TABLE II

ULTRAVIOLET STUDIES[a]

Compound	Neutral media maxima		Basic media maxima[b]	
H·Tyr·OH[c] (L)	λ 223 (ε 8440)	λ 275 (ε 1410)	λ 240 (ε 11,600)	λ 294 (ε 2480)
H·Tyr·OEt (L)	λ 226 (ε 9180)	λ 278 (ε 1710)	λ 245 (ε 12,700)	λ 295 (ε 2550)
H·Tyr(OBz)·OH[d] (L)	λ 225 (ε 1210)	λ 275 (ε 107)	Obscured	λ 275 (ε 128)
B·Val-Tyr·OEt (2L)[e]	λ 225 (ε 9700)	λ 277 (ε 1640)	λ 245 (ε 13,450)	λ 295 (ε 2460)
N,O-(B·Val)$_2$-Tyr·OEt (3L)	λ 220 (ε 9010)	λ 265 (ε 322)	λ 245 (ε 13,150)	λ 295 (ε 2450)
N,O-(Ac)$_2$·Tyr·OH[c] (DL)		λ 264 (ε 321)		λ 295 (ε 2180)

[a] Read on a Beckman DU spectrophotometer or a Cary II recording spectrophotometer. [b] Two drops of 2 *N* sodium hydroxide were added to 3 ml. of neutral solution in a cuvette. The peaks were read 5 min. later. [c] Mann Research Laboratories. [d] Cyclo Chemical Corp. This compound is very insoluble in water and the extinction coefficient is probably low. The relative values from neutral to basic solution are accurate, however. [e] B = *t*-Butyloxycarbonyl.

Experimental

Isolation of By-products.—To a solution of 2.18 g. (10.0 mmoles) of *t*-butyloxycarbonyl-L-valine[6] in 10 ml. of tetrahydrofuran was added 1.95 g. (10.0 mmoles, 83% purity) of N,N'-carbonyldiimidazole.[3] After 30 min., 2.09 g. (10.0 mmoles) of ethyl L-tyrosinate[5] was added and the solution was left standing over the weekend. It was then concentrated under vacuum. The residue was taken up in 50 ml. of ether, washed with 40 ml. of *N* sulfuric acid, 20 ml. of saturated aqueous sodium bicarbonate and 40 ml. of water. The ethereal layer was dried over anhydrous sodium sulfate and evaporated to dryness. Scratching solidified the residue, giving 3.73 g. (91%), m.p. 127.5–150°. One gram of this solid was dissolved in chloroform and placed on 30 g. of silica gel in a column, 3.3 cm. × 8.5 cm. The column was eluted with seven 100-ml. portions of 20% ethyl acetate–80% chloroform, then six 100-ml. portions with progressively higher percentages of ethyl acetate. Each fraction was examined by thin layer chromatography on silica gel using 30% ethyl acetate in chloroform. On development with chlorine gas and *o*-tolidine–potassium iodide reagent,[11] fraction 3 gave one spot R_f 0.62 and fraction 4 showed two spots R_f 0.38 and 0.61. Fractions 5–9 gave only one spot at 0.38 as did a pure sample of I. The other fractions did not contain any material. Fraction 4 was rechromatographed and the new fractions combined with the corresponding old. Fractions 5–9 were recrystallized from ethyl acetate-petroleum ether to give 0.55 g. (50%) of I, m.p. 140–140.5°. Fraction 3 was recrystallized from 3 ml. of isopropyl alcohol to give 0.046 g. (4.2%) of a compound, m.p. 125–126.5°, presumed to be ethyl N,O-bis(*t*-butyloxycarbonyl-L-valyl)-L-tyrosinate (II). The ratio of II to I was 1 to 8. A mixed melting point of II with an authentic sample (see below) gave no depression. Both had identical R_f's on silica gel thin layer chromatography in 30% ethyl acetate in chloroform. The ultraviolet spectra was identical to that of an authentic sample of II.

