761. *The Preparation and Properties of Di-n-butylboronous Anhydride.*

By W. Gerrard, M. F. Lappert, and R. Shafferman.

Di-*n*-butylboronous anhydride, $(Bu^n{}_2B)_2O$, was prepared by reaction of *n*-butylmagnesium bromide and ether–boron trifluoride and subsequent hydrolysis. With alcohols (except *tert.*-butyl) it yielded the appropriate boronites, $Bu^n{}_2B{\cdot}OR$; with boron halides (Cl, Br) the di-*n*-butylboron halides, $Bu^n{}_2BX$; and with phosphorus pentachloride di-*n*-butylboron chloride. With water it gave di-*n*-butylboronous acid, $Bu^n{}_2B{\cdot}OH$. All the products of these reactions could be reconverted into the anhydride. With acetic acid, di-*n*-butylboronous anhydride was de-butylated to give $[Bu^n(AcO)B]_2O$. It did not react with bases, thionyl chloride, or hydrogen chloride. The di-*n*-butylboron halides were readily hydrolysed and formed 2 : 1 complexes with pyridine. The results are discussed.

Di-*n*-butylboronous anhydride, $(Bu^n{}_2B)_2O$, has been prepared[1] by the reaction of aqueous hydrobromic acid with tri-*n*-butylboron, as well as by the addition [2] of *n*-butylmagnesium bromide to *n*-butyl ethylene borate, $Bu^nO{\cdot}B(O{\cdot}CH_2{\cdot})_2$. Of the boronous esters, $R_2B{\cdot}OR'$, only *n*-butyl di-*n*-butylboronite has hitherto been prepared (though not characterised); one method involved the esterification of the anhydride.[1] Di-*n*-butylboron chloride had been obtained[3] from tri-*n*-butylboron ($Bu^n{}_3B + HCl \longrightarrow C_4H_{10} + Bu^n{}_2B{\cdot}Cl$), and by the action of phosphorus pentachloride on *n*-butyl di-*n*-butylboronite.[4] The corresponding bromide had been obtained by treating anhydrous hydrogen bromide or bromine with tri-*n*-butylboron;[1] thermochemical data are available for the hydrolysis of both these butylboron halides to give di-*n*-butylboronous acid.[5] The present paper is the first of a series designed to outline systematically the chemistry of the dialkylboron system, $R_2B{\cdot}Y$, and is concerned mainly with the chemistry of di-*n*-butylboronous anhydride, $(Bu^n{}_2B)_2O$.

Di-*n*-butylboronous anhydride has now been obtained [scheme (1)] in 56% yield by the interaction of *n*-butylmagnesium bromide (2 mols.) with ether–boron trifluoride (1 mol.), subsequent acid hydrolysis, and distillation. This method appears to be superior to earlier ones,[2, 6] both in yield and in number of steps involved:

$$2Bu^nMgBr + BF_3 \xrightarrow{H_2O} Bu^n{}_2B{\cdot}OH \xrightarrow{-H_2O} \tfrac{1}{2}(Bu^n{}_2B)_2O \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

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[1] Johnson, Snyder, and Van Campen, *J. Amer. Chem. Soc.*, 1938, **60**, 115.

[2] Letsinger and Skoog, *ibid.*, 1954, **76**, 4174.

[3] Booth and Kraus, *ibid.*, 1952, **74**, 1415.

[4] Mikhailov and Shchegoleva, *Isvest. Akad. Nauk, Otdel. Khim. Nauk*, 1956, 508.

[5] Skinner and Tees, *J.*, 1953, 3378.

[6] Rothstein and Saville, *J.*, 1952, 2987.

With a representative selection of alcohols the anhydride was directly esterified to give the appropriate boronite [scheme (2)], although as with the corresponding diphenylboronous system,[7] the method was not applicable for the preparation of the *tert.*-butyl ester.

$$(Bu^n_2B)_2O + 2ROH \longrightarrow 2Bu^n_2B{\cdot}OR + H_2O \quad . \ . \ . \ . \ . \ . \ . \ . \quad (2)$$

The (+)-1-methylheptyl ester was also obtained [scheme (3)] by the interaction of (+)-octan-2-ol with di-*n*-butylboron chloride:

$$Bu_2^nBCl + ROH \longrightarrow Bu^n_2B{\cdot}OR + HCl \quad . \ . \ . \ . \ . \ . \ . \ . \quad (3)$$

