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Synthesis of hybrid structures comprising diaziridine and cyclopropane rings in one molecule

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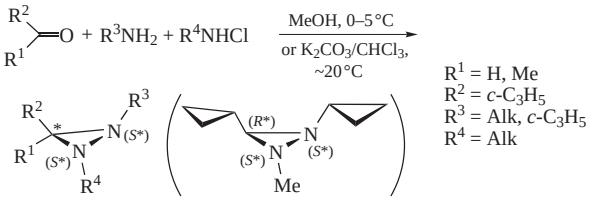
A facile, diastereoselective method for the synthesis of diaziridines with *C*- and *N*-cyclopropyl substituents has been developed, *N*-cyclopropyldiaziridines being for the first time synthesized. The method is based on one-pot three-component condensation of cyclopropyl-containing carbonyl compounds, primary aliphatic amines including cyclopropylamine, and *N*-chloroalkylamines in organic solvents in the presence of bases under mild conditions.



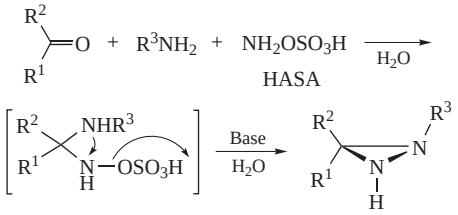
One of the promising trends of modern organic and medicinal chemistry is construction of medicinal tools by combining two or more pharmacologically active moieties in one molecule.[1] Among these moieties, the cyclopropane fragment can be distinguished, since cyclopropane derivatives has been recognized as effective medicinal agents.[2] Some cyclopropane-based drugs are already employed in medical practice, *e.g.*, tranylcypromine (MAO inhibitor), ciprofloxacin (antibacterial drug).

Another representative of three-membered rings, 1,2-diazacyclopropane, or diaziridine, was found to have a pronounced action on the central nervous system (CNS),[3] in particular, 1-[2-(3,3-dimethyldiaziridin-1-yl)ethyl]-3,3-dimethyldiaziridine has shown a high antidepressant activity.[4] Recently, we have developed a facile, diastereoselective synthesis of previously unknown 1,3-di- and 1,3,3-trisubstituted diaziridines coupled with neurotransmitter amino acids (glycine, β-alanine, and γ-aminobutyric acids).[5] We reasoned that association of diaziridine and cyclopropane rings in one molecule can give rise to compounds with new specific biologically active properties.

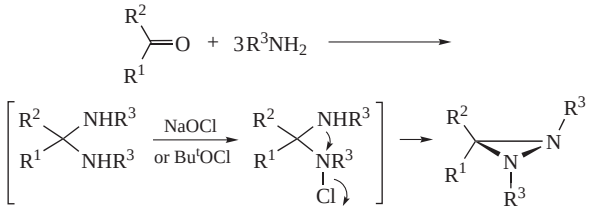
Here we present diastereoselective method for the preparation of new hybrid structures comprising two three-membered diaziridine and cyclopropane rings in one molecule, with the rings being connected by a C–C or by a C–N bond, depending on the cyclopropane reactant, that is a carbonyl compound (cyclopropanecarbaldehyde, cyclopropyl methyl ketone) or cyclopropylamine.

Prior to our investigations, only scarce examples containing a cyclopropane ring connected to carbon atom of diaziridine ring, in particular, 6-cyclopropyl-1,5-diazabicyclo[3.1.0]hexane[6(*a*)] were known. 3-Cyclopropyl-3-methyldiaziridine unsubstituted at the nitrogen atoms was synthesized in low yield at a temperature of –70 °C.[6(*b*)] In addition, two representatives of diaziridines with C-framework substituents including a cyclopropane fragment have been described.[6(*c*),(*d*)] *N*-Cyclopropyldiaziridines were unknown till now.

