Reviews

Reactions of 1,3-dioxacycloalkanes and their 2-arsena, 2-bora, 2-germa, 2-sila, and 2-thia analogs with nitriles

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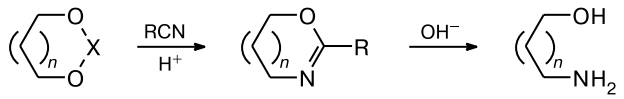
New reactions of five-, six-, and seven-membered 1,3-dioxacycloalkanes and their 2-arsena, 2-bora, 2-germa, 2-sila, and 2-thia analogs with nitriles giving rise to 1,3-oxazacycloalkanes and then to amino alcohols are surveyed.

Key words: 1,3-dioxa-2-carba-, 1,3-dioxa-2-arsena-, 1,3-dioxa-2-bora-, 1,3-dioxa-2-germa-, 1,3-dioxa-2-sila-, 1,3-dioxa-2-thiacycloalkanes, reaction with nitriles, reaction mechanism, amino alcohols, quantum chemical calculations.

One of methods for the preparation of 1,3-amino alcohols is based on alkaline hydrolysis of 4*H*-5,6-dihydro-1,3-oxazines.[1] The diverse approaches to the latter have been characterized in detail in reviews.[2,3] In particular, these compounds can be prepared by reactions of nitriles with 1,3-diols (a modification of the Ritter reaction[2—9]) or oxetanes.[10—14] However, both diols and oxetanes are accessible with difficulty, which substantially limits the use of this route. Moreover, this method can be used only for the preparation of 1,3-amino alcohols. More than 10 years ago, it has been shown[15] that 4*H*-5,6-dihydro-1,3-oxazines could be obtained in preparative yields by a reaction of acetonirile with 1,3-dioxanes, the well-known and readily accessible (for example, by the Prins reaction) representatives of saturated six-membered oxygen-containing heterocycles. More recently, it has been demonstrated that 1,2-, 1,3-, and 1,4-amino alcohols can be synthesized by the reactions of five-, six-, and seven-membered 1,3-dioxacycloalkanes or their analogs containing a heteroatom at position 2 with nitriles[16—18] (Scheme 1).

Scheme 1



X = CHR, S=O, $SiMe_2$, $GePh_2$, As(O)Ph, B—R; *n* = 0, 1, 2

The reactions are carried out in the presence of mineral acids. 4*H*-5,6-Dihydro-1,3-oxazines, 2-oxazolines, and 3,4-dihydro-1,3-benzooxazepines are isolated by treatment of the corresponding salts with an aqueous LiOH

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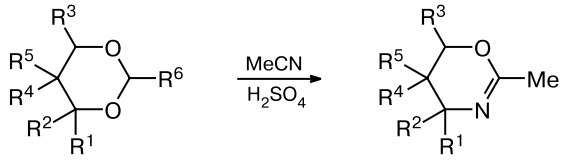
solution under mild conditions. Subsequent alkaline hydrolysis of these heterocycles (refluxing in 20% NaOH) affords the final amino alcohols.

1. Reactions of 1,3-dioxanes with nitriles

The reaction of 1,3-dioxanes (**1**) with acetonitrile produces 2-methyl-4*H*-5,6-dihydro-1,3-oxazines (**2**) (Scheme 2).[15,19–21] The yield of oxazines **2** is determined primarily by the degree of substitution at the C(4) atom. The absence of substituents or the presence of only one alkyl group prevents the formation of the target compounds.[20]

Scheme 2



1 **2**

R^1, R^4, R^5, R^6 = H, Alk; R^2 = Me, Ph; R^3 = Me, H

However, the corresponding oxazines can be isolated in preparative yields from compounds **1** with R^2 = Ph and R^3 = H or R^2 = R^3 = Me. The presence of an electron-withdrawing substituent substantially decreases the yield of the reaction product. The yields of oxazines **2** depend only slightly on the nature and the number of substituents at the C(2) and C(5) atoms in the 1,3-dioxane ring.

This transformation follows the rule for the reactions of nitriles with electrophilic reagents[22,23] and, at the same time, corresponds to the general views about the mechanism of solvolytic reactions of 1,3-dioxacycloalkanes.[24–27]

Table 1. Relative stabilities of the carbocation **B** (AM1, E/kcal mol^{-1}) and yields of oxazines **2** in reactions of 1,3-dioxanes with acetonitrile[28]

