583. *Preparation, Stability, and Complex Formation of Aryloxyboron Compounds.*

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A number of triaryl borates have been prepared by interaction of boron trichloride and the phenol or naphthol, and steric and polar factors concerning their amine addition compounds are discussed. Ammonia forms 3 : 1 and 2 : 1 complexes respectively with tris-2 : 4 : 6-trichlorophenyl and triphenyl borate. Whereas *p*-nitrophenyl, *o*-tolyl, and *o*-chlorophenyl dichloroboronite are unstable, *o*-nitrophenyl dichloroboronite is by comparison remarkably stable, a property ascribed to internal co-ordination; but all form stable pyridine complexes. There is evidence of isolation of the mixed borate, ethyl di-*o*-nitrophenyl borate, which with pyridine gives triethyl borate, and the pyridine complex of the triaryl borate.

TRIARYL BORATES have previously been obtained by heating a mixture of a phenol and boron trichloride (in a sealed tube),[1] boric acid,[2,3] boric oxide,[4] or boron acetate.[5,6] A number of triaryl borates (Table 1) have now been prepared by the addition of a solution of the phenol (3 mols.) in methylene dichloride to boron trichloride (1 mol.) at —70°. Advantages of this procedure are that nearly quantitative yields are obtained, the only other product is gaseous hydrogen chloride, which together with the low-boiling solvent can readily be removed, the reaction is quick and easily controlled at low temperatures, and functional groups may be present.

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[1] Michaelis and Hillringhaus, *Annalen*, 1901, **315**, 41.
[2] Colclough, Gerrard, and Lappert, *J.*, 1955, 907.
[3] U.S.P. 2,260,336—9/1941; 2,300,006/1942.
[4] Thomas, *J.*, 1946, 823.
[5] Ahmad, Haider, and Khundkar, *J. Appl. Chem.*, 1954, **4**, 543.
[6] Pictet and Geleznoff, *Ber.*, 1903, **36**, 2219.

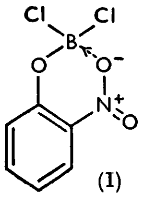
Steric effects in triphenyl borate–alkylamine systems reported by Colclough, Gerrard, and Lappert[2] have been further studied. A number of new 1 : 1 triaryl borate–amine complexes (Table 2) were found to be stable at 20°/0·4 mm. The presence of a single nitro-, chloro-, or iodo-substituent in the *ortho*-position does not prevent co-ordination with pyridine. Molecular models show that two isomeric forms of single *ortho*-substituted phenyl borates should be possible, having a symmetrical or unsymmetrical configuration, of which the latter form, for energetic reasons, is likely to be obtained in the preparation and should itself be capable of forming two isomeric addition compounds depending on which side of the molecule the base is attached. Brown and Sujishi[7] account for the formation of two isomeric addition compounds of tri-α-naphthylboron and amines by postulating the rotation of one α-naphthyl group in the less stable unsymmetrical form to give a more stable form.

The addition products (Table 3) all contain less amine than required for a 1 : 1 complex. In these examples some amine could be removed at 20°/0·4 mm. from an equimolecular mixture of the borate and amine. This reduced tendency to co-ordination is attributed mainly to *F*-strain[8] between the borate and base, but may also be due to *B*-strain in the borate. Molecular models show that by suitable orientation of the methoxy-groups, tris-2 : 6-dimethoxyphenyl borate should just be capable of forming an addition compound with pyridine (cf. Table 3). Tris-2 : 6-dimethyl- and tris-2 : 4 : 6-trichloro-phenyl borate would not co-ordinate with pyridine; this is in accordance with the shielding effect of six rigid *o*-methyl or *o*-chloro-substituents round the boron atom. The electrophilic character of the boron should be decreased by the $+I$ inductive effect of the methyl group, but on the other hand should be increased by the $-I$ inductive effect of the chlorine substituents. Steric effects therefore appear to be predominant here. Hindrance in bis-2 : 4 : 6-trichlorophenyl chloroboronate is insufficient to prevent the formation of a complex with pyridine. Neither diethyl ether nor di-*n*-butyl sulphide co-ordinated with tri-*p*-chlorophenyl or tri-2 : 5-dichlorophenyl borate, a fact attributable to the weaker nucleophilic character of the oxygen and the sulphur atom compared with nitrogen (in amines), and not to steric hindrance.

