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Chiral bidiaziridines by a two-step domino aziridination of *meso*-α-diimines

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Dedicated to Professor Paolo Antonio Tardella on the occasion of his 78th birthday.

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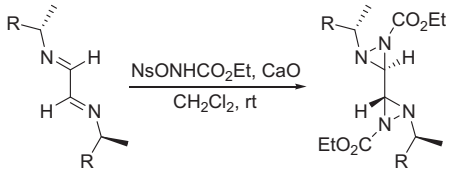
ABSTRACT

Chiral racemic α-diimines, tested in aziridination reactions with $NsONHCO_2Et$, for the first time led to the synthesis of (±)-bidiaziridines, stereoselectively derived from the corresponding *meso* (*E-s-trans-E*)-α-diimines. Moreover, a minor bidiaziridine isomer, probably a *meso* form that was lost under classical work-up conditions, can be obtained by adding water to the crude mixtures at the end of amination reactions. The results definitively prove that the imine aziridination by carbamates is a two-step domino process. The structures of the compounds were determined using 2D NMR on purified bidiaziridines.

1. Introduction

α-Diimines[1] derived from glyoxal and primary aliphatic amines treated with hydroxylamine-*O*-sulfonic acid in anhydrous methanol in the presence of an organic base led to the formation of a complex mixture of the corresponding diastereomeric bidiaziridines.[2]

Recently, we reported a direct aziridination of (*E-s-trans-E*)-α-diimines with ethyl nosyloxycarbamate ($NsONHCO_2Et$; Ns=4-$NO_2C_6H_4SO_2$) promoted by CaO,[3] that led only to one of the possible stereoisomers, the *meso* form, with total retention of the diimine configuration. Moreover, enantiomerically pure bidiaziridines were obtained starting from the corresponding (*R,R*) or (*S,S*) (*E-s-trans-E*)-α-diimines[4] (Scheme 1).



Scheme 1. Synthesis of enantiomerically pure bidiaziridines by $NsONHCO_2Et$.

As previously reported by us, the presence of a minor isomeric amination product was observed in very few cases, as suggested by ^1H NMR performed before the crude work-up.

This minor product was lost in all cases during the work-up steps (hexane addition followed by filtration through plugs filled with silica gel) and in all cases only one amination product was obtained as pure isomer.

Interested in better understanding the stereochemical outcome of aziridination reactions, the aminations of α-diimines that led to the formation of the aforementioned minor product were newly considered to examine the observed ratios between the two isomers. In order to achieve this, some reaction parameters were changed with particular emphasis on the possibility to control the amination products distribution and to avoid the loss of minor compounds during the reaction work-up. Moreover, as an interesting extension of our studies on the bidiaziridine isomerism, new chiral racemic α-diimines, bearing a steric hindrance in β-position, were tested with the aim of evaluating the possible effects of this hindrance on the stereochemical outcome.

2. Results and discussion

The α-diimines derived from glyoxal and carrying the cyclohexyl[5] (**1a**) or the pentyl[6] (**1b**) residues[7] were aziridinated with $NsONHCO_2Et$, in the presence of CaO in CH_2Cl_2,[8] under different

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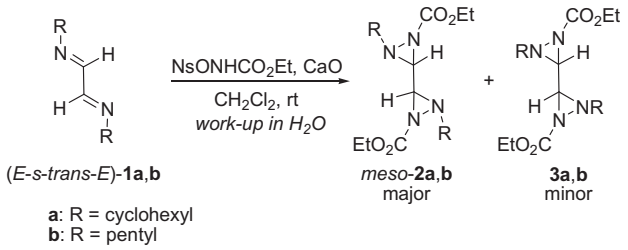
reaction conditions. However, the variation of molar ratios between the reactants and/or time of reaction and also the use of a different inorganic base (NaH) did not lead to any variation of the reaction course. On the contrary, working at low temperature (0 °C) no reaction was observed while adding metal ions (LiCl 5–10%) greatly slowed the reaction without any alteration of the product distribution even after 24 h of stirring. The only significant success was achieved by modifying the work-up step. At the end of the reaction, the crude mixtures were washed twice with H_2O to remove the calcium nosylate salts and the solvent was evaporated under vacuum (Scheme 2). The subsequent NMR analysis confirmed the effectiveness of the new work-up, showing again the presence of the expected two amination products (ca. 25:1). An HPLC purification (eluant: hexane/ethyl acetate=9:1) successfully allowed us to obtain the known bidiaziridines (*meso*)-**2a**,**b** as major products and bidiaziridine structures **3a**,**b** as minor products are thought to be other *meso* isomers or *d*,*l* mixtures (Scheme 2).



