	Temp., °C.: 94°	124°	154°
X = 1-iodo, $R_1R_2R_3$ = H	97.6	99.7	100.0
X = 2-iodo, $R_1R_2R_3$ = H	87.0	99.0	99.5
X = 1-bromo, $R_1R_2R_3$ = H	11.5	79.1	98.5
X = 2-bromo, $R_1R_2R_3$ = H	66.2	85.4	95.4
X = 1-chloro, $R_1R_2R_3$ = H	N.R.[a]	N.R.[a]	4.47
X = 2-chloro, $R_1R_2R_3$ = H	N.R.[a]	N.R.[a]	3.36
X = 1-bromo, R_1R_2 = H $R_3 = CH_3$	6.69	80.3	100.0
X = 1-bromo, R_1R_3 = H $R_2 = CH_3$	4.50	18.9	98.9

[a] No reaction.

While chlorobenzene and fluorobenzene were found to be unreactive to hydrogenolysis under normal conditions,[10] it was found that *o*- or *p*-dichlorobenzene gave triphenyltin chloride in 20% yield on treatment with triphenyltin hydride. Shapiro[14] also has shown that the difluorobenzenes are hydrogenolyzed by triphenyltin hydride (1–3%) and that hydrogenolysis of hexafluorobenzene produced triphenyltin fluoride and pentafluorobenzene in 19% yield.

There are two general types of mechanism which would be consistent with the observed electronic effect of substituents on the aryl bromide: (1) a radical

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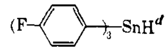
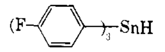
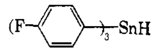
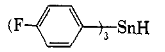
(13) B. Gething, C. R. Patrick, and J. C. Tatlow, *J. Chem. Soc.*, 186 (1962).

(14) P. Shapiro, B.S. thesis, Polytechnic Institute of Brooklyn, 1962.

TABLE V

EFFECT OF CATALYSTS ON THE HYDROGENOLYSIS OF 4-BROMOBIPHENYL[a]

(Temp., 124°)

Ar_3SnH	Catalyst	Moles	Solvent[b]	Reaction time, hr.	% Yield[c]
	. . .	. . .	Xylene[e]	2.5	8.4
	Azobis[f]	5×10^{-6}	Xylene	2.5	52.4
	. . .	. . .	Xylene	3.3	12.3
	Bz_2O_2[g]	5×10^{-6}	Xylene	3.3	24.9
Ph_3SnH[d]	. . .	. . .	Xylene	3	11.0
Ph_3SnH	Azobis	1×10^{-5}	Xylene	3	59.0
Ph_3SnH	Bz_2O_2	3×10^{-6}	Xylene	3	26.5
Ph_3SnH	Hydroquinone	3×10^{-5}	Xylene	3	9.1
Ph_3SnH	Ph_3B[h]	1×10^{-5}	Xylene	3	15.0
Ph_3SnH	Ph_3B	1×10^{-4}	Xylene	3	15.9
Ph_3SnH	. . .	. . .	Diglyme[i]	3	41.0
Ph_3SnH	Azobis	1×10^{-5}	Diglyme	3	84.3
Ph_3SnH	Hydroquinone	9×10^{-5}	Diglyme	3	42.1
Ph_3SnH	Ph_3B	3×10^{-5}	Diglyme	3	54.0
Ph_3SnH	. . .	. . .	Diglyme	20	50.0
Ph_3SnH	Hydroquinone	9×10^{-5}	Diglyme	20	59.5
Ph_3SnH	Ph_3B	13×10^{-5}	Diglyme	20	82.6

[a] 2.2×10^{-3} moles. [b] 5 ml. [c] Determined by titration. [d] 3×10^{-3} moles. [e] Dielectric constant 2.27–2.57 (20°) [A. A. Maryott and E. R. Smith, "Table of Dielectric Constants of Pure Liquids," National Bureau of Standards, Circular 514, 1951. [f] 2,2-Azobis[2-methylpropionitrile]. [g] Benzoyl peroxide. [h] Blank determined and subtracted. [i] Dielectric constant 5.79 [J. L. Down, J. Lewis, B. Moore, and G. Wilkinson, *J. Chem. Soc.*, 3767 (1959)].

chain mechanism or (2) an aromatic displacement reaction, either ionic or of the four-center type. Homolytic reactions are known in which the substituents, both electron donating and electron-withdrawing, increase the rate of reaction. Therefore, the observed electronic effects would favor a heterolytic rather than a homolytic reaction.