Ammonia forms 3 : 1 and 2 : 1 complexes with tris-2 : 4 : 6-trichlorophenyl and triphenyl borate respectively. Conductivity measurements in acetone indicated that the complexes are ionic :

$$[(Cl_3C_6H_2O)_2]^{2-}[Cl_3C_6H_2{\cdot}O{\cdot}B(NH_3)_3]^{2+} \quad [PhO]^{-}[(C_6H_5{\cdot}O)_2B(NH_3)_2]^{+}$$

Comparably with the phenyl esters[2] *p*-nitrophenyl, *o*-chlorophenyl, and *o*-tolyl dichloroboronites readily disproportionate at room temperature and at reduced pressure, but *o*-nitrophenyl dichloroboronite is remarkably stable, probably because of internal B–O co-ordination (cf. I). It did not undergo disproportionation or decomposition into *o*-chloronitrobenzene, even on being heated with ferric chloride. The dichloroboronites of *p*-nitrophenol, *o*-chlorophenol, *o*-cresol, and phenol were stabilised by co-ordination with pyridine. Bettman, Branch, and Yabroff[9] believe that the abnormal weakness of *o*-nitrophenylboronic acid, $o\text{-}NO_2{\cdot}C_6H_4{\cdot}B(OH)_2$, is due to co-ordination of the *o*-nitro-group with the boron atom. Intermolecular co-ordination appears to be less effective, since *p*-nitrophenyl dichloroboronite is unstable and *p*-nitrophenylboronic acid is the strongest known arylboronic acid.[9]



(I)

We confirm an earlier observation[10] of the formation of nitrobenzene–boron trichloride.

Mixed borates of the type $(RO)_2B{\cdot}OR'$ ($R' =$ alkyl[11] or aryl[2]) have not previously been isolated owing to disproportionation : $3(RO)_2B{\cdot}OR' \longrightarrow 2(RO)_3B + (R'O)_3B$. Ethyl di-*o*-nitrophenyl borate has been obtained from ethyl alcohol (1 mol.) and di-*o*-nitrophenyl chloroboronate :

$$(NO_2{\cdot}C_6H_4{\cdot}O)_2BCl + EtOH \longrightarrow (NO_2{\cdot}C_6H_4{\cdot}O)_2B{\cdot}OEt + HCl$$

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[7] Brown and Sujishi, *J. Amer. Chem. Soc.*, 1948, **70**, 2793.

[8] Brown, Schlesinger, and Cardon, *ibid.*, 1942, **64**, 325.

[9] Bettman, Branch, and Yabroff, *ibid.*, 1934, **56**, 1865.

[10] Kinney and Mahoney, *J. Org. Chem.*, 1943, **8**, 526.

[11] Thomas, *J.*, 1946, 823.

Hydrogen chloride (100%) was evolved, and triethyl borate could not be removed under reduced pressure, nor could it be extracted with pentane, as should be possible if disproportionation, $3(NO_2{\cdot}C_6H_4{\cdot}O)_2B{\cdot}OEt \longrightarrow (EtO)_3B + 2(NO_2{\cdot}C_6H_4{\cdot}O)_3B$, had occurred. From a freshly prepared mixture of triethyl borate (1 mol.) and tri-*o*-nitrophenyl borate (2 mols.) all the triethyl borate was immediately removed at low pressure, but after the mixture had been set aside for 15 hr. only 5% of the added triethyl borate could be so removed, indicating the occurrence of the reaction $(EtO)_3B + 2(NO_2{\cdot}C_6H_4{\cdot}O)_3B \longrightarrow 3(NO_2{\cdot}C_6H_4{\cdot}O)_2B{\cdot}OEt$. The mixed borate, however, disproportionates on the addition of pyridine, affording ethyl borate and the pyridine complex of the triaryl borate.

EXPERIMENTAL

General Procedures.—These were described in the previous paper.[2] *o*-Iodophenol was obtained by Whitmore and Hanson's method.[12]

Preparation of Triaryl Borates from Boron Trichloride.—The borates in Table 1 were obtained in yields of 96—100% by the addition of the phenol (3 mols.) in methylene dichloride to boron trichloride (1 mol.) at −60° to −80°. Hydrogen chloride (collected in moist alkali) was evolved in yields of 96—100% as the mixture warmed to 15°. The borates were purified by recrystallisation from methylene dichloride or benzene.