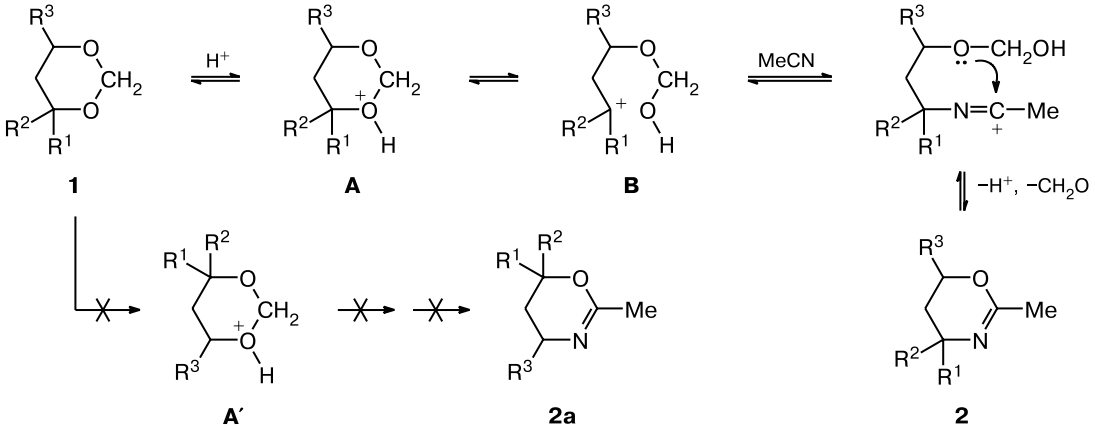
R^1	R^2	R^3	ΔE_{AB}	Yield of **2** (%)
Me	H	H	12.4	3
Me	Me	H	0.5	65
Me	CH_2Cl	H	2.4	9
Me	Me	Me	1.3	80
Ph	H	H	−1.9	46
Ph	Me	H	−9.3	98

Hence, a probable mechanism of the reaction under consideration involves the initial formation of an oxonium ion **A**. This is slowly isomerized to give the carbocation **B**, which then adds the acetonirile molecule (Scheme 3). It was shown[26] that the thermal effect of protonation is virtually identical for different 1,3-dioxanes. Thus, this is not the rate-limiting step. At the same time, the absence of an isomeric oxazine **2a** suggests that the degree of conversion of dioxanes **1** depends on the stability of the carbocation **B**. This fact is confirmed by calculations of the relative energies of the ions **A** and **B** by the SCF LCAO MO method using the AM1 parametrization.[28]

The results of calculations demonstrate that the relative stability of the ion **B** (the energy difference between the most stable conformers **A** and **B**, $\Delta E_{AB} = E_B - E_A$) increases with an increase in the number of the methyl groups or with the presence of the phenyl substituent at the C(4) atom of the starting 1,3-dioxane (a decrease in ΔE_{AB}) with a simultaneous increase in the yield of oxazine **2** (Table 1).

Based on these data, one can reliably expect rather high preparative yields of 4*H*-5,6-dihydro-1,3-oxazines from 1,3-dioxanes containing *gem*-dialkyl groups at the C(4) atom of the ring.

Scheme 3

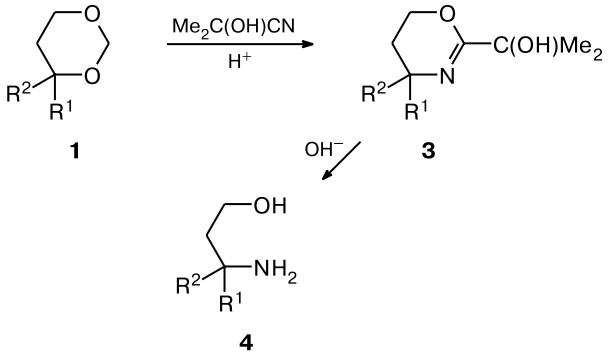


1 **A** **B** **A′** **2a** **2**

R^1 = R^2 = Me; R^1 = Ph, R^2 = H, Me; R^3 = H, Me

The reactions of 4-phenyl- and 4,4-dimethyl-1,3-dioxanes with acetone cyanohydrin give the corresponding 2-(1-hydroxy-1-methylethyl)-5,6-dihydro-1,3-oxazines **3** (Scheme 4) as the primary products.[19,29]

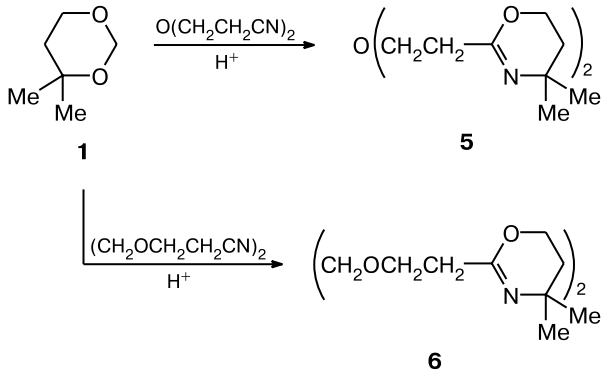
Scheme 4



Subsequent alkaline hydrolysis afforded the corresponding amino alcohols **4**.