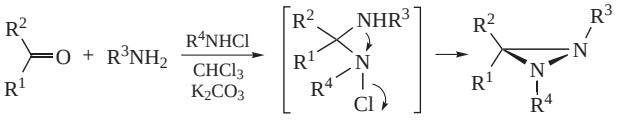
In our group a wide range of methods for the synthesis and transformation of diaziridines with different types of substituents has been developed.[7] The simplest and most general method for the construction of monocyclic diaziridines is the three-component condensation of a carbonyl compound, a primary aliphatic amine, and an aminating reagent (Scheme 1). These reactions are usually performed in three variants (Schemes 1–3). When hydroxylamine-*O*-sulfonic acid (HASA) is used as the aminating reagent, the reaction is conducted in water or in a water–alcohol (methanol or ethanol) mixture at controlled pH (8.5–10) at 0–5 °C. The pH value is maintained by adding aqueous NaOH. In these cases, diaziridines with one unsubstituted nitrogen atom are formed (Scheme 1). To prepare diaziridines with identical substituents at both nitrogen atoms, treatment of a mixture of a carbonyl compound and excess of primary aliphatic amine with NaOCl or Bu^{t}OCl is used (Scheme 2). The reaction with NaOCl is carried out in water or in water–alcohol mixture. The reaction with Bu^{t}OCl is usually performed in either MeOH or $CHCl_3$. In the latter case, K_2CO_3 is applied to neutralize HCl. To prepare diaziridine with different substituents on the nitrogen atoms, the carbonyl compound and the primary aliphatic amine in 1:1 molar ratio are added to one equivalent of a pre-synthesized *N*-chloro-



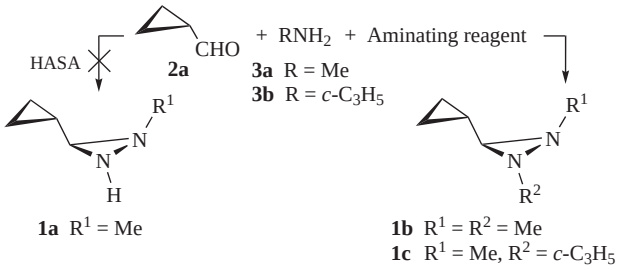
Scheme 1



Scheme 2



Scheme 3



Scheme 4

Table 1 Optimization of reaction conditions for the synthesis of diaziridines **1** with cyclopropyl substituents from cyclopropanecarboxaldehyde **2a** and methyl- and cyclopropylamines **3a**,**b**.

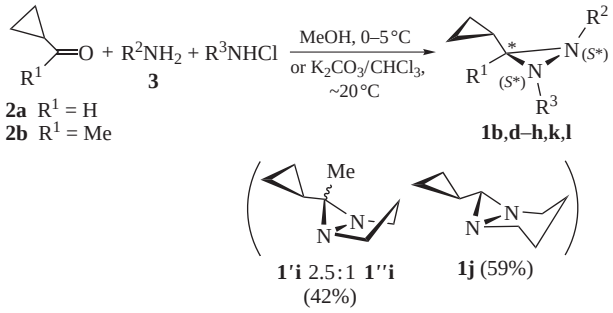
Entry	Amine **3** (equiv.)	Aminating reagent (equiv.)	Solvent	*T*/°C	Additive (equiv.)	Product, yield (%)
1	**3a** (1)	HASA (1)	H_2O	0–5	NaOH (2)	Decomp.
2	**3a**,**b** (1)	HASA (1)	MeOH–H_2O	0–5	NaOH (2)	Decomp.
3	**3a** (2)	NaOCl (1)	H_2O	0–5		Decomp.
4	**3a**,**b** (2)	NaOCl (1)	MeOH–H_2O	0–5		Decomp.
5	**3a**,**b** (3)	Bu^{t}OCl (1)	MeOH	0–5		Decomp.
6	**3a** (2)	MeNHCl[a] (1)	MeOH	0-5		**1b** (25)
7	**3a** (3)	MeNHCl[a] (1)	MeOH	0-5		**1b** (34)
8	**3a** (2)	MeNHCl (1)	$CHCl_3$	0-5		**1b** (13)
9	**3a** (2)	MeNHCl (1)	$CHCl_3$	0–5→20		**1b** (22)
10	**3a** (1)	MeNHCl (1)	$CHCl_3$	0–5→20	K_2CO_3 (1.5)	**1b** (23)
11	**3a** (1)	MeNHCl (1)	$CHCl_3$	0–5→20	K_2CO_3 (2.0)	**1b** (33)
12	**3b** (1)	MeNHCl (1)	$CHCl_3$	0–5→20	K_2CO_3 (2.0)	**1c** (13)

[a] Generated *in situ* from excess $MeNH_2$ and Bu^{t}OCl.

alkylamine with a different substituent in $CHCl_3$ in the presence of K_2CO_3 as a base (Scheme 3).