TABLE 1. *Triaryl borates.*

		Found (%)				Required (%)			
Ar	M. p.	B	ArO	C	H	B	ArO	C	H
p-C_6H_4Cl	55°	2·85	98·0	—	—	2·74	97·2	—	—
2 : 5-$C_6H_3Cl_2$ *	106	2·26	97·5	—	—	2·18	97·8	—	—
2 : 4 : 6-$C_6H_2Cl_3$ * ...	173	1·71	—	36·4	1·3	1·81	—	36·0	1·0
2 : 6-$C_6H_3Me_2$	135	2·70	96·3	—	—	2·86	97·1	—	—
4-*n*-Octylphenyl * ...	82	1·63	97·5	—	—	1·73	98·3	—	—
o-$NO_2{\cdot}C_6H_4$ *†	108	2·62	—	50·4	3·1	2·54	—	50·9	2·9
2 : 6-$(MeO)_2C_6H_3$ * ...	134	2·36	—	59·7	6·0	2·31	—	61·3	5·8
α-$C_{10}H_7$	108	2·48	—	81·5	5·0	2·46	—	81·9	4·8
β-$C_{10}H_7$	120	2·44	—	81·3	5·0	2·46	—	81·9	4·8
o-C_6H_4I *	90	1·66	—	32·3	2·0	1·83	—	32·4	1·8

* New compounds. † Found : N, 10·1. Required, N. 9·9%.

Interaction of Amines with Naphthyl and Substituted Phenyl Borates.—The new 1 : 1 borate–amine *complexes* in Table 2 were obtained by the addition of pyridine (1 mol.) to a solution of the borate (1 mol.) in methylene dichloride. Solvent was removed at 20°/30—15 mm. No dissociation of the complexes occurred at 20°/0·4 mm. or on being mixed with *n*-pentane.

TABLE 2.

			Found (%)			Reqd. for 1 : 1 complex (%)		
Ar	Amine	M. p.	B	ArO	Amine	B	ArO	Amine
p-C_6H_4Cl	Pyridine	68—70°	2·39	78·5	16·7	2·28	81·1	16·7
2 : 5-$C_6H_3Cl_2$	,,	146—148	1·97	82·2	13·6	1·88	84·4	13·7
p-Octylphenyl ...	,,	112	1·53	87·5	11·2	1·52	87·5	11·2
o-$NO_2{\cdot}C_6H_4$	,,	167	2·33	—	14·0	2·14	—	15·6
Ph	α-Picoline	104	2·69	73·3	24·8	2·83	72·7	24·4
α-$C_{10}H_7$	Pyridine	174	2·15	—	14·6	2·09	—	15·2
α-$C_{10}H_7$	2 : 4 : 6-Collidine	*	1·88	—	21·2	1·93	—	21·6
β-$C_{10}H_7$	Pyridine	57—60	1·93	—	14·3	2·09	—	15·2
β-$C_{10}H_7$	α-Picoline	66—68	1·91	—	16·2	2·03	—	17·4
β-$C_{10}H_7$	2 : 4 : 6-Collidine	110	1·89	—	22·7	1·93	—	21·6
o-C_6H_4I	Pyridine	52—55	1·40	—	11·1	1·45	—	10·6
o-C_6H_4I	α-Picoline	110	1·46	—	12·7	1·42	—	12·1

* Semiliquid.

Non-Stoicheiometric Triaryl Borate–Amine Addition Products.—The amines (1 mol.) were added to the borate (1 mol.) in methylene dichloride. Solvent was removed at 20°/30—15 mm. As much amine as possible was removed at 20°/0·4 mm. or by extraction with *n*-pentane, and was recovered. The borate retained base to the extent shown in Table 3.

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[12] *Org. Synth.*, Coll. Vol. I, Wiley and Sons, Inc., New York, 1947, pp. 161, 326.

TABLE 3.

			Found (%)		Calc. for 1 : 1 complex (%)	
Ar	Amine	M. p.	B	Amine	B	Amine
2 : 6-$(MeO)_2C_6H_3$	Pyridine	108°	2·5	5·6	2·27	16·5
2 : 5-$C_6H_3Cl_2$	Diethylamine	175	1·78	10·1	1·90	12·8
Ph	2 : 4 : 6-Collidine	*	—	16·0	—	29·4
2 : 5-$C_6H_3Cl_2$	Triethylamine	*	—	8·1	—	16·9
p-C_6H_4Cl	,,	*	—	13·1	—	20·5

* Semi-liquid.