The reactions of 4,4-dimethyl-1,3-dioxane with dinitriles (*e.g.*, bis(2-cyano)diethyl ether and 1,2-bis(2-cyanoethoxy)ethane) occur analogously (Scheme 5).[30]

Scheme 5



Derivatives **5** and **6** were obtained in 10% yield. The IR spectra of these compounds have an intense band ν(C=N) at 1650 cm^{-1}. In addition, their compositions and structures were confirmed by ^1H NMR spectroscopy and mass spectrometry.[30]

A substantial decrease in the yields of compounds **5** and **6** in this reaction as compared with that in the reaction of dioxane **1** with acetonitrile is presumably due to various side polymerization reactions involving dinitriles[22] as well as to hydrolytic instability of oxazines **5** and **6**. Appreciable amounts of 3-amino-3-methylbutan-1-ol were detected in the reaction products by GLC. This compound is produced in 70—74% yields upon hydrolysis of compounds **5** and **6** with an aqueous alkali.

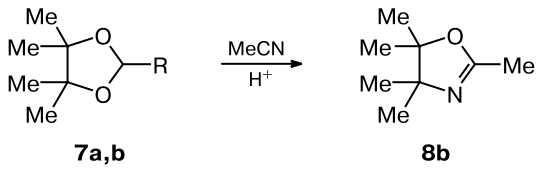
Table 2. Yields of 2-oxazoline **8b** and the degrees of conversion of 1,3-dioxolanes **7a,b** depending on the reaction conditions[31]

7	Reaction conditions			Yield of **8b** (%)	Conversion of **7** (%)
	Acid	Solvent	*T*/°C		
a	H_3PO_4	excess MeCN	20	Traces	0
	H_3PO_4	excess MeCN	80	Traces	0
	H_2SO_4	excess MeCN	20	40	56
	H_2SO_4	excess MeCN	80	7	95
	H_2SO_4	$CHCl_3$	60	9	35
b	H_2SO_4	excess MeCN	20	8	90

2. Reactions of 1,3-dioxolanes with acetonitriles

Five-membered analogs of 1,3-dioxanes, *viz.*, substituted 1,3-dioxolanes, react with nitriles in a similar way to afford 2-methyl-2-oxazolines as the major products. The reactions of 4,4,5,5-tetramethyl- and 2-ethyl-4,4,5,5-tetramethyl-1,3-dioxolanes (**7a,b**) with acetonitrile produce 2,4,4,5,5-pentamethyl-2-oxazoline (**8b**) (Scheme 6).[17,31] The yield of the latter depends substantially on the reaction conditions (Table 2).

Scheme 6



The maximum yield of oxazoline **8b** was 40%. Unsubstituted and 4-methyl-substituted 1,3-dioxolanes did not react with acetonitrile.

As in the case of 1,3-dioxanes, the observed regularities can be associated with the stability of the probable intermediate, *viz.*, the carbocation **B″** ($E_{B''} - E_{A''} = \Delta E_{A''B''}$, Table 3, Scheme 7).

The stability of the carbocation **B″** noticeably increases if the C(4) atom in the starting heterocycle bears two methyl groups. Hence, it can be expected that the corresponding oxazolines can be produced in preparative yields (40% and higher) starting already from 4,4-dimethyl-1,3-dioxolane.[31]

Alkaline hydrolysis of oxazoline **8** affords 3-amino-2,3-dimethylbutan-2-ol in 68% yield.[32]

Scheme 7

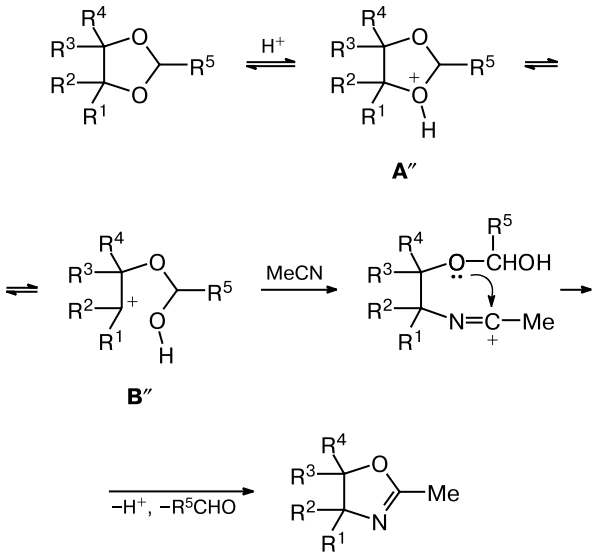


Table 3. Relative stabilities of the ion **B″** (AM1, E/kcal mol^{-1})

R^1	R^2	R^3	R^4	R^5	$\Delta E_{A''B''}$
H	H	H	H	H	21.8
Me	H	H	H	H	6.4
Me	Me	H	H	H	–3.7
Me	Me	Me	H	H	–2.5
Me	Me	Me	Me	H	–0.4
Me	Me	Me	Me	Me	1.1
Me	Me	Me	Me	Et	2.8

This reaction expands the range of chemical transformations of substituted 1,3-dioxolanes and along with the known procedures[33,34] provides an additional approach to the synthesis of 2-oxazolines.