To access the desired compounds **1a**–**c**, cyclopropanecarboxaldehyde **2a** and two primary aliphatic amines, methylamine **3a** and cyclopropylamine **3b**, were selected as model substrates, and all reaction conditions shown in Schemes 1–3 were tested (Scheme 4, Table 1). In contrast to syntheses of 1,3-di- or 1,2,3-trialkyldiaziridines, all attempts to obtain diaziridines **1a**–**c** by condensation of cyclopropanecarboxaldehyde **2a**, amines **3a**,**b**, and aminating agents (HASA or action of NaOCl on two moles of amines **3a**,**b**) in water or water–alcohol medium failed. Decomposition of aldehyde **2a** and amine **3b** with formation of polymeric products insoluble in polar solvents occurred (Table 1, entries 1–5). 3-Cyclopropyldiaziridine **1a** unsubstituted on one nitrogen atom was not obtained. 1,2-Dimethyl-3-cyclopropyldiaziridine **1b** was formed in moderate yields, when aldehyde **2a** and excess of methylamine **3a** were treated with an equimolar amount of Bu^{t}OCl at low temperature in MeOH (entries 6, 7). The replacement of MeOH by $CHCl_3$ and use of pre-synthesized MeNHCl at a lower temperature also resulted in diaziridine **1b**, but in a lower yield (entry 8). The yield of compound **1b** was increased when this reaction was carried out in $CHCl_3$ at 20 °C (entry 9). The best results were achieved when equimolar amounts of compounds **2a**, **3a** and MeNHCl were treated in $CHCl_3$ with K_2CO_3 at 20 °C (entries 10, 11).[9] These conditions proved to be also suitable for the preparation 1,3-dicyclopropyl-2-methyldiaziridine **1c**, although in lower yield 13% (entry 12). Possibly, cyclopropane fragment in compounds **2a** and **3b** would decompose even under these mild non-aqueous conditions.

The optimal conditions found for the synthesis of compound **1b** were used for the preparation of other 3-cyclopropyldiaziridines **1d**–**l** from cyclopropanecarbonyl compounds (cyclopropanecarboxaldehyde **2a**, cyclopropyl methyl ketone **2b**), different

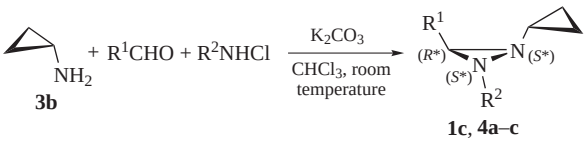


(1*S**,2*S**) **1b** R^1 = H, R^2 = R^3 = Me (34%)
(1*S**,2*S**) **1d** R^1 = H, R^2 = R^3 = Et (31%)
(1*S**,2*S**) **1e** R^1 = H, R^2 = R^3 = Pr (35%)
(1*S**,2*S**) **1f** R^1 = H, R^2 = R^3 = $Ph(CH_2)_2$ (41%)
(1*S*,*2*S**) **1g** R^1 = H, R^2 = R^3 = $HO(CH_2)_2$ (30%)
(1*S**,2*S**) **1h** R^1 = R^2 = R^3 = Me (17%)
(1*S**,2*S**,3*R**) **1′k** R^1 = H, R^2 = Me, R^3 = $Ph(CH_2)_2$ } (31%)
(1*S**,2*S**,3*S**) **1′′k** R^1 = H, R^2 = $Ph(CH_2)_2$, R^3 = Me } *dr* 1.7:1
(1*S**,2*S**,3*R**) **1′l** R^1 = H, R^2 = Me, R^3 = $HO(CH_2)_2$ } (39%)
(1*S**,2*S**,3*S**) **1′′l** R^1 = H, R^2 = $HO(CH_2)_2$, R^3 = Me } *dr* 2.5:1

Scheme 5

primary aliphatic alkylamines (methyl-, ethyl-, *n*-propyl-, 2-phenylethyl- and 2-hydroxyethylamines) and corresponding *N*-chloroalkylamines (Scheme 5). The conditions found for the synthesis of compound **1c** were applied to the preparation of *N*-cyclopropyldiaziridines **4a**–**c** (Scheme 6).†