The sulfuric acid wash of the crude product from above was examined for ethyl O-(*t*-butyloxycarbonyl-L-valyl)-L-tyrosinate, this type of compound having been described[12] recently. The acidic wash was neutralized with sodium bicarbonate and quickly extracted with ether. The ethereal solution was dried over anhydrous sodium sulfate and then evaporated to dryness. An ultraviolet spectrum was taken on this residue in neutral and in basic media. Normally, compounds with a free phenolic group such as tyrosine, ethyl tyrosinate and I experience a shift in from λ278 to λ295. This is accompanied by an increase in optical density of a factor of 1.5. O-Acylated compounds, as described below, have a shift from λ 264 to λ 295 with a 7.7-fold shift in optical density. The residue had a 2.3-fold shift from λ 277 to λ 295 indicating the presence of an O-acylated phenol. The λ 264 peak being small was obscured in neutral media. Thin layer chromatography of this residue on silica gel showed the presence of an unknown that was neither starting material nor product. No trace of II was detected.

Ethyl N,O-Bis(*t*-butyloxycarbonyl-L-valyl)-L-tyrosinate (II).—After 1.57 g. (7.23 mmoles) of *t*-butyloxycarbonyl-L-valine had been dissolved in 10 ml. of tetrahydrofuran, 1.41 g. (7.23 mmoles, 83% pure) of N,N'-carbonyldiimidazole was added. Thirty minutes later 2.95 g. (7.23 mmoles) of I was added. After 1 hr. the solution was concentrated under vacuum to a clear oil. The oil was worked up 16 hr. later by dilution with 10 ml. of water and 20 ml. ether. The ethereal layer was dried over anhydrous sodium sulfate, evaporated to dryness and the residue dissolved in chloroform. This was placed on a silica gel column and eluted with 20% ethyl acetate–80% chloroform. The ultraviolet absorption of each fraction was taken at λ 265 and λ 277. When a fraction came off whose absorption was greater at λ 277 than at λ 265, it was assumed the N,O-compound (II) was off the column. The purity of the various fractions was checked by thin layer chromatography. Those fractions with the correct ultraviolet absorption and with only one spot, R_f 0.62, were combined and recrystallized from 30 ml. of isopropyl alcohol. The product crystallized and was collected, 2.05 g. (34%), m.p. 124.5–126.5°, $[\alpha]^{25}_{D}$ −35.4° ± 1.2° (*c* 4, ethanol).

Anal. Calcd. for $C_{31}H_{49}N_3O_9$: C, 61.26; H, 8.13; N, 6.91. Found: C, 61.48; H, 8.26; N, 7.09.

Proof of Structure.—A spot of I and one of II on paper were sprayed with ferric chloride in *n*-butyl alcohol and heated to 60° for 48 hr. The N,O-compound (II) gave a negative test indicating the phenolic hydroxyl was not free. The dipeptide (I) gave a positive test.

Ultraviolet spectra are described in Table II. Since phenolic esters are activated, one would expect base to convert II to I. The spectra bear this out. An ultraviolet spectrum of N,O-diacetyl-DL-tyrosine[13] gave detailed structure at the λ 264 peak (shoulder at λ 258, maximum at λ 264 and small peak at λ 272) corresponding exactly to the fine structure of the ethyl N,O-bis(*t*-butyloxycarbonyl-L-valyl)-L-tyrosinate (II) peak at λ 265.

Acknowledgment.—We thank Mr. L. Brancone and staff for analysis and Mr. W. Fulmor and staff for optical rotations.

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[11] F. Reindel and W. Hoppe, *Ber.*, **87**, 1103 (1954).

[12] J. Ramachandran, Abstracts of Papers presented at the 140th National Meeting of the American Chemical Society, Chicago, Ill., September 3–8, 1961, p. 49C.

[13] Mann Research Laboratories.

Preparation and Reactions of Trialkyltinlithium

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Recent reports from this laboratory[1b] have shown a convenient preparation for triphenyltinlithium. This organometallic can be prepared through the reaction between metallic lithium and either triphenyltin chloride or hexaphenylditin in tetrahydrofuran (THF). This paper concerns the extension of the same general synthetic procedure to prepare trialkyltinlithium compounds.