3. Reactions of 1,3-dioxa-2-silacycloalkanes with acetonitrile

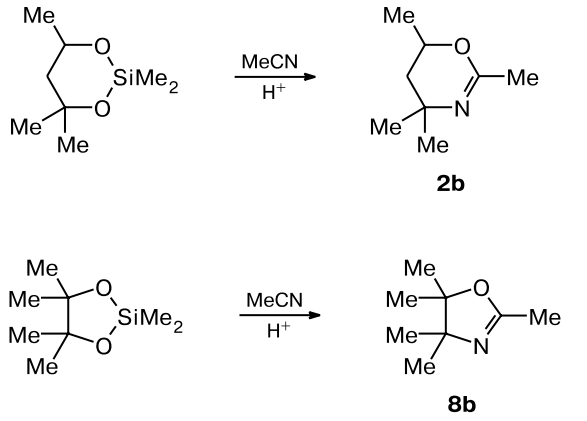
The reaction of 2,2,4,4,6-pentamethyl-1,3-dioxa-2-silacyclohexane with acetonitrile also produces the corresponding 2-methyl-5,6-dihydro-1,3-oxazine (**2b**) (Scheme 8).[35]

The yield of oxazine **2b** was 72%, whereas the reaction with the five-membered analog gave 2-oxazoline **8b** in a yield of at most 26%.[36]

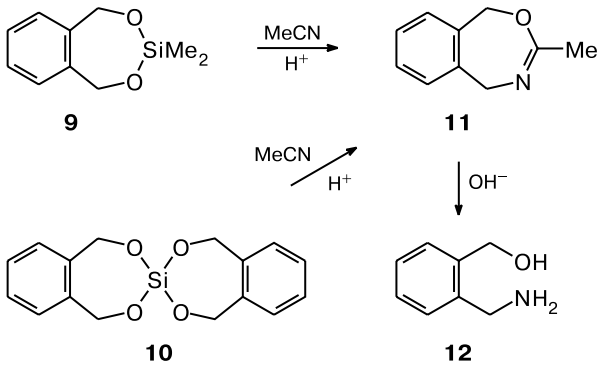
The reactions of siladioxepanes, *viz.*, 5,6-benzo-2,2-dimethyl-1,3-dioxa-2-silacycloheptane (**9**) and 3,4,10,11-dibenzo-1,6,8,13-tetraoxa-7-silaspiro[6.6]tridecane (**10**), with acetonitrile produce 2-methyl-4,7-dihydro-5,6-benzo-1,3-oxazepine (**11**) (Scheme 9).[18,37]

Hydrolysis of oxazepine **11** affords amino alcohol **12** in 44% and 49% yields (from **9** and **10**, respectively).

Scheme 8



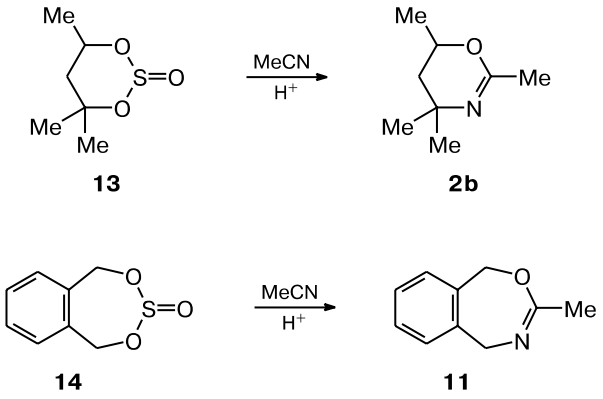
Scheme 9



4. Reactions of cyclic sulfites with acetonitrile

The reaction of 4,4,6-trimethyl-1,3,2-dioxathiane 2-oxide (**13**) with acetonitrile gives the expected oxazine **2b** in 80% yield (Scheme 10).[38]

Scheme 10



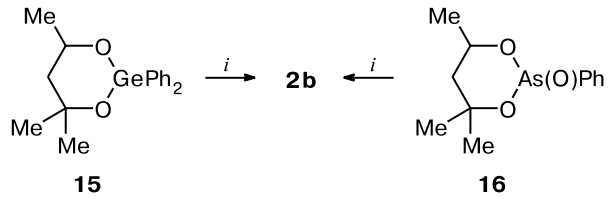
By contrast, the yield of the corresponding oxazine from 4-methyl-1,3,2-dioxathiane 2-oxide was at most 3%. Thus, this reaction obeys the general regularities typical of all the heterocyclic compounds under consideration.