The *n*-butyl ester was hydrolysed by azeotropic distillation, with excess of water to remove the alcohol formed, but hydrolysis of the 1-methylheptyl ester was carried out with aqueous sodium hydroxide:

$$Bu^n_2B{\cdot}OR + H_2O \longrightarrow Bu^n_2B{\cdot}OH + ROH \quad . \ . \ . \ . \ . \ . \ . \ . \quad (4)$$

Di-*n*-butylboronous anhydride was also readily hydrolysed by cold water and the reaction proved reversible at low pressure:

$$(Bu^n_2B)_2O \underset{-H_2O}{\overset{H_2O}{\rightleftharpoons}} 2Bu^n_2B{\cdot}OH \quad . \ . \ . \ . \ . \ . \ . \ . \ . \ . \quad (5)$$

By adding the anhydride to boron trichloride or tribromide, the appropriate di-*n*-butylboron halide was readily formed and the accompanying unstable boron oxyhalide decomposed:

$$(Bu^n_2B)_2O + BX_3 \longrightarrow 2Bu^n_2BX + BOX \quad . \ . \ . \ . \ . \ . \ . \ . \quad (6)$$

$$3BOX \longrightarrow B_2O_3 + BX_3 \quad . \ . \ . \ . \ . \ . \ . \ . \ . \ . \quad (7)$$

Di-*n*-butylboron chloride was also obtained by reaction (8) :

$$(Bu^n_2B)_2O + PCl_5 \longrightarrow 2Bu_2^nBCl + POCl_3 \quad . \ . \ . \ . \ . \ . \ . \ . \quad (8)$$

Both the halides formed 2 : 1 complexes with pyridine (9); by the slow addition of an ethereal solution of water they gave either the acid [scheme (10)], or the anhydride [scheme (11)] depending on the proportions used.

$$2C_5H_5N + Bu^n_2BX \longrightarrow (C_5H_5N)_2,Bu^n_2BX \quad . \ . \ . \ . \ . \ . \ . \ . \quad (9)$$

$$Bu^n_2BX + H_2O \longrightarrow Bu^n_2B{\cdot}OH + HX \quad . \ . \ . \ . \ . \ . \ . \ . \quad (10)$$

$$2Bu^n_2BX + H_2O \longrightarrow (Bu^n_2B)_2O + HX \quad . \ . \ . \ . \ . \ . \ . \ . \quad (11)$$

When a mixture of acetic acid (in excess) and di-*n*-butylboronous anhydride was heated, reaction (12) ensued. The novel acetoxy-*n*-butylboronous anhydride was characterised by hydrolysis with cold water to *n*-butylboronic acid (13).

$$(Bu^n_2B)_2O + 2AcOH \longrightarrow (Bu^nB{\cdot}OAc)_2O + 2C_4H_{10} \quad . \ . \ . \ . \ . \ . \quad (12)$$

$$(Bu^nB{\cdot}OAc)_2O + 3H_2O \longrightarrow Bu^nB(OH)_2 + 2AcOH \quad . \ . \ . \ . \ . \ . \quad (13)$$

Di-*n*-butylboronous anhydride did not react with pyridine, diethylamine, ammonia, thionyl chloride, or hydrogen chloride under the conditions reported, although heat of mixing with diethylamine was noted.

Discussion.—Contrary to the observations of earlier workers,[1] it appears that both the boronites and di-*n*-butylboronous anhydride [schemes (4) and (5)] can be readily hydrolysed by water or aqueous sodium hydroxide.

(+)-Octan-2-ol afforded (+)-1-methylheptyl di-*n*-butylboronite, having nearly identical optical rotation, whether obtained from di-*n*-butylboron chloride (3) or di-*n*-butylboronous anhydride (2). As reaction (3) must have taken place without alkyl–oxygen fission, it follows that the esterification (2) could also not have involved alkyl–oxygen fission (I), but proceeded as shown in (II). The hydrolysis (4) of (+)-1-methylheptyl di-*n*-butylboronite gave (+)-octan-2-ol having the same rotation as that of the

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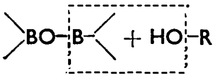
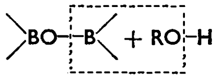
[7] Abel, Gerrard, and Lappert, *J.*, 1957, 112.

