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BORYLATION OF SATURATED HETEROCYCLES WITH SEVERAL HETEROATOMS. (REVIEW)

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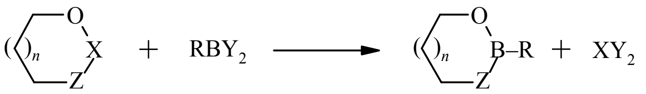
New examples of the borylation of a series of five- and six-membered saturated 1,3- and 1,3,2-heterocycles with the formation of cyclic boric esters are described. The probable mechanism of the reaction is discussed.

Keywords: cyclic boric esters, 1,3-dioxacycloalkanes, 1,3-dioxa-2-heterocycloalkanes, tetrahydro-1,3-oxazines, borylation, reaction mechanism, quantum chemistry.

A promising trend in the synthesis of cyclic boric esters, which are important reagents in organic chemistry, is the reaction of alicyclic boron substrates with saturated 1,3- or 1,3,2-heterocycles.



Z = O, NR; X = CR^1R^2, SiR^1R^2, S=O, $GePh_2$, As(O)Ph; Y = OR, Cl; *n* = 0, 1

Early papers on this subject, which appeared in the second half of the twentieth century, were fragmentary in nature and were devoted to the borylation of 1,3-dithia-3-silacyclopentanes [1, 2], bis(1,3-diphenylimidazolidin-2-ylidene) [3], substituted 1,3-dioxanes [4-7], and six-membered silicon- and nitrogen-containing heterocycles [8]. In the last decade, however, it has been possible to discover interesting new examples of such reactions, to investigate their occurrence and stereochemistry in detail, and to obtain information about their probable mechanism.

1. BORYLATION OF 1,3-DIOXANES

The borylation of 1,3-dioxanes **1** with the esters of monosubstituted boric acids leads to the formation of 1,3,2-dioxaborinanes **2** and acyclic acetals. Cyclic formals, acetals, and ketals enter into this reaction [9-12]. In a number of cases the reaction begins even at room temperature when equimolar amounts of the initial reagents are mixed. The degree of conversion of compounds **1** is increased considerably by heating at 130°C in the presence of catalytic amounts of $ZnCl_2$ or $BF_2{\cdot}OEt_2$. The degree of transformation of 1,3-dioxanes depends on the degree of substitution of the acetal carbon atom (Table 1).

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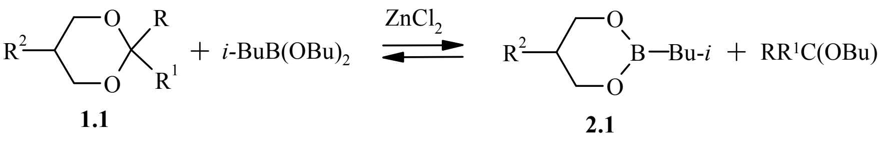
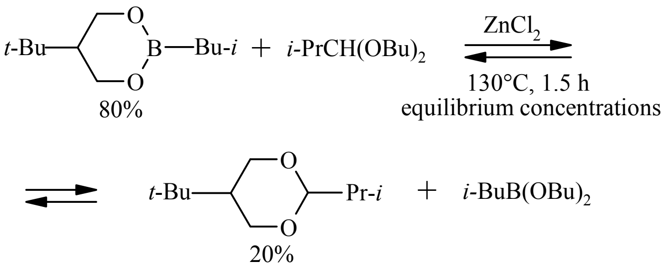
TABLE 1. The Ratio of 1,3-Dioxanes **1.1** and 1,3,2-Dioxaborinanes **2.1** According to GLC, mole %, in the Reaction Mixture