Kuivila, Menapace, and Warner[7] have shown that the mechanism of hydrogenolysis of alkyl halides cannot be of SN1 or SN2 type when using tributyltin hydride. Kupchik and Kiesel,[9] working with 1-bromonorbornane, also showed that an SN2 mechanism was unlikely.

Very importantly, Kuivila, *et al.*, have shown that addition of azobisisobutyronitrile increased the yields in the hydrogenolysis of bromobenzene with tri-*n*-butyltin hydride. We also have observed increased yields upon addition of azobisisobutyronitrile, or of benzoyl peroxide, in the hydrogenolysis of 4-bromobiphenyl with triphenyltin hydride or with tris(*p*-fluorophenyl)tin hydride (Table V).

However, hydroquinone does not significantly decrease yields in the hydrogenolysis of 4-bromobiphenyl (Table V) and, in long period reactions, actually increased the yield of triaryltin bromide. This behavior of hydroquinone suggests that either the normal hydrogenolysis reaction does not involve a radical chain mechanism or that hydroquinone does not act as an inhibitor in this type of reaction. Rothman[15] also has reported that similar yields of triphenyltin bromide were obtained from the reduction of bromobenzene when the reaction was heated for two, six, or thirteen hours, or when either triphenyltin hydride or bromobenzene was present in excess, or when the reaction was conducted in the dark.

Further observations made here are that catalytic quantities of triphenylboron, a Lewis acid, increase yields and that it is more effective in diglyme than in xylene.

Furthermore, the yield increases with the dielectric constant of the solvent. In the case of trimesityltin hydride, the yields went from 7% (dioxane) to 20% (pyridine) and 22% (butanol) (see Table V) and, in the series with triphenyltin hydride, from 11–12% (xylene) to 41% (diglyme) (see Table VI). While not yet as inclusive as they might be, the solvent effects noted are self-consistent for two different hydrides and for six solvents.

TABLE VI

EFFECTS OF SOLVENT ON HYDROGENOLYSIS OF IODOBENZENE WITH TRIMESITYLTIN HYDRIDE[a]

Solvent[b]	Dielectric constant[c]	Reaction time, hr.	Temp., °C.	% Yield[d]
p-Dioxane	2.209 (25°)	22.5	100	7.2
Mesitylene	2.27 (20°)	48	150	17.0
Pyridine	12.3 (25°) 9.4 (116°)	22.5	100	20
n-Butyl alcohol	17.8 (20°) 8.2 (118°)	22.5	100	22

[a] Mole ratio of halide/hydride, 1:1. [b] 5 ml. [c] Footnote *e*, Table V. [d] Determined by titration.

In view of the electronic effects, the noninhibition by hydroquinone and the solvent effects, it is suggested that the uncatalyzed hydrogenolysis reaction of aromatic halides with triaryltin hydrides is more a heterolytic than a homolytic reaction, and quite probably of the four-center type. Lastly, carbon–halogen

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[15] L. A. Rothman, Ph.D. thesis, Polytechnic Institute of Brooklyn, 1960.

bond breakage must be involved in the rate-determining step in order to explain the order of removal of the halogens,[16] which, in this case, is the reverse of other nucleophilic aromatic substitution reactions.

Experimental

Materials.—All organic halides used in the hydrogenolyses reactions were purified using standard procedures. Melting points, refractive indices, and vapor phase chromatographs, where applicable, were used to check the purity.