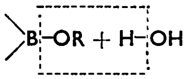
alcohol used for the esterification, hence this reaction also must have involved boron–oxygen fission (III).



(I)



(II)

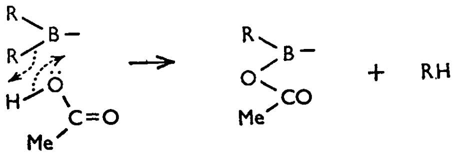


(III)

The atomic refractivity for boron, in the esters, calculated by using Vogel's atomic refractivities,[8] was 1·95 ± 0·15 (see Table 1) compared with 4·39 in the diphenyl esters,[7] which probably show exaltation.

Of the two methods described for the preparation of di-*n*-butylboron chloride, the second (8) is superior as the distillation of the chloride is not accompanied by slow decomposition of any intermediates as in (6) and (7). For the bromide, however, the use of phosphorus pentabromide is excluded owing to its dissociation on heating, as well as the difficulty that would be involved in separating di-*n*-butylboron bromide and phosphorus oxybromide.

The formation (12) of acetoxy-*n*-butylboronous anhydride is exceptional in that carbon–boron fission takes place in preference to boron–oxygen fission. Owing to the technical simplicity of this reaction and the subsequent ease of hydrolysis (13), this is probably the best available method for the conversion of the boronous into the boronic system. Reaction (12) probably proceeds by the usual type of mechanism involving substitution at a boron atom,[9] as shown in (14), although the timing of the covalency changes cannot as yet be decided.



. . . . (14)

The failure of di-*n*-butylboronous anhydride to form complexes is attributed to back co-ordination from the oxygen atom and the strong +*I* effect of the butyl groups, rather than steric hindrance. The failure of diethylamine to cause boron–oxygen fission is in accordance with previous work[10] and it is noteworthy that, by contrast with the acetic acid system, boron–carbon fission does not take place.

EXPERIMENTAL

General Techniques and Preparations.—Di-*n*-butylboronous compounds are very unstable to oxygen, and experiments were carried out in a vacuum or under dry nitrogen. Refractive indices were measured on an Abbé-type refractometer. Molecular weights were determined cryoscopically in *cyclo*hexane. M. p.s were taken in sealed capillary tubes. The ether–boron trifluoride complex was prepared by saturating ether with boron trifluoride at 0°. Boron was determined by using hydrogen peroxide (10 c.c.; 100-vol.; 15 minutes' standing) to oxidise the compounds to their boric acid derivatives.[11] Chlorine and bromine were estimated acidimetrically as hydrogen halide formed on hydrolysis, or by the Volhard method. Amines were estimated by steam-distillation with alkali and titration of the distillate to bromophenol-blue. In the case of acetoxy-*n*-butylboronous anhydride, the acetate radical was determined by Gerrard and Wheelan's method [12] and boron was estimated by conversion into sodium borate. The phenyl group was estimated by hydrolysis to phenol and iodometric estimation by conversion into the tribromo-derivative. As is often found [6] in these types of compounds carbon analyses were generally low; control experiments carried out on the microcombustion apparatus

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[8] Vogel, *J.*, 1946, 133; 1948, 616, 644, 654.
[9] Lappert, *Chem. Rev.*, 1956, **56**, 959.
[10] Gerrard, Lappert, and Pearce, *J.*, 1957, 381.
[11] Snyder, Kuck, and Johnson, *J. Amer. Chem. Soc.*, 1938, **60**, 105.
[12] Gerrard and Wheelans, *J.*, 1956, 4296.

were found to be 1% below standard for carbon, after analysis (C and H), probably owing to formation of boron carbide. Rotatory powers are recorded for l = 10 cm.

Preparation (1) *of Di-*n*-butylboronous Anhydride.*—*n*-Butylmagnesium bromide (2 moles) in ether (1600 c.c.) was added during 6 hr. with stirring to boron trifluoride (1 mole) in ether (400 c.c.) at −70°. The mixture was allowed to warm (18 hr.) to 20°, then hydrolysed with 3N-hydrochloric acid (1 l.) to give two layers. After extraction with ether and drying ($MgSO_4$) all volatile matter was removed at 25°/9 mm. Distillation of the residue yielded a forerun (17 g.), b. p. 40—55°/0·5 mm., and di-*n*-butylboronous anhydride (76 g., 56%), b. p. 137°/9 mm., n_D^{20} 1·4269, d_4^{20} 0·7906 (Found: C, 70·9; H, 13·4; B, 8·1%; *M*, 272. Calc. for $C_{16}H_{36}OB_2$: C, 72·3; H, 13·6; B, 8·1%; *M*, 266).