Scheme 2. Synthesis of bidiaziridines with $NsONHCO_2Et$.

To gain further insight,[9,10] 2D NMR ROESY experiments[11] were performed directly on the pure amination products and comparison were made between them (Fig. 1).[12]

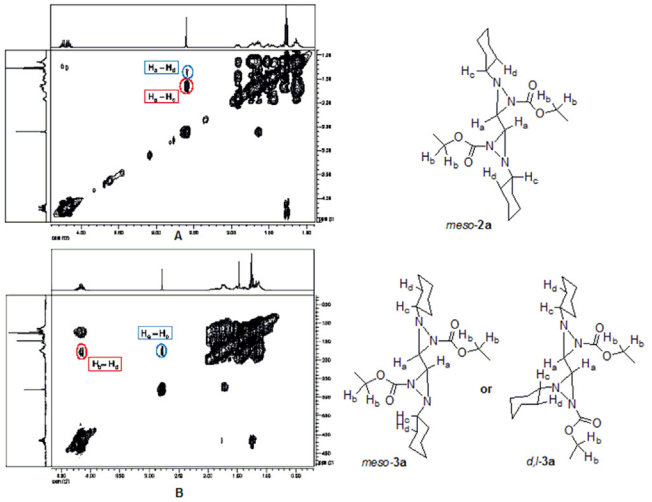


Fig. 1. ROESY spectrum of pure *meso*-**2a** (A). Spatial correlations between H_a and H_c and H_d are evident. On the basis of molecular models, the existence of the latter cross peak is possible only if the diaziridine protons and those of *N*-alkyl substituents face the same plane. ROESY spectrum of pure **3a** (**B**). Spatial correlations between H_a and H_c and between H_b and H_d are evident. On the basis of molecular models, the existence of the latter cross peak is possible only if the carbamic moiety protons and those of *N*-alkyl substituents face the same plane. Moreover, a lack of dipolar couplings between H_a and H_d indicates that the diaziridine protons and those of nitrogen substituents are directed toward opposite planes.

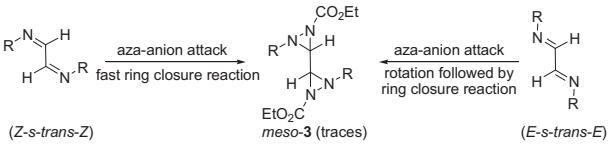
In the ROESY spectrum of pure *meso*-**2a** the most useful dipolar couplings are the ones observed among the aziridine protons H_a, the methylene protons of the carbamic moiety H_b and the protons belonging to the *N*-alkyl substituent (H_c, H_d). The carbamic moieties face in the opposite planes of both the diaziridine protons and the *N*-alkyl substituent protons, since no correlation was detected between the carbamic protons and the other protons of the molecule, for example, H_a, H_c, and H_d.[4,10]

Compound **3** showed a much different ROESY pattern compared to the major form **2**. In fact, as reported in Fig. 1 for **3a**, diaziridine protons show only a correlation with H_c protons (interproton distance between 3.5 and 4.5 Å) while a correlation between H_b and H_d protons (interproton distance between 3.0 and 3.8 Å) is clearly present. These interproton distances indicate that both nitrogen substituents are on the opposite plane of H_a, according to only one of the two reported structures, *meso*- or *d*,*l*-**3a** (Fig. 1).

Considering that diimine aziridination reactions via carbamate takes place only by the attack of aza-anion ($NsON^-CO_2Et$) across the opposite faces of the conjugated system,[4] the most probable structure of **3a** is a *meso* form, since the *d*,*l* form could only be derived from the attack of aza-anion on the same faces.