Using benzo-1,3,2-dioxathiepine 2-oxide (**14**) as an example, it was demonstrated that seven-membered cyclic sulfites undergo analogous transformations (see Scheme 10), benzooxazepine **11** being the reaction product (yield 64%).[18,39]

5. Reactions of 1,3-dioxa-2-germa- and 2-arsenacyclohexanes with acetonitrile

Similar compounds containing other Group IV and V elements as a heteroatom at position 2 of the ring also react with acetonitrile. For example, the reaction of 4,4,6-trimethyl-2,2-diphenyl-1,3-dioxa-2-germacyclohexane (**15**) affords oxazine **2b** in 32% yield (Scheme 11).[18,40]

Scheme 11



i. MeCN, H^+.

Presumably, the reaction also gives a germanium-containing polymer or oligomer.

The yield of oxazine **2b** in the reaction of 2-oxo-4,4,6-trimethyl-2-phenyl-1,3-dioxa-2-arsenacyclohexane (**16**) did not exceed 30% as well.[41]

The absence of arsenic derivatives in the organic phase suggests the possible formation of sodium phenylarsonate in the step of treatment of oxazine hydrogensulfate with an alkali. A comparison of the yields of oxazine **2b** in reactions of the corresponding 1,3-dioxanes, cyclic sulfites, and 1,3-dioxa-2-silacyclohexane with the yields obtained from compounds **15** and **16** shows that the degree of conversion of 1,3-dioxa-2-heteracyclohexanes into dihydrooxazines depends substantially on the nature of the leaving group.

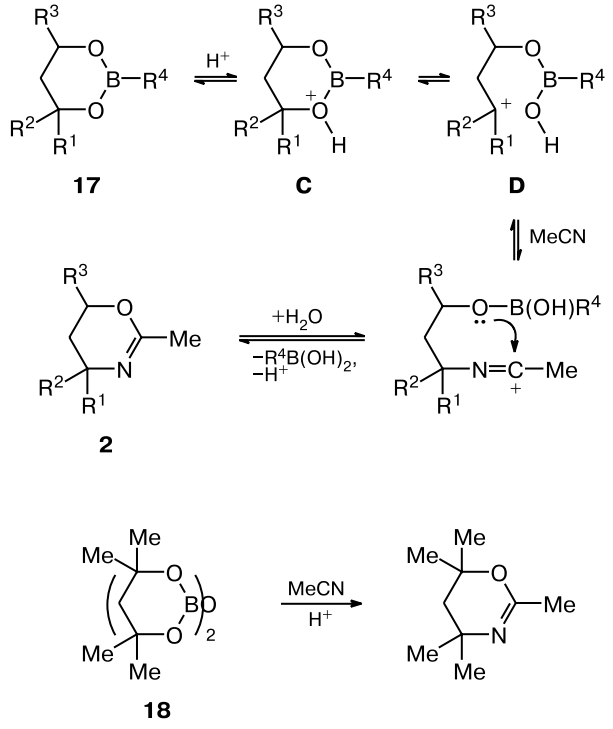
6. Reactions of cyclic boronates and borates with nitriles

The above-described reactions of heterocycles with nitriles are also typical of cyclic boronates and borates (**17**) (Scheme 12).[16—18] The yields of 1,3-oxazines **2** in the reactions of 1,3,2-dioxaborinanes with acetonitrile depends on both the number of C-alkyl groups and the nature of the substituent at the boron atom. The corresponding results[42,43] are presented in Table 4.

Table 4. Relative stabilities of the intermediates **D** (AM1, E/kcal mol^{-1}) and yields of oxazines **2** in reactions of substituted 1,3,2-dioxaborinanes **17** with acetonitrile

$R^1 = R^2$	R^3	R^4	$E_D - E_C$	Yield of **2** (%)
H	H	Me	30.7	0
H	Me	Me	12.6	0
H	Me	OH	6.5	≤2 (GLC)
Me	H	Me	−1.8	46
Me	H	OH	−6.2	62
Me	Me	Me	−1.0	42
Me	Me	OH	−5.5	59

Scheme 12

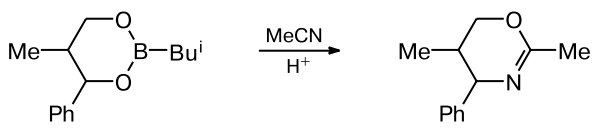


As in the case of 1,3-dioxanes, the absence of geminal alkyl substituents at the C(4) atoms in the starting heterocycle results in poor yields of the corresponding oxazines irrespective of the substituent at the boron atom. At the same time, the yield of dihydrooxazine in the reaction of 2,2′-oxybis(4,4,6,6-tetramethyl-1,3,2-dioxaborinane) (**18**) with acetonitrile is at most 23%.[44]

The ratio of the *cis* to *trans* isomers of the reaction product of 5-methyl-4-phenyl-1,3,2-dioxaborinane **19** differs substantially from the stereoisomeric composition of the starting ester[45] (Scheme 13).