R	R^1	R^2	Immediately after mixing	After standing with $ZnCl_2$ for 2 h	Heating with $ZnCl_2$ at 130°C	
					5 min	30 min
H	H	*i*-Bu	100:0	100:0	75:25	60:40
H	H	*t*-Bu	100:0	100:0	60:40	50:50
H	H	C_7H_{15}	100:0	100:0	100:0	31:69
H	H	C_9H_{19}	100:0	70:30	45:55	7:93
H	H	$C_{12}H_{25}$	100:0	80:20	20:80	14:86
Pr	H	*i*-Bu	100:0	65:35	17:83	0:100
i-Bu	H	*i*-Bu	100:0	60:40	20:80	0:100
Ph	H	*i*-Bu	0:100	—	—	—
i-Pr	H	Me	100:0	82:18	72:28	45:55
i-Pr	H	*i*-Pr	100:0	85:15	70:30	42:58
Me	Me	*i*-Bu	100:0	60:40	0:100	—
Et	Me	*i*-Bu	82:18	57:43	0:100	—
i-Bu	Me	*i*-Bu	85:15	55:45	0:100	—
t-Bu	Me	*i*-Bu	90:10	60:40	10:90	0:100
Ph	Me	*i*-Bu	0:100	—	—	—
$(CH_2)_5$		*i*-Bu	31:69	0:100	—	—



Investigation of the reverse reaction for the reaction of isopropyldibutylacetal with 5-*tert*-butyl-2-isobutyl-1,3,2-dioxaborinane indicated that the equilibrium was displaced toward the cyclic boric ester [10].



It is known that the degree of conversion under the conditions of equilibrium is related to the relative thermodynamic stability of the initial and final compounds. Determination of the latter (in the form of the so-called strain energy of the ring [13, 14]) showed that 1,3,2-dioxaborinanes are more stable than 1,3-dioxanes [15, 16]. It is quite difficult to determine the general stability of all the reaction components on account of the unknown thermodynamic data for the acyclic derivatives. Nevertheless, it can be seen that the displacement of the equilibrium toward 1,3,2-dioxaborinanes is due to the greater degree of stabilizing *n*–π-electronic interaction at the endocyclic B–O bond compared with the acyclic boric esters [7, 16]

The relative reactivity of the 1,3-dioxanes at the kinetic stage of the process is due to the stability of the intermediates. In [17] a computer model of the mechanism of the reaction unsubstituted and also 2- and 2,3-substituted 1,3-dioxanes with dihydroxyborane, methylboric acid, and its dimethyl ester was produced by the AM1 method [18, 19] using HyperChem software [20] (Table 2). Here it was assumed that the reaction

TABLE 2. The Relative Stability of the Ion **B** (kcal/mol) and the Ratio of Compounds **1.2** and **2.2** According to GLC

R	R^1	$E_B–E_A$	The ratio of (**1.2**) and (**2.2**), %, in the presence of $ZnCl_2$*	
			After 2 h at 20°C	After 5 min at 130°C
			1.2 + $HB(OH)_2 \rightarrow$ **2.2a**	
H	H	7.8	100 : 0	80 : 20
i-Pr	H	4.5	82 : 18	72 : 28
i-Bu	H	3.7	60 : 40	20 : 80
Ph	H	2.6	0 : 100	0 : 100
Me	Me	3.7	60 : 40	0 : 100
Me	Et	2.9	57 : 43	0 : 100
Me	*i*-Bu	1.3	55 : 45	0 : 100
Me	*t*-Bu	3.7	60 : 40	10 : 90
$(CH_2)_5$		0.8	0 : 100	0 : 100
Me	Ph	1.1	0 : 100	0 : 100
			1.2 + $MeB(OH)_2 \rightarrow$ **2.2b**	
H	H	8.6	100 : 0	80 : 20
i-Pr	H	3.9	60 : 40	20 : 80
Ph	H	0.5	0 : 100	0 : 100
			1.2 + $MeB(OMe)_2 \rightarrow$ **2.2b**	
i-Pr	H	9.9	60 : 40	20 : 80
Ph	H	7.4	0 : 100	0 : 100