Tris-*p*-tolyltin Hydride.—Tris(*p*-tolyl)tin chloride[17] (21.4 g., 0.050 mole) dissolved in 250 ml. of ether was added slowly to 2.0 g. (0.053 mole) of lithium aluminum hydride suspended in 150 ml. of ether contained in a three-necked flask equipped with stirrer, reflux condenser, nitrogen inlet tube, and addition funnel. After addition was completed, the reaction mixture was refluxed for 2.5 hr. and stirred for an additional half hour without heating. The reaction mixture was cooled in an ice bath and 10 ml. of water was cautiously added. A solution of 40 g. of sodium potassium tartrate in 160 ml. of water was added, the ether layer separated, and the aqueous layer washed with additional ether. The combined ether fractions were then washed with water and dried over anhydrous sodium sulfate.

The ether solution was then distilled at reduced pressure leaving a white fluffy solid, m.p. 81.8–84.4°. Recrystallization from methanol-water gave 17 g. (88%) of white needles melting at 82.3–84.9°.

Anal. Calcd. for $C_{21}H_{22}Sn$: C, 64.16; H, 5.64; Sn, 30.20. Found: C, 64.24; H, 6.03; Sn, 30.19.

Tris(*p*-fluorophenyl)tin Chloride.—Tetrakis(*p*-fluorophenyl)tin was prepared by the method of Fuchs and Post[18] and purified by recrystallization from hexane, m.p. 144–145°.

Stannic chloride (8.1 g., 0.032 mole) was added to 42.8 g. (0.097 mole) of tetrakis(*p*-fluorophenyl)tin contained in a round-bottomed flask equipped with reflux condenser and magnetic stirrer. The reaction mixture was heated between 190–200° for 3 hr. and on cooling an oil remained which crystallized upon addition of hexane. Two recrystallizations from hexane and cyclohexane yielded 33.6 g. (60%) of product, m.p. 118.2–120.5°.

Anal. Calcd. for $C_{18}H_{12}ClFSn$: C, 49.20; H, 2.75; Cl, 8.07; Sn, 27.01. Found: C, 49.42; H, 2.80; Cl, 8.10; Sn, 26.84.

Tris(*p*-fluorophenyl)tin Hydride.—A solution of 17 g. (0.039 mole) of tris(*p*-fluorophenyl)tin chloride in 300 ml. of ether was added over a period of half an hour to 5 g. (0.13 mole) of lithium aluminum hydride suspended in 200 ml. of ether in a three-necked flask equipped with reflux condenser, stirrer, nitrogen inlet tube, and addition funnel. After addition was completed, the reaction mixture was stirred at room temperature for half an hour and then, while stirring was continued, cooled in an ice bath for an additional half hour. Water was cautiously added until there was no evidence of gas evolution, whereupon a solution of 40 g. of sodium potassium tartrate in 100 ml. of water was added. The ether layer was separated, and the aqueous layer washed with additional ether. The combined ether fractions were then washed with water and finally dried over anhydrous sodium sulfate.

The ether solution was then distilled at reduced pressure, leaving a white, highly viscous oil. Extraction of the oil with pentane left behind a white solid, m.p. 219–222°.

The pentane solution was distilled at reduced pressure leaving an oily white solid. Recrystallization from ethanol-water gave a product, m.p. 47.7–49.1°. Sublimation of this material at 40° and 1-mm. pressure yielded 5.3 g. (33%) of white product, m.p. 48.7–50.4°.

Anal. Calcd. for $C_{18}H_{13}F_3Sn$: C, 53.38, H, 3.24; Sn, 29.31. Found: C, 53.33; H, 3.30; Sn, 29.20.

The solid, m.p. 219–222°, was recrystallized from cyclohexane to give 7.7 g. (50%) of hexakis(*p*-fluorophenyl)ditin, melting at 221–222.7°.

Anal. Calcd. for $C_{36}H_{24}F_6Sn_2$: C, 53.51; H, 2.99; Sn, 29.38. Found: C, 53.49; H, 3.01; Sn, 29.30.