No addition compounds between pyridine and tris-2 : 4 : 6-trichlorophenyl borate or tris-2 : 6-dimethylphenyl borate were isolated. The amine (1 mol.) was completely removed from either borate (1 mol.) at 20°/0·4 mm. or by washing with *n*-pentane.

Bis-2 : 4 : 6-*trichlorophenyl Chloroboronate*.—2 : 4 : 6-Trichlorophenol (15·18 g., 2 mols.) in methylene dichloride (30 ml.) was added to boron trichloride (4·5 g., 1 mol.) at −70°. Volatile matter was removed at 20°/0·4 mm., and white crystalline *bis*-2 : 4 : 6-*trichlorophenyl chloroboronate* (16·9 g., 100%), m. p. 115° [Found : easily hydrolysed (e.h.) Cl, 7·9; B, 2·49. $C_{12}H_4O_2Cl_7B$ requires e.h. Cl, 8·1; B, 2·48%], remained. Pyridine (2·76 g., 1 mol.) in *n*-pentane (30 ml.) was added slowly to a shaken suspension of the chloroboronate (15·23 g., 1 mol.) in methylene dichloride (30 ml.) at −60°. Solvent was removed at 22°/30 mm., but no pyridine was removed during 2 hr. at 22°/0·4 mm., or on washing with *n*-pentane. The *complex* (17·9 g., 96%) had m. p. 89—91° (decomp.) (Found : e.h. Cl, 6·7; B, 1·9; C_5H_5N, 14·6. $C_{17}H_9O_2NCl_7B$ requires e.h. Cl, 6·8; B, 2·09; C_5H_5N, 15·3%).

Interaction of Ammonia and Tris-2 : 4 : 6-*trichlorophenyl Borate*.—Dry ammonia was passed into a solution of the borate (12·73 g.) in methylene dichloride (100 ml.) for 3 hr. A white precipitate formed. The solvent was removed at 20°/15 mm. (2 hr.), and at 20°/0·4 mm. (2 hr.). The residue (13·15 g.), m. p. 138°, contained ammonia and borate in the mol. ratio 1·45 : 1. More ammonia was added to a suspension of this solid (9·4 g.) in ether (100 ml.) and methylene dichloride (50 ml.). When further precipitation had ceased (1 hr.), filtration, and drying at 20°/15 mm. (2 hr.) and at 20°/0·4 mm. afforded *tris*-2 : 4 : 6-*trichlorophenyl borate–triammonia compound* (8·72 g.), m. p. 150° [Found : B, 1·75; NH_3, 7·74. $(Cl_3C_6H_2\cdot O)_3B,3NH_3$ requires B, 1·68; NH_3, 7·85%], stable (7 days) in moist atmosphere.

Interaction of Ammonia and Triphenyl Borate.—Dry ammonia was passed into a solution of the borate (25·6 g.) in ether (100 ml.). The white precipitate and solvent were cooled to −60° and more ammonia was allowed to condense until saturation was reached. After 15 hr. at 20°, solvent and the excess of ammonia were removed at 21°/1·5 mm. (2 hr.), whereupon the *diammonia compound* (27·5 g.), m. p. 125° [Found : B, 3·21; PhO, 85·6; NH_3, 10·7 $(C_6H_5O)_3B,2NH_3$ requires B, 3·35; PhO, 86·0; NH_3, 10·5%], unstable (7 days) in moist atmosphere, was obtained.

Conductivity of Ammonia Complexes.—The triammonia–tris-2 : 4 : 6-trichlorophenyl borate compound was insoluble in nitrobenzene and in nitromethane, but slightly soluble in acetone, conductivity, $\kappa = 9{\cdot}10 \times 10^{-6}$ mho for 0·0007 molar concentration, whereas for pure acetone $\kappa = 2{\cdot}45 \times 10^{-6}$ mho at 20°.