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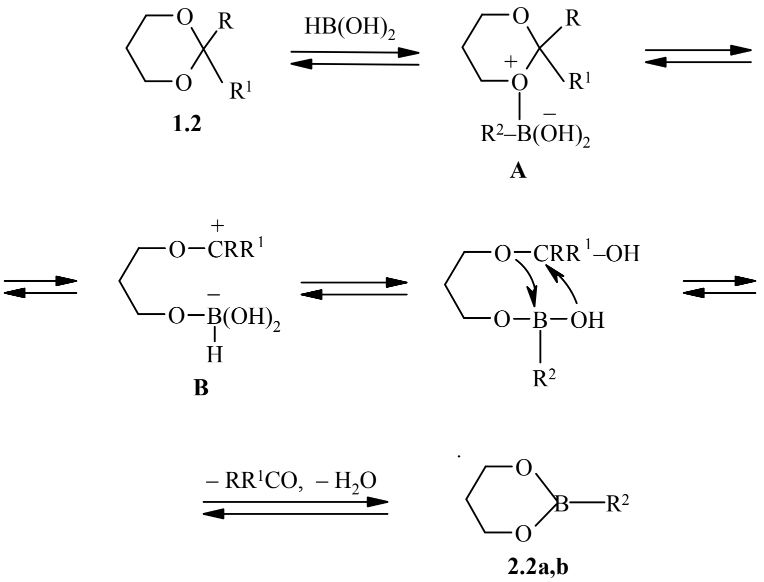
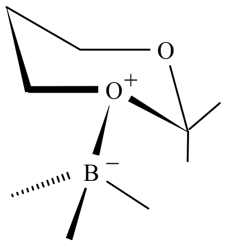
* According to data from [9-12].

obeys the main relationships of the heterolytic reactions of 1,3-dioxacycloalkanes [21] and takes place through a stage involving coordination of the acetal oxygen of 1,3-dioxane (**1.2**) at the boron atom with the formation of the complex **A**, which slowly isomerizes to the bipolar ion **B**. The latter is converted by eliminating a molecule of the acyclic acetal into the final 1,3,2-dioxaborinane (**2.2**).

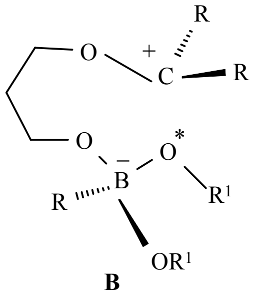


a = R^2 = H; **b** R^2 = Me

In all probability the catalysts $ZnCl_2$ or $BF_3 \cdot OEt_2$ facilitate the formation of structures **A** and **B**. According to the calculated results, the energy minimum for structure **A** corresponds to the equatorial conformer (0.4-2.6 kcal/mol more stable than the axial form), while the main minimum of structure **B** corresponds to the form with close acetyl and boric ester fragments.



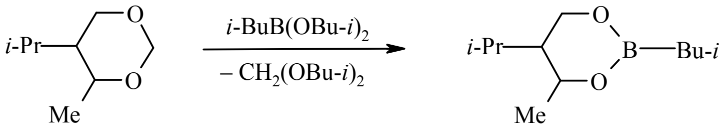
A



B

The calculated length of the B–O coordination bond amounts to 1.83-2.00 Å. According to the data in Table 2, increase in the degree of substitution of the acetal carbon atom increases the stability of ion **B** compared with the oxonium complex **A** (the difference $E_{\mathbf{B}} - E_{\mathbf{A}}$ decreases). In parallel with this the content of the cyclic boric ester **2** in the mixture in the kinetically controlled region is increased. The most stable is structure **B** based on 2-methyl-2-phenyl-1,3-dioxane (R = Me, R^1 = Ph); according to experimental data, the reaction in this case goes without a catalyst [11].

The *cis* and *trans* isomers of 2,5-dialkyl-1,3-dioxanes have identical reactivity toward the acyclic boric esters; their ratio, determined earlier in [22], does not change in the range between 30:70 and 40:60 respectively [9, 10]. However, the position changes in the case of 4,5-dialkyl-1,3-dioxanes, the stereochemistry of which was investigated in [23]; according to GLC, *cis*-4-methyl-5-isopropyl-1,3-dioxane reacts with the acyclic boric substrate more rapidly that the *trans* form (Table 3) [24].