This suggests that the mechanism of this reaction is in principle similar to that considered above for 1,3-dioxanes.

Scheme 13

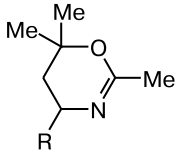


19 (83%)

cis : *trans* ≈ 4 : 1 *cis* : *trans* = 1 : 1

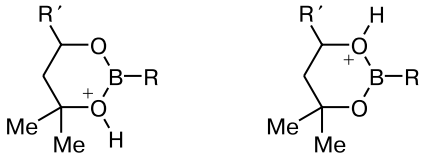
The probable reaction pathway was estimated by the AM1 method (see Table 4).[46] This pathway involves protonation of the starting borate giving rise to the oxonium ion **C**, which is slowly isomerized into the carbocation **D**, and the addition of the acetonitrile molecule then gives the final oxazine (see Table 4).

It is easily seen that, in the context of the scheme under discussion, the inertness of 1,3,2-dioxaborinanes, which either contain no substituents or contain one substituent at the C(4) atom, is associated with relative instability of the carbocationic intermediate **D** ($\Delta E_{CD} > 0$). On the contrary, esters containing the *gem*-dimethyl groups at the C(4) atom are characterized by relative stability of the ion **D** ($\Delta E_{CD} < 0$) and rather high yields of dihydrooxazines. The presence of the OH group instead of the methyl substituent at the boron atom increases the stability of the intermediate **D** and, correspondingly, increases the yield of oxazines. The formation of the intermediate carbocation **D** is confirmed by the steric outcome of the transformation of ester **19**. In addition, the reactions of esters **17** afford no isomeric oxazines **20**.



20: R = H, Me

Consequently, as in the case of 1,3-dioxanes, the oxygen atom bound to the carbon atom bearing one aryl or two alkyl substituents is the reaction site in the borate molecule. Calculations demonstrated that the ions **C′** are less stable than **C**.[46]



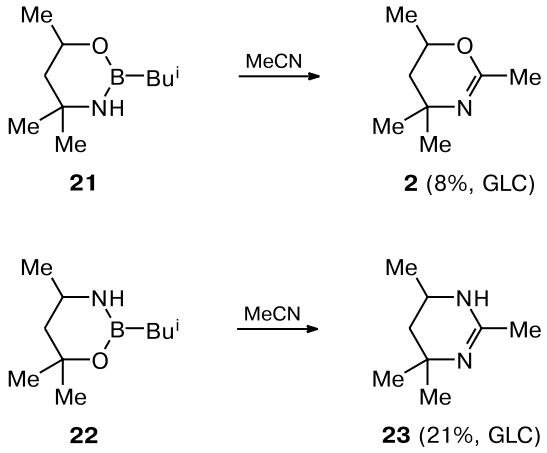
C **C′**

$E_{\mathbf{C}} - E_{\mathbf{C}'} = (1.1—7.8)$ kcal mol^{-1} (AM1)

The reactions of substituted 1,3,2-oxazaborinanes (**21** and **22**) with acetonitrile produce either dihydrooxazine (**2b**)[47] or substituted 1,4,5,6-tetrahydropyrimidine (**23**) depending on the structures of the starting compounds (Scheme 14).[48]

Five-membered analogs, *viz.*, 1,3,2-dioxaborolanes (**24**), also react with acetonitrile to give, as in the case of

Scheme 14

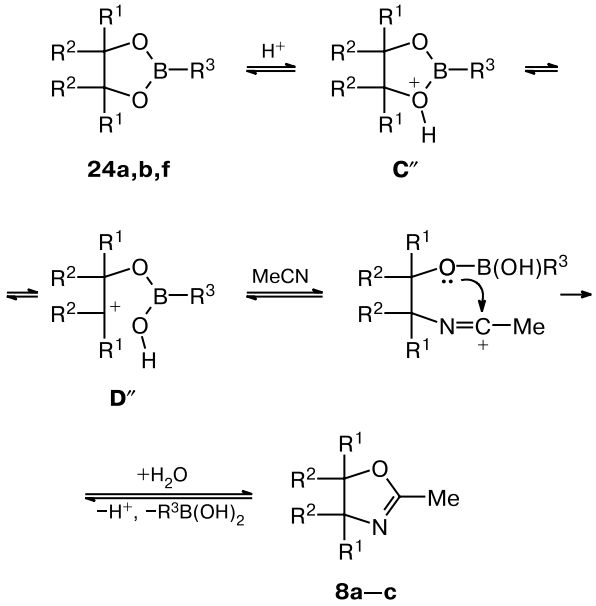


21 **2** (8%, GLC)