The diammonia–triphenyl borate compound was insoluble in nitromethane and in nitrobenzene, but soluble in acetone, its conductivity κ being $9{\cdot}61 \times 10^{-6}$ mho for 0·0035 molar concentration.

o-*Nitrophenyl Dichloroboronite*.—Tri-*o*-nitrophenyl borate (8·24 g., 1 mol.) in methylene dichloride (30 ml.) was added to boron trichloride (4·56 g., 2 mols.) at −70°. After 1 hr. at −70°, the solution was allowed to warm to 20°. Solvent, but no boron trichloride, was removed at 20°/15 mm. (2 hr.), leaving red crystalline o-*nitrophenyl dichloroboronite* (13·5 g., 98%), m. p. 63° (Found : Cl, 31·6; B, 4·84. $C_6H_4O_3NCl_2B$ requires Cl, 32·3; B, 4·92%). Whilst the compound (4·95 g.) was heated for 6 hr. at 100° a trace (0·04 g.) of boron trichloride was evolved. During 4·5 hr. at 95°/45 mm., boron trichloride (Found : Cl, 0·07 g.) and a small amount of *o*-nitrophenol (indicated by the red colour in the sodium hydroxide used for absorption) were evolved, and the residue (4·8 g.) (Found : Cl, 28·4%) had darkened.

No boron trichloride was evolved when the dichloroboronite (6·76 g.) was heated in the presence of ferric chloride (0·047 g., 0·7% w/w) at 100° (7 hr.). During 5 hr. at 100°/20 mm., some boron trichloride (Found : Cl, 0·13 g.) and nitrophenol were collected in the alkali trap. The residue (6·3 g.) (Found : B, 4·90; Cl, 23·6%) had darkened. The dichloroboronite was not distillable at pressures down to 0·01 mm.

Pyridine (2·66 g., 1 mol.) in methylene dichloride (15 ml.) was added slowly to the dichloroboronite (11·1 g., 1 mol.) in methylene dichloride (30 ml.) at −70°. The residue (13·41 g., 98%) after evaporation of solvent at 20°/15 mm. was washed with *n*-pentane (50 ml.). Residual solvent (but no pyridine) was removed at 20°/0·4 mm., whereupon the *pyridine–*o*-nitrophenyl dichloroboronite compound* (9·61 g., 70%), m. p. 172° (Found : Cl, 23·1; B, 3·68; C_5H_5N, 26·7. $C_{11}H_9O_3N_2Cl_2B$ requires Cl, 23·7; B, 3·63; C_5H_5N, 26·5%), remained. Pyridine (1 mol.) was recovered from a mixture of the complex (1 mol.) and base (1 mol.).

Very little hydrolysis took place when the complex (0·871 g.) was shaken with water (100 ml.) for 1 hr. at 20°. On filtering, the washings contained boric acid (0·006 g.) and chloride ion (0·014 g.), and the insoluble residue weighed 0·7 g. after being washed with ether to remove any nitrophenol (Found : Cl, 21·8; C_5H_5N, 26·4%). After 4 days complete hydrolysis to *o*-nitrophenol, boric acid, hydrochloric acid, and pyridine had occurred.

Disproportionation of p-*Nitrophenyl Dichloroboronite.*—A suspension of *p*-nitrophenol (2·76 g., 1 mol.) in methylene dichloride (30 ml.) was added slowly with shaking to boron trichloride (2·33 g., 1 mol.) at −70°. After 2 hr. the solution was allowed to warm to 20°. Hydrogen chloride (0·7 g., 100% according to $ArOH + BCl_3 \longrightarrow ArO{\cdot}BCl_2 + HCl$) and boron trichloride [0·74 g., 94% according to $2ArO{\cdot}BCl_2 \longrightarrow BCl_3 + (ArO)_2BCl$] were evolved at 20°/12 mm. (3 hr.). No boron trichloride was evolved during a further 6 hr. and the product at 20°/12 mm. *di-*p*-nitrophenyl chloroboronate* (3·1 g., 96%), m. p. 187—190° (Found : Cl, 11·0; B, 3·46. $C_{12}H_8O_6N_2ClB$ requires Cl, 11·0; B, 3·36%), remained.