While diaziridines in cis-configuration were already established by NMR analysis,[13] to the best of our knowledge an analogous configuration for heterocyclic rings in bidiaziridines was never obtained.[14]

The formation of the minor isomers *meso*-**3** could be explained by the presence in the starting materials of traces (≤5%) of a different α-diimine isomer,[15] namely the (*Z*-*s*-*trans*-*Z*)-α-diimine,[16] or through the complete rotation around the two single C–N bonds derived by the *anti*-attack of aza-anion on the (*E*-*s*-*trans*-*E*) isomer, followed by ring closure reaction with loss of the starting diimine configuration (Scheme 3).



Scheme 3. Possible synthetic pathways for minor *meso*-**3**.

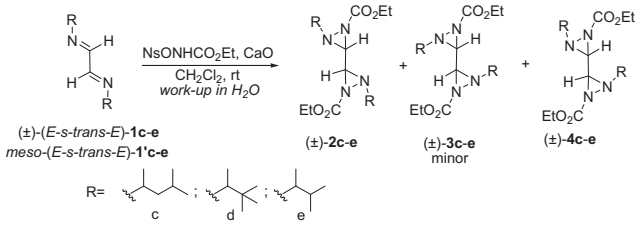
The latter hypothesis could be the first direct experimental evidence that even the diaziridine synthesis by carbamate, as the aminating agent of imines is a two-step domino process,[17] which involves an aza-anionic addition to C=N bond, similar to that observed on EWG-substituted olefins.[18]

Continuing our studies, α-diimines carrying two racemic chiral centers were considered as more complex substrates.

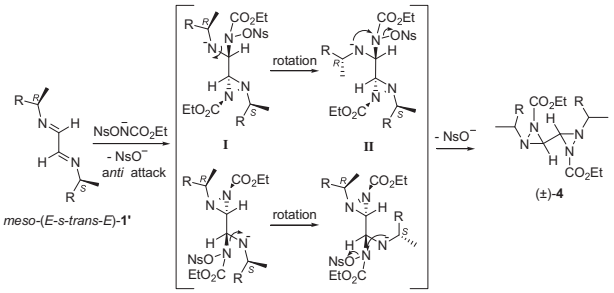
Starting from chiral racemic amines, the condensation reaction with glyoxal obviously leads to the synthesis of diastereomeric α-diimines **1** and **1′c**–**e**, as clearly shown by NMR spectra.[19] Unfortunately, all attempts to separate the two isomers failed, achieving only more-or-less enriched mixtures (ca. 60:40) of one of the two diastereomers, and the aziridination reactions were directly performed on the diastereomeric mixture (ca. 1:1). After the work-up in water, routine NMR analysis showed the presence of three different amination products, the *meso*-**2** and the *meso*-**3** forms[20] and a new amination compound never seen before in this kind of aziridination reactions, characterized by the presence of two doublets due to magnetically non-equivalent diaziridine protons (Scheme 4).

To unequivocally identify all the observed compounds, an HPLC methodology was optimized to separate the isomers and then ROESY NMR experiments were acquired on the purified compounds (Fig. 2).

So, **2** and **3c**–**e** were identified as the (±)-structures (A and B, Fig. 2) and the structures **4** were confirmed by comparison between the ROESY spectra (C, Fig. 2). In fact, the spatial correlations of the two diaziridine protons in **4** are different in the two rings, since the



Scheme 4. Aziridination of racemic mixtures of α-diimines.



Scheme 5. Possible pathway for the synthesis of (±)-**4** starting from *meso* **1′**.