1.3 **2.3**

TABLE 3. The Variation of the Stereoisomeric Composition of the Initial and Final Compounds **1.3** and **2.3** during the Reaction of 5-Isopropyl-4-methyl-1,3-dioxane with Isobutylboric Ester

cis:*trans*-**1.3**	Position on reaction coordinate	*cis*:*trans*-**2.3**	**1.3** : **2.3**, %
60:40	Before beginning of reaction	—	100:0
45:55	After 24 h without catalyst	67:33	85:15
37:63	After heating for 30 min with $ZnCl_2$*	65:35	12:88
47:53	After heating for 30 min with $BF_3 \cdot OEt_2$*	63:37	14:86
39:61	Before beginning of reaction	—	100:0
11:89	After 24 h without catalyst	47:53	71:29
9:91	After heating for 30 min with $ZnCl_2$*	55:45	11:89
11:89	After heating for 30 min with $BF_3 \cdot OEt_2$*	37:63	9:91

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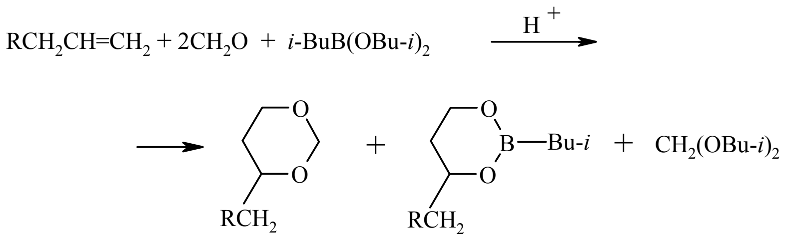
* Control tests indicated that configurational isomerization of compounds **1.3** and **2.3** under the influence of the catalyst is impossible [24].

Study of the transformation of two samples of the initial 1,3-dioxane with different configurational composition [23, 25] showed that the *cis* isomer reacted more quickly than the *trans* isomer in both cases; here the borylation is strictly stereospecific; the *cis* isomer is only transformed into the *cis* form of 1,3,2-dioxaborinane while the *trans* form of the cyclic boric ester is formed exclusively from the *trans* isomer. (The stereochemistry of 2-isobutyl-5-isopropyl-4-methyl-1,3,2-dioxaborinane was studied in detail in [26].) Computer modelling of the transformation showed that the increased reactivity of the *cis* isomer of 1,3-dioxane is due to the lower activation energy of this process [24].

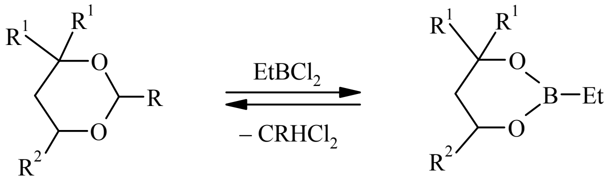
The reaction of a series of olefins with paraformaldehyde and diisobutyl isobutylborate under the conditions of the Prins reaction (catalyst, conc. sulfuric acid) leads to 2,4-dialkyl-1,3,2-dioxaborinane **2.4**, the acyclic formal, and 4-alkyl-1,3-dioxane **1.4** in amounts comparable with the ester **2.4** [27].



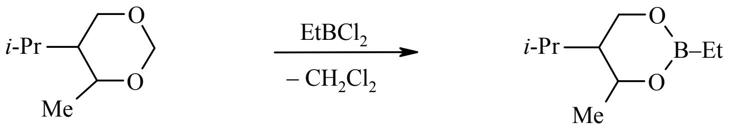
1.4 **2.4**

R = i-Pr, Bu, C_5H_{11}

The borylation of 1,3-dioxanes with alkyldichloroboranes also leads to the corresponding 1,3,2-dioxaborinanes [28]; the reaction here takes place at room temperature and without catalysts.