Esterification (2) *of Di-*n*-butylboronous Anhydride.*—This was carried out on approx. 0·05 molar scale and the method used was similar to that of Abel, Gerrard, and Lappert.[7] Excess of the alcohol was added to the anhydride and the water formed was distilled as a water–alcohol azeotrope. The results are shown in Table 1. This method was not successful with *tert.*-butyl alcohol, even when benzene was added in an attempt to remove the water as a ternary azeotrope. The anhydride was recovered in 95% yield.

Preparation of 1-*Methylheptyl Di-*n*-butylboronite by Reaction* (3).—(+)-Octan-2-ol (2·10 g., 1 mol.; α_D^{20} +6·96°) in *n*-pentane was added dropwise with shaking to di-*n*-butylboron chloride (2·59 g., 1 mol.) in *n*-pentane at 20°. Considerable heat of reaction was observed and hydrogen chloride evolved. Volatile matter was removed at 20°/10 mm., to leave (+)-1-methylheptyl di-*n*-butylboronite (3·20 g., 78%), b. p. 114°/0·4 mm., n_D^{20} 1·4278, α_D^{20} +11·04° (Found: B, 4·3%).

Hydrolysis (4) *of* (+)-1-*Methylheptyl Di-*n*-butylboronite.*—3N-Sodium hydroxide (35 c.c.) was added to the ester (3·11 g.; α_D^{20} +11·32°). The mixture was vigorously shaken, then set aside for 24 hr. and extracted with ether. After drying ($MgSO_4$), the ethereal solution afforded (+)-octan-2-ol (1·62 g., 98%), b. p. 82°/18 mm., n_D^{20} 1·4295, α_D^{20} +7·06°.

Hydrolysis (4) *of* n*-Butyl Di-*n*-butylboronite.*—A mixture of the ester (11·45 g.) and water (6·0 g.) was fractionated, to afford the butanol–water azeotrope, b. p. 91°, as a forerun and then di-*n*-butylboronous anhydride (5·10 g., 66%), b. p. 94—124°/ 0·5—1 mm. (redistilled, b. p. 118—124°/14 mm.), d_4^{20} 0·812 (Found: B, 8·1%).

Hydrolysis (5) *of Di-*n*-butylboronous Anhydride.*—Water (0·23 g., 1 mol.) in ether (17 c.c.) was added to the anhydride (3·33 g., 1 mol.) in *n*-pentane (10 c.c.) at 25°. No heat of addition was noted and after vigorous shaking (15 min.) volatile matter was removed at 25°/18 mm., to leave a gelatinous viscous residue of *di-*n*-butylboronous acid* (3·42 g., theor. 3·55 g.), which melted with decomposition in the range 55—65° (Found: B, 7·6. $C_8H_{19}OB$ requires B, 7·6%). Di-*n*-butylboronous acid (3·09 g.) was kept at 20°/0·2 mm. for 6 hr., whereafter a liquid residue of di-*n*-butylboronous anhydride (2·90 g., 100%), b. p. 98°/1·5 mm., n_D^{20} 1·4260 (Found: B, 7·9%), was obtained. A condensate of water (0·19 g., 100%), n_D^{20} 1·3370, m. p. 0°, was trapped at −80°.

TABLE 1.

R in $Bu^n_2B·OR$	Yield (%)	B. p./mm.	d_4^{20}	n_D^{20}	$[R_L]_D$ Found	$[R_L]_D$ Calc.†
Bu^n *	88	114°/20	0·8060	1·4210	62·2	62·3
Bu^i	85	94°/8	0·8004	1·4178	62·3	62·3
Bu^s	78	96°/9	0·7998	1·4170	62·2	62·3
1-Methylheptyl‡	76	116°/0·4	0·8099	1·4279	80·7	80·8
Allyl	78	105°/14	0·8158	1·4279	57·4	57·3
Ph	70	116°/0·1	0·9253	1·4892	67·9	67·9