3-Cyclopropyldiaziridines **1d**–**g** with identical substituents on both nitrogen atoms of the diaziridine ring were synthesized from aldehyde **2a** and primary aliphatic amines under the action of Bu^{t}OCl in MeOH in the presence of an excess of the corresponding amine at lower temperature. Under the same conditions, bicyclic diaziridines **1h**,**i** from carbonyl compounds **2a** or **2b** and 1,3-diaminopropane or 1,4-diaminobutane, respectively, were prepared. Diaziridines **1j**,**k**,**l** were obtained only under the action of pre-synthesized MeNHCl on the mixture of the corresponding carbonyl compound and primary aliphatic amine in $CHCl_3$ in the presence of K_2CO_3.



(1*S**,2*S**,3*R**) **1c** R^1 = *c*-C_3H_5, R^2 = Me (*dr* 2.5:1, 13%)
(1*S**,2*S**,3*R**) **4a** R^1 = R^2 = Me (*dr* 2.5:1, 21%)
(1*S**,2*S**,3*R**) **4b** R^1 = Et, R^2 = Me (*dr* 2.0:1, 23%)
(1*S**,2*S**,3*R**) **4c** R^1 = Me, R^2 = Et (*dr* 2.0:1, 24%)

Scheme 6 Only the major diastereomers are shown.

† *Synthesis of compounds* **1b**,**d**–**g**,**i**,**j**. A solution of Bu^{t}OCl (0.05 mol, 5.43 g) in MeOH (5 ml) was added dropwise with stirring at 0–5 °C to a solution of the specified amine (0.2 mol) in MeOH (100 ml). Then carbonyl compound (0.05 mol) was added. The reaction mixture was stirred at 0–5 °C for 12 h, the formed precipitate was filtered off, the organic phase was dried with K_2CO_3 for 30 min, the solvent was removed on a rotatory evaporator, and the residue was distilled *in vacuo*.

Synthesis of compounds **1c**,**h**,**k**,**l** and **4a**–**c**. A freshly prepared solution of *N*-chloroalkylamine (0.05 mol) in $CHCl_3$ (100 ml) was mixed with finely ground K_2CO_3 (0.075 mol, 10.35 g) and with the specified amine (0.05 mol). The carbonyl compound (0.05 mol) was added to the obtained suspension with stirring at 15–20 °C, and the reaction mixture was stirred at 20–22 °C for 24 h. The formed precipitate was filtered and washed with $CHCl_3$ (25 ml), the solvent was evaporated, and the residue was distilled *in vacuo*. The diastereomers of compound **1l** were separated by column chromatography on Silica gel $60F_{254}$ (eluent $CHCl_3$ + 5% EtOH).

The structures of synthesized diaziridines **1** were determined by ^{1}H and ^{13}C NMR, and IR spectroscopy and mass spectrometry), including the {^{1}H-^{1}H}g NOESY 2D NMR spectra (see Online Supplementary Materials).[8] It is known[9] that the diaziridine ring has two stereogenic nitrogen centers; however, these centers are prone to interconversion with barriers of 18–24 kcal mol^{-1}. The adjacent substituents on the nitrogen atoms of diaziridine ring exist only in an antiperiplanar conformation, which results in a coupled interconversion, and so both nitrogen atoms in each diaziridine ring have the same absolute configuration. Compounds **1b**–**h** contain the same substituents on the nitrogen atoms, the carbon atoms in these compounds are not stereogenic and, therefore, these compounds are 1*S**,2*S**-racemates. In the bicyclic structures **1i**,**j** the nitrogen atoms are firmly fixed and so they are not stereogenic; however, bicyclic compound **1i** is formed as a mixture of diastereomers **1′i** and **1′′i** (see Scheme 5 and Online Supplementary Materials). Compounds **1k**,**l** contain different substituents on nitrogen atoms and therefore the carbon atom becomes stereogenic. These compounds are formed as mixtures of diastereomers **1′k**, **1′′k** and **1′l**, **1′′l** with one of them predominating. Since both nitrogen atoms in them have the same configuration, these structures are mixtures of four epimers, namely two enantiomeric forms, (1*S*,2*S*,3*S*) and (1*R*,2*R*,3*R*), and two *meso*-forms, (1*S*,2*S*,3*R*) and (1*R*,2*R*,3*S*), which are revealed in TLC and in the ^{1}H NMR spectra as two diastereomers, (1*S**,2*S**,3*S**) and (1*S**,2*S**,3*R**) (see Scheme 5). The predominant diastereomer of compound **1′l** was isolated and characterized by a {^{1}H–^{1}H}g NOESY NMR spectrum (see Online Supplementary Materials). The minor diastereomer **1′′l** always contained a trace of the major one due to light-induced epimerization. The diastereomers of compound **1k** (**1′k**, **1′′k**) and of other analogous structures (**1c**, **4a**–**c**) synthesized in this study were not separated and were characterized as diastereomer mixtures; however, the spectral characteristics were derived for each diastereomer (see Online Supplementary Materials).