As in the previous case, the borylation rate of the *cis* isomer of the stereoisomeric 5-isopropyl-4-methyl-1,3-dioxane is appreciably higher than that of the *trans* form [29].



cis:trans = 67:43	before the reaction	—
cis:trans = 50:50	after 24 h	88:12

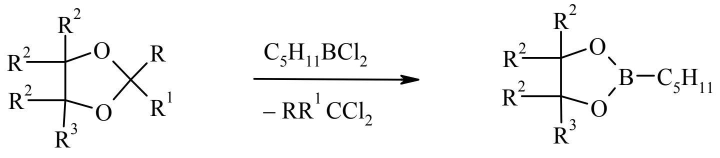
At the same time the reaction is stereospecific.

2. BORYLATION OF 1,3-DIOXOLANES

The reaction of amyldichloroborane with 1,3-dioxolanes **3** leads to the corresponding 1,3,2-dioxaborolanes **4.1** (Table 4) [30].

TABLE 4. The Ratio of 1.3-Dioxolanes **3** and 1,3,2-Dioxaborolanes **4.1**, According to GLC, 6 h after the Beginning of the Reaction

R	R^1	R^2	R^3	**3** : **4.1** mol. %
H	H	H	H	0 : 100
i-Pr	H	H	H	79 : 21
C_7H_{15}	H	H	H	43 : 57
H	H	Me	Me	70 : 30
Me	Me	H	Me	44 : 56
=O		H	Me	>98 : <2

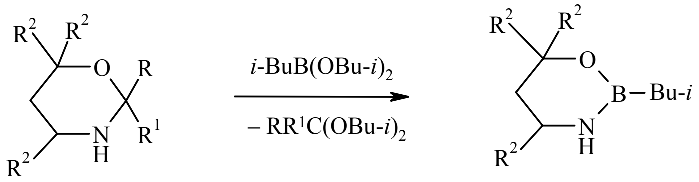


3 **4.1**

The obtained data indicate that the degree of conversion of the 1,3-dioxolanes depends substantially on the nature and position of the substituents. As in the case of 1,3-dioxanes it can be supposed that the observed relationship is due to the relative stability of the respective intermediates.

3. BORYLATION OF TETRAHYDRO-1,3-OXAZINES

The borylation of tetrahydro-1,3-oxazines **5** with diisobutyl isobutylborate takes place slowly when a mixture of the initial compounds is heated to 100-150°C and, for individual compounds, takes place at room temperature in the presence of catalytic amounts of zinc chloride. As in the case of 1,3-dioxanes, the ketals have higher rates and degrees of conversion that the acetals (Table 5). Increase in the number of substituents in the aliphatic part of the ring has practically no effect on the degree of conversion of the oxazines; at the same time the presence of a secondary radical at the acetal carbon atom appreciably reduces the equilibrium concentration of the respective 1,3,2-oxazaborinanes **6** [31].



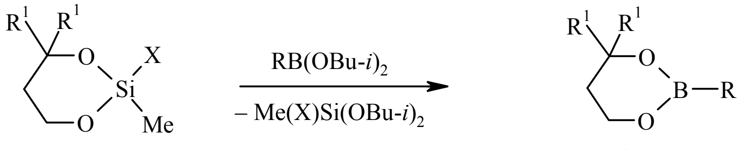
5 **6**

4. BORYLATION OF 1,3-DIOXA-2-SILACYCLOHEXANES

The borylation of 1,3-dioxa-2-silacyclohexanes **7** with an equimolar amount of the alkylboric esters also leads to 1,3,2-dioxaborinanes. The degree of conversion of the cyclic silicate esters here depends on the nature of the substituent X [32].