R in $Bu^n_2B·OR$	Found (%) C	Found (%) H	Found (%) B	Formula	Required (%) C	Required (%) H	Required (%) B
Bu^n *	71·6	13·5	5·3	$C_{12}H_{27}OB$	72·8	13·7	5·5
Bu^i	71·8	13·3	5·5				
Bu^s	71·8	13·5	5·4				
1-Methylheptyl	75·5	13·8	4·2	$C_{16}H_{35}OB$	75·7	13·8	4·3
Allyl	70·5	12·3	5·9	$C_{11}H_{23}OB$	72·6	12·7	6·0
Ph	(Ph, 35·5)		4·9	$C_{14}H_{23}OB$	(Ph, 35·4)		5·0

* Known compound. ‡ α_D^{20} +11·32, from ROH, α_D^{20} +7·28°. † Using a value of 1·95 for boron.

Interaction (6) *of Boron Halides with Di-*n*-butylboronous Anhydride.*—(a) *Boron trichloride.* The anhydride (5·80 g., 1 mol.) was added dropwise with shaking to boron trichloride (2·85 g., 1 mol.) at −70°. Volatile matter was removed at 20°/20 mm. Distillation of the residue yielded a forerun (1·00 g.), di-*n*-butylboron chloride (4·9 g., 83%), b. p. 72—73°/18 mm., n_D^{20} 1·4243 (Found: Cl, 21·9; B, 6·7. Calc. for $C_8H_{18}ClB$: Cl, 22·1; B, 6·7%), and a distillation residue (0·92 g.) of crude boric oxide (Found: B, 26·9. Calc. for O_3B_2: B, 31·1%).

(b) *Boron tribromide.* The anhydride (4·58 g., 1 mol.) was added dropwise with shaking to the tribromide (4·46 g., 1 mol.) at 0°. Distillation of the resultant mixture yielded di-*n*-butylboron bromide (5·00 g., 72%), b. p. 53—55°/0·5 mm., n_D^{20} 1·4450 (Found: Br, 39·0; B, 5·4. Calc. for $C_8H_{18}BrB$: Br, 39·1; B, 5·3%), and a solid residue (0·11 g.) of crude boric oxide (Found: B, 24·9%). Boron tribromide (1·24 g., 86%) (Found: Br, 93·1; B, 4·2. Calc. for Br_3B: Br, 95·7; B, 4·3%) was trapped as a distillation condensate at −80°.

Interaction (8) *of Phosphorus Pentachloride and Di*-n-*butylboronous Anhydride.*—The anhydride (6·21 g., 1 mol.) was added to the pentachloride (4·90 g., 1 mol.) and heated under reflux at 130° until it became homogeneous. Fractionation then yielded phosphorus oxychloride (2·92 g., 76%), b. p. 106°/760 mm., n_D^{20} 1·4615 (Found: Cl, 77·6. Calc. for OCl_3P: Cl, 79·1%), and di-*n*-butylboron chloride (6·49 g., 87%), b. p. 68°/19 mm., n_D^{20} 1·4240 (Found: Cl, 22·1; B, 6·6%). There was a distillation residue (0·74 g.).

Pyridine Complexes (9) *of Di*-n-*butylboron Halides.*—(a) *Chloride.* Pyridine (1·49 g., 2 mols.) in *n*-pentane (10 c.c.) was added slowly with shaking to di-*n*-butylboron chloride (1·51 g., 1 mol.) in *n*-pentane (10 c.c.) at 20°. Considerable heat was evolved, and a white precipitate formed instantly. Volatile matter was removed at 20°/18 mm. and the residue washed with dry *n*-pentane to leave the white, crystalline, water-soluble 2 : 1 *pyridine complex* (2·92 g., 97%), m. p. 63—66° (Found: Cl, 11·1: B, 3·2; C_5H_5N, 49·5. $C_{18}H_{28}N_2ClB$ requires Cl, 11·2; B, 3·4; C_5H_5N, 49·7%). Di-*n*-butylboron chloride did not form a 1 : 1 pyridine complex whatever the temperature or the order of addition.

(b) *Bromide.* As in (a), but with addition at 0°, pyridine (1·32 g., 2 mols.) and di-*n*-butylboron bromide (1·70 g., 1 mol.) afforded the white, solid, water-soluble 2 : 1 *pyridine complex* (2·90 g., 96%), m. p. 125—132° (Found: Br, 22·3; B, 2·9; C_5H_5N, 43·4. $C_{18}H_{28}N_2BrN$ requires Br, 22·1; B, 3·0; C_5H_5N, 43·6%). Di-*n*-butylboron bromide did not form a 1 : 1 pyridine complex.