22 **23** (21%, GLC)

1,3-dioxolanes, 2-methyl-2-oxazolines **8** (Scheme 15). However, unlike 1,3,2-dioxaborinanes, which react with acetonitrile at room temperature or at 0 °C, the latter reactions proceed under more drastic conditions (Table 5).[32] An attempt to prepare oxazoline **8a** by the reaction of C-unsubstituted 1,3,2-dioxaborolane **24a** failed, and the starting ester was recovered. The maximum yields of oxazolines **8** (47—55%) were achieved by refluxing 4,4,5,5-tetramethyl-2-alkoxy derivatives in acetonitrile in the presence of concentrated H_2SO_4. As in the case of six-membered esters, the observed regularities are associated with the stability of the probable intermediate **D″** derived from the corresponding oxonium ion **C″** (see Scheme 15) (calculated at the AM1 level, see Table 5).[32]

Scheme 15



24a,b,f **C″**

D″

8a—c

R^1 = R^2 = H (**8a**), Me (**8b**); R^1 = H, R^2 = Ph (**8c**)

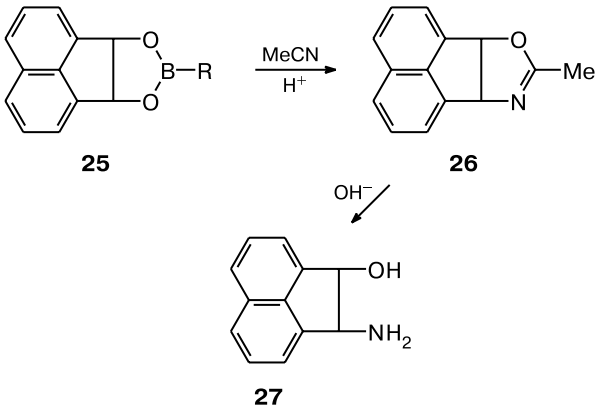
Table 5. Yields of 2-oxazolines depending on the number and nature of substituents in the starting 1,3,2-dioxaborolanes and reaction conditions

24	R^1	R^2	R^3	Reaction conditions		Yield of **8** (%)
				Solvent	*T*/°C	
a	H	H	OBu	MeCN	80	0 (**8a**)
b	Me	Me	OBu	MeCN	80	50 (**8b**)
c	Me	Me	OBu^i	MeCN	80	55 (**8b**)
				Hexane	10	20 (**8b**)
				$CHCl_3$	60	13 (**8b**)
d	Me	Me	OPr^i	MeCN	80	53 (**8b**)
e	Me	Me	OEt	MeCN	80	47 (**8b**)
f	Me	Me	Bu^i	MeCN	80	25 (**8b**)
g	H	Ph	Pr^i	MeCN	80	15 (**8c**)

Note: Relative stabilities of the intermediate, $\Delta E_{C''D''}$/kcal mol^{-1}: 18.3 (**24a**), –2.1 (**24b**), and 2.1 (**24f**).

The reaction of 2-alkylacenaphtho[1,2-*d*]-1,3,2-dioxaborolanes (**25**) with acetonitrile affords rather unstable 2-methylacenaphtho[1,2-*d*]-2-oxazoline (**26**) (Scheme 16), which is partially decomposed to form its alkaline hydrolysis product, 1-amino-2-hydroxyacenaphthene (**27**), during isolation.[49]

Scheme 16

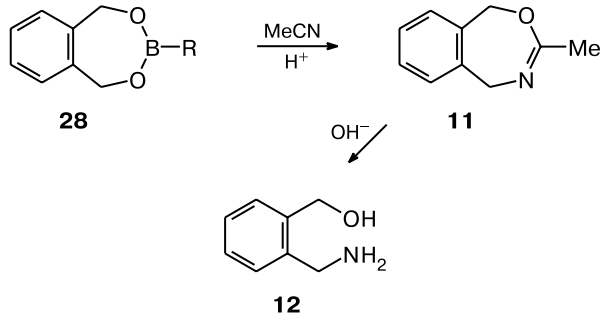


The presence of oxazoline **26** in a mixture with amino alcohol **27** was confirmed by mass spectrometry and IR spectroscopy. Subsequent heating of this mixture with a KOH solution gives rise to amino alcohol **27**.