TABLE 5. The Ratio of Tetrahydro-1,3-oxazines **5** and 1,3,2-Oxazaborinanes **6**, According to GLC, 6 h after the Beginning of the Reaction

R	R^1	R^2	**5 : 6**, mol %
i-Pr	H	H	66 : 34
Me	Me	H	5 : 95
Et	H	Me	14 : 86
Pr	H	Me	12 : 88
i-Pr	H	Me	63 : 37
Me	Me	Me	0 : 100



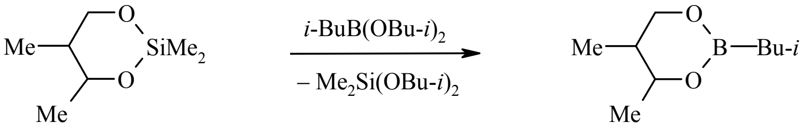
7a–f **2.5**

R = Me, *i*-Bu

7 a X = Me, R^1 = H; **b** X = R^1 = Me; **c** X = Ph, R^1 = Me; **d** X = OAc, R^1 = Me;
e X = NEt_2, R^1 = H; **f** X = NEt_2, R^1 = Me

The reaction of the alkyl-substituted derivatives **7a**,**b** takes place almost instantaneously, while the ester **7c** has a lower reaction rate; without the catalyst the equilibrium ratio of this compound and the corresponding 1,3,2-dioxaborinane **2.5** amounted to 65:35. Only heating at 100°C in the presence of zinc chloride for 5 min shifted the equilibrium almost completely toward the cyclic boric ester. On the whole the reaction is characterized by a larger equilibrium concentration of the desired compounds (the substituted 1,3,2-dioxaborinanes) than the silylation of 1,3-dioxanes with dimethyldimethoxysilane [33, 34].

Study of the stereochemistry of the borylation of cyclic silicate esters for compound **7g** (a mixture of stereoisomers with various compositions [35]) showed that the reaction was stereospecific [36, 37]; within the GLC error limits (±3%) the ratio of the *cis* and *trans* isomers of **7g** corresponds to the ratio for the expected 2-isobutyl-4,5-dimethyl-1,3,2-dioxaborinane. The highest degree of conversion in the presence of catalytic amounts of boron trifluoride etherate in this case amounted to 95%.

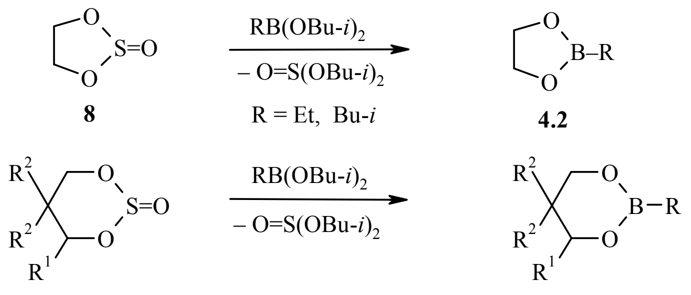


7g **2.6**

cis:trans = 50:50 *cis:trans* = 50:50
cis:trans = 70:30 *cis:trans* = 70:30

5. BORYLATION OF CYCLIC SULFITES

The reaction of 1,3,2-dioxathiolane 2-oxide (**8**) and 1,3,2-dioxathiane 2-oxides **9a**,**b** with alkylboric esters leads to the corresponding 1,3,2-dioxaborolanes **4.2** and 1,3,2-dioxaborinanes **2.7**.

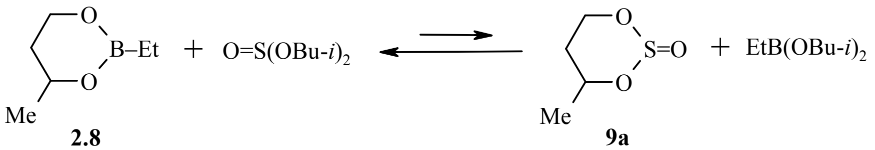


8 → **4.2**

9a,b → **2.7**

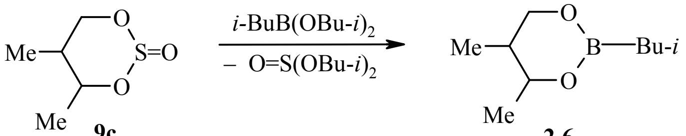
a R^1 = Me, R^2 = H; **b** R^1 = H, R^2 = Me

The reaction hardly goes at all at room temperature. On heating to 140°C, however, a constant ratio of the concentrations of compounds **8** and **4.2** is established after 75-80 min, while for compounds **9a**,**b** and **2.7** a constant ratio is established after 40-60 min. The transformation does not require catalysts, and the degree of conversion of the cyclic sulfites in all cases amounts to 90-95% [38, 39]. Study of the reverse reaction for the case of diisobutyl sulfite and 4-methyl-2-ethyl-1,3,2-dioxaborinane showed that the equilibrium is displaced almost completely toward the formation of the cyclic boric esters; the degree of conversion to the sulfite **9a** does not exceed 6%.