Trialkyltinlithium compounds have been prepared

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previously by Gilman and Rosenberg[2] through the reaction between the alkyllithium compound and stannous chloride. Tributyltinlithium was treated with

$$2RLi + SnCl_2 \longrightarrow R_2Sn + 2LiCl \tag{1}$$

$$R_2Sn + RLi \longrightarrow R_3SnLi \tag{2}$$

$$R = C_2H_5 \text{ and } C_4H_9$$

butyl bromide to yield tetrabutyltin (52.5%) and with iodobenzene to yield a mixture of tetrabutyltin (27.8%) and tributylphenyltin (27.6%). Recently Blake, Coates, and Tate[3] utilizing the same procedure prepared tributyltinlithium and studied the reaction between it and chlorotrimethylsilane. The expected compound, trimethylsilyltributyltin [$(CH_3)_3SiSn(C_4H_9)_3$], was not obtained. According to these investigators the reaction products obtained, butyltrimethylsilane and tetrabutyltin, indicated that the tributyltinlithium prepared according to equation 1 and 2 acted as though it were a mixture of butyllithium and dibutyltin.

In our studies on the preparation of trialkyltinlithium compounds we have found that the organometallic forms by the same mechanism suggested for the preparation of the triphenyltinlithium compound.[1b]

$$R_3SnCl + 2Li \longrightarrow R_3SnLi + LiCl \tag{3}$$

$$R_3SnLi + R_3SnCl \longrightarrow R_3SnSnR_3 + LiCl \tag{4}$$

$$R_3SnSnR_3 + 2Li \longrightarrow 2R_3SnLi \tag{5}$$

$$R = CH_3 \text{ and } C_4H_9$$

This is based on the following observations: (a) both the R_3SnCl and R_3SnSnR_3 will react with lithium in tetrahydrofuran to produce R_3SnLi, and (b) in the preparation of the organometallic from R_3SnCl, a by-product always found was the R_3SnSnR_3.

The trialkyltinlithium compounds gave a positive Color Test I[4] which is conveniently used to follow reactions of the organometallic with other reagents. Attempts to prepare the trialkyltinlithium from the trialkyltin chloride and metallic lithium in diethyl ether were unsuccessful. The trialkyltinlithium prepared in this study via equations 3, 4, and 5 undergoes some interesting reactions as seen in Table I. In no case was R_4Sn isolated, which is in contrast with the preparations mentioned above[2,3] which utilized equations 1 and 2.

TABLE I

R_3SnLi	Reactant	Product	% Yield
$(CH_3)_3SnLi^a$	C_2H_5Br	$(CH_3)_3SnC_2H_5$	59
$(CH_3)_3SnLi^a$	$(C_4H_9O)_3PO$	$(CH_3)_3SnC_4H_9$	31
$(CH_3)_3SnLi^a$	$(C_6H_5)_3SnCl$	$(C_6H_5)_3SnSn(CH_3)_3$ +	51
		$(C_6H_5)_3SnSn(C_6H_5)_3$	32
$(C_4H_9)_3SnLi^a$	H_2O	$(C_4H_9)_3SnH$ +	54
		$(C_4H_9)_3SnSn(C_4H_9)_3$	29
$(C_4H_9)_3SnLi^b$	H_2O	$(C_4H_9)_3SnH$ +	67
		$(C_4H_9)_3SnSn(C_4H_9)_3$	20
$(C_4H_9)_3SnLi^b$	$(CH_3)_3SiCl$	$(C_4H_9)_3SnSi(CH_3)_3$	78

[a] Prepared from R_3SnCl. [b] Prepared from R_3SnSnR_3.

In the reaction between trimethyltinlithium and triphenyltin chloride a metal–halogen interchange must have occurred; this would account for obtaining hexaphenylditin as one of the products. No attempt was made to isolate hexamethylditin, the other product of the metal–halogen interchange. In contrast with the previously reported work[3] (equations 1 and 2), tri-*n*-butyltinlithium (prepared *via* equations 3, 4, and 5) does react with chlorotrimethylsilane to give the desired product, trimethylsilyltributyltin, in 78% yield.

The results of this investigation and those reported earlier for triphenyltinlithium[1b] provide a convenient general method for the preparation of either trialkyltinlithium or triphenyltinlithium from the reaction of lithium in tetrahydrofuran with either R_3SnCl or R_3SnSnR_3.