Like other seven-membered heteroanalogs, 2-substituted 5,6-benzo-1,3,2-dioxaborepanes (**28**) react with acetonitrile to form 2-methyl-3,4-dihydro-1,3-benzooxazepine (**11**) (Scheme 17), which is readily saponified to give the corresponding amino alcohol, *viz*., 2-aminomethyl-1-hydroxymethylbenzene (**12**).[50]

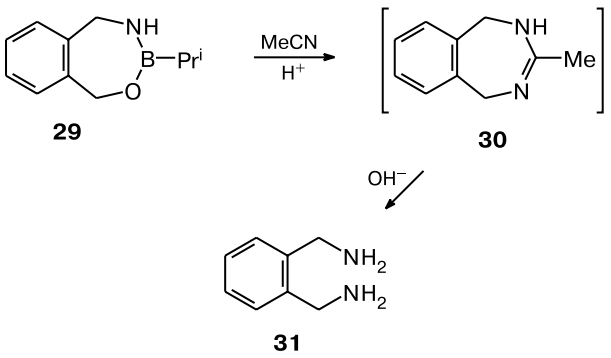
5,6-Benzo-1,3,2-oxazaborepane (**29**) also reacts with acetonitrile (Scheme 18). However, the expected

Scheme 17



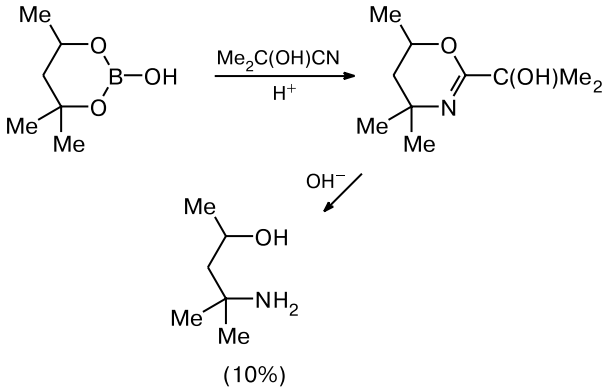
2-methyl-3,4-dihydro-1,3-benzodiazepine (**30**) was not isolated due, apparently, to its hydrolytic instability in an alkaline medium. The corresponding diamine, *viz*., 1,2-bis(aminomethyl)benzene (**31**), was the only identified reaction product.[51]

Scheme 18



The reaction of cyclic borates wit acetone cyanohydrin proceeds analogously (Scheme 19).[52]

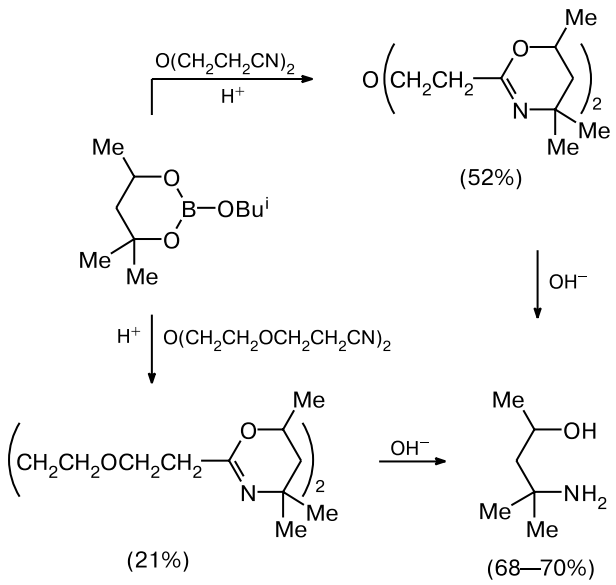
Scheme 19



The reactions of 2-isobutoxy-4,4,6-trimethyl-1,3,2-dioxaborinane with dinitriles, bis(2-cyano)diethyl ether,

and bis[2-(2-cyanoethoxy)]diethyl ether, also give rise to the corresponding substituted dihydrooxazines, *viz.*, bis[2-(5,6-dihydro-4,4,6-trimethyl-1,3-oxazin-2-yl)]diethyl ether and bis{2-[2-(5,6-dihydro-4,4,6-trimethyl-1,3-oxazin-2-yl)ethoxy]}diethyl ether (Scheme 20).[53—56]

Scheme 20



The IR spectra of the resulting oxazines have an intense band ν(C=N) at 1650—1640 cm^{-1}. Their compositions and structures were also confirmed by ^1H NMR spectroscopy and mass spectrometry.[53]

Hydrolysis of these compounds with an aqueous alkali affords 4-amino-4-methylpentan-2-ol.[53]

* * *

To summarize, the data surveyed in the present review contribute to a knowledge of the chemical transformations of 1,3-dioxacycloalkanes and their 2-heteroatomic analogs and extends the synthetic ways to various functional derivatives based on these compounds.

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