Hydrolysis (10 *and* 11) *of Di*-n-*butylboron Halides.*—This was carried out on an approximately 0·03 molar scale. An ethereal solution (5%) of the exact quantity of water was slowly added with shaking to the halide in dry *n*-pentane (10 c.c.) at 20°. Considerable heat of reaction and evolution of hydrogen halide were observed. Volatile matter was removed at 20°/10 mm. to leave the hydrolysis product as a residue; the results are shown in Table 2.

TABLE 2.

Reactant	Hydrolysis product	Yield (%)	B. p./mm.	M. p.* (decomp.)	n_D^{20}	Found (%) B	Calc. (%) B
Bu^n_2BCl	$Bu^n_2B{\cdot}OH$	99	—	55—63°	—	7·8	7·6
Bu^n_2BCl	$(Bu^n_2B)_2O$	94	93°/0·8	—	1·4264	7·9	8·1
Bu^n_2BBr	$Bu^n_2B{\cdot}OH$	90	—	59—62°	—	7·5	7·6
Bu^n_2BBr	$(Bu^n_2B)_2O$	93	130°/8	—	1·4265	7·9	8·1

* Since the acid is a viscous gel rather than a solid this is not a true m. p.

Interaction (12) *of Acetic Acid with Di*-n-*butylboronous Anhydride.*—Acetic acid (6·0 g., 2 mols., +3·9 g., excess) was added to di-*n*-butylboronous anhydride (4·54 g., 1 mol.) and the mixture heated under reflux (6 hr.) at 130—150°. A condensate of butane (1·68 g., 85%), characterised by means of gas-liquid chromatography by comparison with an authentic sample, b. p. −1°, was trapped at −80°. Excess of acetic acid (3·89 g., 100%) (Found: Equiv., 59·4. Calc. for $C_2H_4O_2$: equiv., 60·0) (also identified by gas-liquid chromatography) was the sole material removed at 20°/0·05 mm. and was collected at −80°. A solid, yellowish-white residue of *acetoxy*-n-*butylboronous anhydride* (4·53 g., 99%), m. p. 33—36°, was left (Found: B, 7·9; OAc, 41·5%; *M*, 234. $C_{12}H_{24}O_5B_2$ requires B, 8·0; OAc, 43·8%; *M*, 270). Its total acidity was 69·9 (Calc.: 67·4).

Hydrolysis (13) *of Acetoxy*-n-*butylboronous Anhydride.*—Water (1·10 g., 3 mols.) in dry ether (25 c.c.) was slowly added with shaking to the anhydride (5·46 g., 1 mol.) in dry ether (10 c.c.) at 20°. Solvent was removed at 20°/75 mm. and acetic acid (2·39 g., 99·2%) (Found: equiv., 59·6) was removed at 20°/0·05 mm. and collected at −80°. The residue was washed with *n*-pentane, to leave *n*-butylboronic acid (4·12 g., 99·3%), m. p. 93—94°, mixed m. p. 94° (Found: B, 10·7. Calc. for $C_4H_{11}O_2B$: B, 10·6%).

*Lack of Reaction between Di-*n*-butylboronous Anhydride* (1 *mol.*) *and Certain Reagents.*—(i) *Bases.* The anhydride was recovered in high yield from: (*a*) a mixture with diethylamine (2 mols. at 20° with heat of mixing; and 4 mols. after 4 hr. at 140—150°); and (*b*) after passage of ammonia gas (2 hr. at 0° in *n*-pentane solution). With pyridine (1 mol.) lack of interaction was indicated by molecular-weight determination (Found: *M*, 182. Calc. for 1 : 1 mixture: *M*, 172), although complete separation of the components by distillation was not achieved.

(ii) Thionyl chloride (4 mols.) did not react (15 min. at 60°) with the anhydride, which was recovered (93%).

(iii) Hydrogen chloride after being bubbled (30 min. at 20°, 100°, and 150°, severally), in absence or presence of anhydrous ferric chloride (1%), through the anhydride effected no reaction; the pure anhydride (80—95%) was recovered on distillation.

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