Experimental

Preparation of Trimethyltinlithium.—A solution of 39.8 g. (0.2 mole) of trimethyltin chloride in 150 ml. of tetrahydrofuran was added to a stirred, cooled suspension of 13.9 g. (2 g.-atoms) of lithium clippings in 150 ml. of tetrahydrofuran slowly enough to enable maintaining the mixture below 5°. After only one-third of the solution had been added the light green mixture gave an intense positive Color Test I.[4] The reaction was exothermic and between 1 and 2 hr. after the addition had been completed the mixture turned dark green. After being stirred for 3 hr. the mixture was filtered through glass wool and derivatives were prepared as described below.

Preparation of Ethyltrimethyltin.—To a stirred solution of trimethyltinlithium prepared as described above from 0.2 mole of trimethyltin chloride was added a solution of 21.8 g. (0.2 mole) of ethyl bromide in 160 ml. of tetrahydrofuran slowly enough to enable maintaining the mixture below 0°. The light brown mixture gave a negative Color Test I and was hydrolyzed with saturated ammonium chloride. The organic layer was combined with ether extracts of the aqueous layer and dried over magnesium sulfate. Evaporation of solvents left a liquid residue which was distilled to give 22.6 g. (58.7%) of ethyltrimethyltin, b.p. 104–105.5° (lit.,[5] b.p. 106° at 746 mm.), n^{20}_D 1.4527.

Anal. Calcd. for $C_5H_{14}Sn$: C, 31.14; H, 7.32. Found: C, 31.10, 30.88; H, 7.02, 7.08.

Preparation of *n*-Butyltrimethyltin.—To a stirred solution of trimethyltinlithium prepared as described above from 0.05 mole of trimethyltin chloride was added a solution of 13.3 g. (0.05 mole) of tri-*n*-butyl phosphate in 30 ml. of tetrahydrofuran during 25 min. The black mixture gave a negative Color Test I and was stirred for 80 min., then was hydrolyzed with water. The organic layer was combined with ether extracts of the aqueous layer and dried over sodium sulfate. Evaporation of solvents left a liquid residue which was distilled to give 3.4 g. (31%) of *n*-butyltrimethyltin, b.p. 46–46.5° (14 mm.) (lit.,[5] b.p. 149–150° at 724 mm.), n^{20}_D 1.4567.

Anal. Calcd. for $C_7H_{18}Sn$: C, 38.06; H, 8.21. Found: C, 38.38, 37.83; H, 8.11, 8.21.

Preparation of 1,1,1-Trimethyl-2,2,2-triphenylditin.—To a stirred solution of trimethyltinlithium prepared as described above from 0.1 mole of trimethyltin chloride was added a solution of 38.5 g. (0.1 mole) of triphenyltin chloride in 75 ml. of tetrahydrofuran slowly enough to enable maintaining the mixture below 0°. The dark brownish black mixture gave a negative Color Test I and was hydrolyzed with saturated ammonium chloride. The organic layer was combined with ether extracts of the aqueous layer and dried over magnesium sulfate. Evaporation of solvents left a white solid which was swirled with boiling ethanol leaving undissolved 11.3 g. (32.3%) of hexaphenylditin, m.p. 230–234° cor., identified by a mixture melting point with an authentic sample. From the filtrate was obtained 26.2 g. (50.9%) of 1,1,1-trimethyl-2,2,2-triphenylditin, m.p. 103–106° cor. Recrystallization from ethanol gave white crystals, m.p. 107–108.5° cor. (lit.,[6] m.p. 106°).

Anal. Calcd. for $C_{21}H_{24}Sn_2$: C, 49.09; H, 4.71; Sn, 46.20; mol. wt., 513.8. Found: C, 49.10, 49.22; H, 4.67, 4.74; Sn, 45.61, 46.04; mol. wt., 525, 507.

Preparation of Tri-*n*-butyltinlithium from Hexa-*n*-butylditin.—A stirred mixture of 58.0 g. (0.1 mole) of hexa-*n*-butylditin, 6.9 g.

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(2) H. Gilman and S. D. Rosenberg, *J. Am. Chem. Soc.*, **75**, 2507 (1953).

(3) D. Blake, G. E. Coates, and J. M. Tate, *J. Chem. Soc.*, 618 (1961).