Trimesityltin Hydride.—To 2.0 g. (0.053 mole) of lithium aluminum hydride suspended in 50 ml. of anhydrous ether was added dropwise 8.0 g. (0.014 mole) of trimesityltin bromide[19] in 100 ml. of anhydrous ether. Upon completion of addition, the reaction mixture was refluxed under a nitrogen atmosphere for 3 hr. The reaction was allowed to cool, decomposed by the careful addition of water, separated, washed with several portions of a dilute sodium potassium tartrate solution, followed by two portions of water. The ether solution was then dried overnight over anhydrous sodium sulfate, filtered, and distilled leaving a nearly white solid. One recrystallization from cyclohexane afforded 5.0 g. (75% yield) of a white crystalline material, m.p. 176–178° (evacuated melting point capillary). Recrystallization from tetrahydrofuran–methanol gave the analytical sample, m.p. 177.7–179.0°.

Anal. Calcd. for $C_{27}H_{34}Sn$: C, 67.95; H, 7.18. Found: C, 68.17; H, 7.05.

Hydrogenolyses Reactions.—All hydrogenolyses reactions were run under comparable conditions. Weighed amounts of aromatic halide and triaryltin hydride, in a mole ratio of 1 to 1.36, were placed in a ground glass stoppered test tube and the tube was heated in an oil bath which was maintained to within 1.0°. After 2 hr. the reaction mixture was extracted from the reaction vessel with the titration solvent (50 ml. of ethanol, 50 ml. of benzene), and then titrated with 0.2 *N* aqueous sodium hydroxide, using thymol blue as indicator.

Analytical Determination of Triaryltin Halides.—Accurately weighed samples (0.1–0.4 g.) of triaryltin halides were dissolved in 100 ml. of a 1:1 benzene–ethanol solution and titrated with aqueous sodium hydroxide (0.200 *N*) to a Thymol Blue end point (yellow to blue). The weights determined by titration agreed with the weights of triaryltin halides (except for triphenyltin fluoride) taken, to 0.1–0.4% error.

Reaction of Triphenyltin Hydride with *p*-Dichlorobenzene.—A solution of 1.15 g. (7.8 mmoles) of *p*-dichlorobenzene and 9.7 g. (28 mmoles) of triphenyltin hydride in a round-bottom flask equipped with reflux condenser was heated at 165° for 4 hr. The reaction mixture was cooled and extracted with the titration solvent (100 ml. of methanol, 20 ml. of chloroform, and 50 ml. of water). Titration with 0.200 *N* aqueous sodium hydroxide to a phenolphthalein end point required 10.30 ml. (24.5%).

Reaction of Triphenyltin Hydride with *o*-Dichlorobenzene.—Using the procedure of the preceding paragraph, but starting with 0.200 g. (1.37 mmoles) of *o*-dichlorobenzene and 2.00 g. (5.7 mmoles) of triphenyltin hydride and a heating time of 8 hr., titration with the 0.200 *N* base required 1.59 ml. (23.2%).

Reaction of Triphenyltin Hydride with Fluorobenzenes.[3]—Known weights of triphenyltin hydride and the fluorobenzene were weighed into a necked-down, thick-walled Pyrex tube. The tubes were cooled to −80° and sealed under vacuum. After warming to room temperature, they were heated in an oil bath at 150° for 90–100 hr. After cooling to room temperature, the tubes were opened and extracted with a 1:1 ethanol–benzene solution. To this solution was added 1 ml. of 1.0% phenolphthalein (in ethanol) and a 200% excess of standard sodium hydroxide, as well as enough water to form a two-phase system. This mixture was then back-titrated with standard hydrochloric acid to the phenolphthalein end point.

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(16) G. S. Hammond and L. R. Parks, *J. Am. Chem. Soc.*, **77**, 340 (1955).

(17) (a) E. Krause and R. Becker, *Ber.*, **53**, 173 (1920); (b) K. A. Kocheshkov, M. M. Nad, and A. P. Aleksandrov, *Ber.*, **67**, 1348 (1934).

(18) O. Fuchs and H. W. Post, *Rec. trav. chim.*, **78**, 566 (1959).

(19) I. I. Lapkin and V. A. Sedel'nikova, *Zh. Obshch. Khim.*, **30**, 2771 (1960).