2.8 **9a**

The reaction is stereospecific; borylation of the sulfite **9c** (as a mixture of stereoisomers, data on the configurational assignment are given in [40]) leads to the corresponding compound **2.6** with analogous stereoisomeric composition, although *cis*-**9c** reacts more quickly than *trans*-**9c** [41].

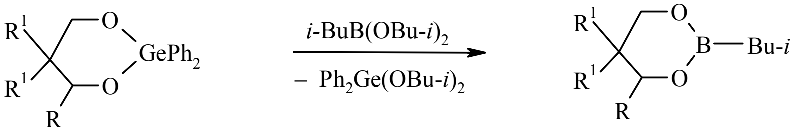


9c → **2.6**

9c	Reaction time, h	**2.6**
cis:trans = 50:50	—	—
	1.5	*cis:trans* = 75:25
	3.0	*cis:trans* = 50:50

6. BORYLATION OF 1,3-DIOXA-2-GERMACYCLOHEXANES

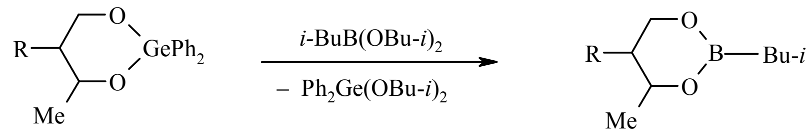
The reaction of 2,2-diphenyl-1,3-dioxa-2-germacyclohexanes **10a-c** with isobutylboric ester does not require the presence of a catalyst and leads to the corresponding 1,3,2-dioxaborinanes [42].



10a–c → **2.9**

a R = R^1= H; **b** R = Me, R^1= H; **c** R = H, R^1= Me

However, the yield of the esters **2.9**, isolated by fractional vacuum distillation, amounted to between 29 and 32%. Like the processes discussed above, this reaction is stereospecific [37, 43]; the stereoisomeric composition of the esters **10d**,**e** corresponds approximately to the composition for the borylation products 2,4,5-substituted 1,3,2-dioxaborinanes [44-49].



10d,e **2.6**

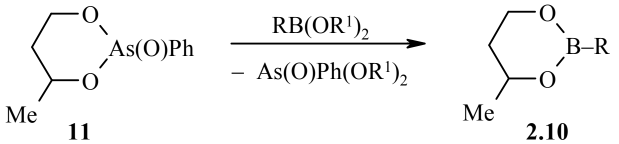
d R = Me, **e** R = *i*-Pr

d *cis:trans* = 50:50 *cis:trans* = 50:50
e cis:trans = 70:30 *cis:trans* = 82:18

The degree of conversion of compounds **10d**,**e** does not exceed 30%.

7. BORYLATION OF 2-ARSENACYCLOHEXANES

The reaction of the cyclic ester of phenylarsonic acid **11** with the acyclic derivatives of boric acids also led to the corresponding 1,3,2-dioxaborinanes [50]. The cyclic esters **2.10** were isolated with yields of 15-41%.



11 **2.10**

R = Bu-*i*, R^1 = OH; R = OBu-*i*, R^1 = Bu-*i*

Thus, the borylation of six-membered heterocycles containing heteroatoms of period V (Ge, As) are characterized by relatively low conversion into 1,3,2-dioxaborinanes.

The investigated borylation reactions supplement the familiar chemical transformations in various types of saturated heterocycles [7, 21, 33, 51-56] and demonstrate that a heteroatom at the second position of the ring has an appreciable effect on the degree of transformation to cyclic boric esters.

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