(4) H. Gilman and F. Schulze, *J. Am. Chem. Soc.*, **47**, 2002 (1925).

(5) Z. M. Manulkin, *J. Gen. Chem. USSR*, **13**, 42 (1943); *Chem. Abstr.*, **38**, 331 (1944).

(6) C. A. Kraus and R. M. Bullard, *J. Am. Chem. Soc.*, **48**, 2131 (1926).

(1.0 g.-atom) of lithium clippings, and 200 ml. of tetrahydrofuran, after not reacting for 30 min. at room temperature, was heated to 53°. Within 4 min. the mixture turned olive-green, evolved a small amount of heat, and gave a strong positive Color Test I. After being stirred for 4 hr. the black mixture gave a very intense positive Color Test I. The mixture was filtered through glass wool and derivatives were prepared as described below.

Preparation of Trimethylsilyltri-*n*-butyltin.—To a stirred solution of tri-*n*-butyltinlithium prepared as described above from 0.1 mole of hexa-*n*-butylditin, was added a solution of 23.9 g. (0.22 mole) of chlorotrimethylsilane in 100 ml. of tetrahydrofuran slowly enough to enable maintaining the mixture below 0°. The black mixture gave a negative Color Test I and was filtered. After distilling the tetrahydrofuran and filtering the lithium chloride, the residue was vacuum distilled to give 56.6 g. (77.8%) of trimethylsilyltri-*n*-butyltin, b.p. 88° (0.2 mm.), $n^{20}\mathrm{D}$ 1.4873. The infrared spectrum shows peaks at 1250 cm.$^{-1}$ and 840 cm.$^{-1}$ characteristic of the trimethylsilyl group.

Anal. Calcd. for $C_{15}H_{36}SiSn$: C, 49.60; H, 9.99. Found: C, 49.40; 49.46; H, 9.73, 9.70.

Preparation of Tri-*n*-butyltin Hydride from Hexa-*n*-butylditin.—A solution of tri-*n*-butyltinlithium prepared as described above from 0.1 mole of hexa-*n*-butylditin was hydrolyzed with water. The organic layer was combined with ether extracts of the aqueous layer and dried over magnesium sulfate. Evaporation of solvents left a liquid residue which was distilled to give 39.1 g. (67%) of tri-*n*-butyltin hydride, b.p. 63–64° (0.41–0.48 mm.) (lit.,[7] b.p. 76–81° at 0.7–0.9 mm.), $n^{22}\mathrm{D}$ 1.4721.

Anal. Calcd. for $C_{12}H_{28}Sn$: C, 49.52; H, 9.69. Found: C, 49.56, 49.26; H, 9.35, 9.40.

Hexa-*n*-butylditin was also obtained (11.8 g., 20%); this was identified by comparison of its infrared spectrum with that of an authentic sample.[8]

Preparation of Tri-*n*-butyltinlithium from Tri-*n*-butyltin Chloride.—A mixture of 65.1 g. (0.2 mole) of tri-*n*-butyltin chloride and 15.0 g. (2.0 g.-atoms) of lithium clippings was stirred for 1 hr. Although the mixture turned dark, Color Test I was negative. When tetrahydrofuran (100 ml.) was slowly added the reaction became exothermic, the mixture turned dark green, and Color Test I became positive. After being stirred for 2 hr. the mixture was filtered through glass wool and a derivative was prepared as described below.

Preparation of Tri-*n*-butyltin Hydride from Tri-*n*-butyltin Chloride.—A solution of tri-*n*-butyltinlithium prepared as described above from 0.2 mole of tri-*n*-butyltin chloride was hydrolyzed with water. The mixture was treated in the same way as described above in the preparation of tri-*n*-butyltin hydride from hexa-*n*-butylditin. The yield of tri-*n*-butyltin hydride was 31.6 g. (54%), b.p. 46–49° (0.18 mm.), $n^{22}\mathrm{D}$ 1.4720. Hexa-*n*-butylditin was also obtained (16.5 g., 28%), $n^{22}\mathrm{D}$ 1.5089. The infrared spectrum of this material was identical with that of an authentic sample.[8]

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[7] J. G. Noltes and G. J. M. van der Kerk, "Functionally Substituted Organotin Compounds," Tin Research Institute, Greenford, Middlesex, England, 1958, p. 94.