Disproportionation of o-*Chlorophenyl Dichloroboronite.*—*o*-Chlorophenol (3·82 g., 1 mol.) was added to boron trichloride (3·5 g., 1 mol.) at −70°. After 1 hr. at −70°, the dichloroboronite was warmed to 20°. Hydrogen chloride and boron trichloride were readily evolved at 20°/15 mm. After 9·5 hr. the temperature was raised to 130° to compensate for the reduction in rate of evolution of trichloride. The residue (3·73 g., 95% after 16·5 hr.) was mainly tri-*o*-chlorophenyl borate (Found : e.h. Cl, 1·1; B, 3·3; $C_6H_4Cl{\cdot}O$, 95·9. Calc. for $C_{18}H_{12}O_3Cl_3B$: B, 2·74; $C_6H_4Cl{\cdot}O$, 97·3%).

Disproportionation of o-*Tolyl Dichloroboronite.*—Similarly *o*-cresol (2·65 g., 1 mol.) and boron trichloride (1 mol.) in methylene dichloride (35 ml.) gave impure tri-*o*-tolyl borate (2·46 g., after being at 24°/30 mm., for 6 hr., and at 140°/70 mm. for 6 hr.) (Found : e.h. Cl, 3·0; B, 3·7%).

Aryl Dichloroboronite–Pyridine Complexes.—The *complex* when aryl = p-*nitrophenyl* had m. p. 194—196° (Found : Cl, 22·4; B, 3·60; C_5H_5N, 25·6. $C_{11}H_9O_3N_2Cl_2B$ requires Cl, 23·7; B, 3·61; C_5H_5N, 26·4%), when aryl = o-*chlorophenyl* had m. p. 67—70° (Found : e.h. Cl, 23·9; B, 3·61; C_5H_5N, 28·6. $C_{11}H_9ONCl_3B$ requires e.h. Cl, 24·6; B, 3·75; C_5H_5N, 27·4%), and when aryl = o-*tolyl* had m. p. 50—55° (Found : Cl, 26·2; B, 3·8; C_5H_5N, 28·7. $C_{12}H_{12}ONCl_2B$ requires Cl, 26·5; B, 4·04; C_5H_5N, 29·5%), and was obtained by the addition of pyridine (3 mol.) to a solution of the dichloroboronite (3 mol.) (*i.e.*, borate, 1 mol., + boron trichloride, 2 mol.) in methylene dichloride at −70°.

*Interaction of Di-*o*-nitrophenyl Chloroboronate* (1 *Mol.*) *with Ethanol* (1 *Mol.*).—Ethanol (5·94 g., 3 mol.) in *n*-pentane was added dropwise to the chloroboronate (45·5 g., 3 mol.) (obtained from the borate, 2 mol., + boron trichloride, 1 mol., at −70°) in methylene dichloride at −70°. Hydrogen chloride (5·0 g., 100%) was evolved at 25°/20 mm., but no triethyl borate was removed first at 22°/0·1 mm. (1 hr.) then at 20°/760 mm. (15 hr.) and at 25°/0·1 mm. (1·5 hr.). The red liquid residue (48·7 g., 98·2%) was presumably *ethyl di-*o*-nitrophenyl borate* (Found : B, 3·22. $C_{14}H_{13}O_7N_2B$ requires B, 3·26%). No triethyl borate could be extracted by *n*-pentane.

Addition of pyridine (2·75 g., 3 mol.) to this compound (12·01 g., 3 mol.) at −70° caused disproportionation. Triethyl borate (1·62 g., 1·0 mol.) and pyridine (0·92 g., 0·99 mol.) were removed at 25°/0·2 mm. (1 hr.), and the pyridine–tri-*o*-nitrophenyl borate compound (13·4 g., 94%), m. p. 138° (Found : C_5H_5N, 13·2%), remained. Yields are based on $3(ArO)_2B{\cdot}OEt + 3C_5H_5N \longrightarrow B(OEt)_3 + C_5H_5N + 2(ArO)_3B,C_5H_5N$.

*Interaction of Triethyl Borate with Tri-*o*-nitrophenyl Borate.*—Immediately after triethyl borate (1·25 g., 1 mol.) had been added to tri-*o*-nitrophenyl borate (7·23 g., 2 mol.) triethyl borate (1·22 g., 97%) was removed at 25°/0·1 mm. (3 hr.). Tri-*o*-nitrophenyl borate (7·16 g., 99%), m. p. 86—89°, remained.

When the primary mixture was stored at 20° for 15 hr., only 5% of the added ethyl borate was removed at 20°/0·1 mm. (5 hr.).

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