The NOESY 2D spectrum of the major diastereomer of compound **1l** (**1′l**) showed coupling of the CH_{ring} proton and both protons of the *N*-CH_2 group of the $N(CH_2)_2OH$ moiety. It is evident, that the more bulky cyclopropyl and 2-hydroxyethyl substituents occupy the *trans*-orientation and the *N*-Me and cyclopropyl substituents occupy the *cis*-orientation relative to the plane of the diaziridine ring. Hence, the major diastereomer of compound **1l** represents a racemic mixture of two *meso*-forms **1′l**, (1*S**,2*S**,3*R**), and the minor diastereomer is a mixture of two racemic forms, (1*S**,2*S**,3*S**) **1′′l** (see Online Supplementary Materials). The diastereomer ratio was established by comparison of the integral intensities of *N*-Me group signals in the ^{1}H NMR spectra (see Online Supplementary Materials). The major and minor diastereomers of compound **1k** were assigned in a similar manner.

1,3-Dicyclopropyl-2-methyldiaziridine **1c** proved to be accessible from equimolar amounts of cyclopropanecarboxaldehyde **2a**, cyclopropylamine **3b**, and MeNHCl in $CHCl_3$ in the presence of excess K_2CO_3 at 20 °C (see Table 1, entry 12). The negative result obtained for this reaction in MeOH evidently is connected with possible *trans*-chlorination between MeNHCl and cyclopropylamine resulting in decomposition of the *N*-chloro derivative of the latter. Especially, all attempts to synthesize *N*-chlorocyclopropylamine failed, and only polymeric products were formed. Therefore, to prepare other *N*-cyclopropyldiaziridines **4a**–**c**, the reaction was carried out in $CHCl_3$. For this aim cyclopropylamine **3b**, two aliphatic aldehydes (MeCHO, EtCHO) and *N*-chloroalkylamines (MeNHCl and EtNHCl) as aminating reagents were used (Scheme 6). Compounds **1c** and **4a**–**c** were also formed as mixtures of major and minor diastereomers in 2.0–2.5:1 ratio (see Online Supplementary Materials).

In summary, we developed a general, facile, and diastereoselective method for the preparation under very mild conditions (MeOH, 0–5 °C or $CHCl_3$ at 20 °C) of new hybrid structures comprising diaziridine and cyclopropane rings in one molecule with the rings being connected to each other by either C–C or C–N bonds depending on the source of cyclopropane ring, carbonyl compound or cyclopropylamine. This method is based on one-pot three-component condensation of a carbonyl compound, a primary amine, and an AlkNHCl in the presence of excess amine (for the reaction in MeOH) or K_2CO_3 (for the reaction in $CHCl_3$). The compounds with cyclopropyl substituents on the nitrogen atom of the diaziridine ring proved to be accessible only in aprotic solvent from equimolar amounts of the aldehyde, cyclopropylamine and a pre-synthesized AlkNHCl using K_2CO_3 as a base. Diaziridines with different substituents on nitrogen atoms were formed as mixtures of two diastereomers. The predominated diastereomer is the racemic mixtures of two *meso*-forms and the minor diastereomer is racemic mixtures of two enantiomers. On the whole, the developed method provides a powerful tool for the synthesis of an extensive series of a new kind of hybrid structures containing two pharmacophoric moieties, the cyclopropane and the diaziridine rings, in one molecule, *N*-cyclopropyldiaziridines being synthesized for the first time.

This paper is dedicated to the memory of our colleague, Professor Remir G. Kostyanovsky.

Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.1016/j.mencom.2018.09.015.

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