[8] The hexa-*n*-butylditin was obtained from Metal and Thermit Corp., Rahway, N. J. The sample, $n^{25}\mathrm{D}$ 1.5090, was analytically pure.

The Polarographic Reduction of *p*-Fluoroiodobenzene

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The polarographic reduction of *p*-fluoroiodobenzene was found to proceed normally to fluorobenzene contrary to the report of Colichman and Liu.[1] The early wave at −0.72 volt ascribed by these investigators to the reduction of the fluorine atom, is probably caused by the presence of *p*-nitrofluorobenzene as an impurity. The purified sample used in this study from vapor-phase chromatographic analysis still contained about 1% of this compound. *p*-Diiodobenzene which could arise in the preparation of *p*-fluoroiodobenzene from *p*-fluoroaniline was eliminated as another possible contributor to this early wave by its polarographic behavior.

TABLE I

POLAROGRAPHIC BEHAVIOR OF SUBSTITUTED BENZENES

Benzenes	E$^{1}/_{2}$ (S.C.E.)	i_d
Iodobenzene	−1.73	2.86
p-Fluoroiodobenzene	−1.69	3.15
p-Nitrofluorobenzene	−0.74	5.40
p-Diiodobenzene	−1.61	2.96
	−1.79	2.96

The polarographic data for the various compounds and iodobenzene are reported in Table I. *p*-Diiodobenzene gave two waves of equal height close together. The half-wave potentials were calculated by using one-fourth and three-fourths of the total diffusion current.

The half-wave potentials for the first three compounds are slightly more negative than the values reported by Colichman.[1] The value for iodobenzene is, however, in good agreement with the data reported by others.[2]

Experimental

The current–voltage curves were obtained with a Sargent Model XXI Polarograph.

All measurements were made in a water thermostat at 25° ± 0.1° using an H cell fitted with a calomel electrode. The buffer solution used had a pH of 7 and contained 0.060 *M* lithium chloride, 0.024 *M* potassium acetate, and 0.013 *M* acetic acid in 90% alcohol. The composition was the same as that used by Colichman.[1]

The dropping mercury electrode at a pressure of 72 cm. had a drop time of 3.00 seconds (open circuit) in distilled water. The value of m was 1.97 mg. sec.$^{-1}$ with a calculated value of $m^{2/3}t^{1/6}$ of 1.90 mg.$^{2/3}$sec.$^{-1/2}$.

The iodobenzene was obtained from stock. *p*-Fluoronitrobenzene and *p*-diiodobenzene were obtained from the Eastman Kodak Co. *p*-Fluoroiodobenzene was obtained from the Pierce Chemical Co., Rockford, Ill. All samples were checked for purity by vapor-phase chromatography.

Gas chromatograms were obtained using a didecyl phthalate column at 171° with helium as the carrier gas at 20 p.s.i. The retention times of *p*-fluoroiodobenzene and *p*-nitrofluorobenzene were 12 min., and 16 min. and 45 sec., respectively, at a flow rate of 1 ml./sec.

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[1] E. L. Colichman and S. K. Liu, *J. Am. Chem. Soc.*, **76**, 913 (1954).

[2] C. S. Ramanathan and R. S. Subrahmanya, *Proc. Indian Acad. Sci.*, **47A**, 379 (1958).

The Reductive Cleavage of 2,5-Dimethyltetrahydrofuran Hydroperoxide in the Presence of Carbon Tetrachloride[1]

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The reductive cleavage of certain hydroperoxides has been reported in the literature.[3–5] The reaction in-

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[1] Taken from the Master's thesis of Donald F. Anderson.

[2] To whom inquiries should be sent.

[3] Jennings H. Jones and Merrell R. Fenske (to Esso Research and Engineering Co.), U. S. Patent 2,989,563 (June 20, 1961).

[4] J. Braunworth and G. W. Crosby, Abstracts of Papers presented at the 139th National Meeting of the American Chemical Society, St. Louis, Mo., March, 1961, 13-O.

[5] W. Cooper and W. H. T. Davidson, *J. Chem. Soc.*, 1180 (1952).