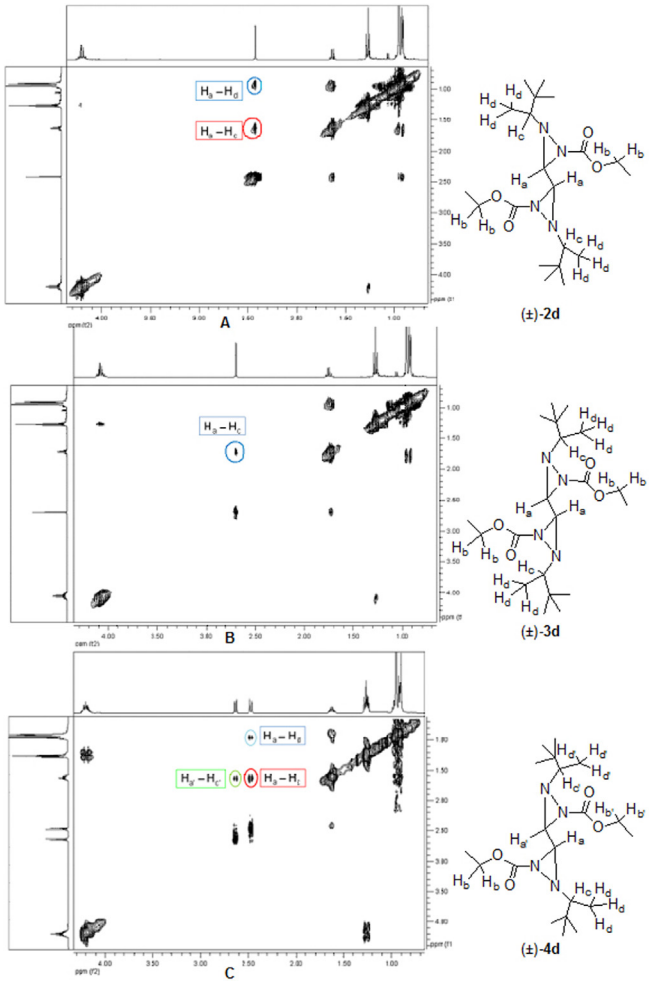


Fig. 2. ROESY spectrum of pure (±)-**2d** (A) and (±)-**3d** (B). Contrary to what observed for **3a**, no dipolar couplings were observed between H_b and H_d in B due to the greater flexibility of this molecule compared to the cyclic **3a**. ROESY spectrum of pure (±)-**4d** (**C**). Spatial correlations between H_a and H_c, H_a and H_d, and $H_{a'}$ and $H_{c'}$ are evidenced. Contrary to what observed for the other structures, the diaziridinic protons H_a and $H_{a'}$ clearly show different spatial correlation patterns, the first interacting with H_c and H_d while the latter interact only with $H_{c'}$.

H_a proton has dipolar couplings similar to the diaziridine protons of *meso*-**2a**,**b** and (±)-**2c**-**e**, while the $H_{a'}$ proton has the same correlation pattern of *meso*-**3a**,**b** and (±)-**3c**—**e** structures.

To explain the formation of *d*,*l*-**4**, it is possible to assume that a first aziridine ring is formed very quickly after aza-anion ($NsON^-CO_2Et$) attack on the diiminic conjugated system, so retaining the *E* configuration of C=N double bond, while the other ring is formed only after rotation around the formed single C—N bond of the intermediate **I**, giving an aziridine ring with complete inversion of the starting α-diimine configuration (Scheme 5).

Probably, the ring closure reaction to form the second diaziridine ring is slowed down in the intermediate **I** by steric hindrance, which is less important in the intermediate **II**. Moreover, considering that the optically pure α-diimines (*S*,*S*)- or (*R*,*R*)-**1d**,**e** yielded only the corresponding enantiomerically pure bidiaziridines,[4] (±)-**4** must be formed only from α-diimine *meso*-**1′c**—**e** by a two-step domino process that lead to *d*,*l* structures analogous to those obtained by an aziridination reaction of conjugated nitro alkenes using $NsONHCO_2Et$ as aminating agent.[21]

3. Conclusions

In conclusion, the aziridination reaction of α-diimines promoted by CaO with $NsONHCO_2Et$ as an aminating agent is finally demonstrated to be a two-step domino process. Also, the reported aziridination methodology is confirmed to be more stereoselective than the other methodology reported in the literature. In fact, if the reaction is performed on the *meso*-α-diimine and (*E*,*E*)-(±) mixtures, the first isomer gave stereoselectively the synthesis of only one of several possible (±)-bidiaziridine isomers, while the second one only led to one of the possible *d*,*l* forms, in which the diaziridine rings retain the (*E*,*E*)-diimine configuration, a different *meso* form remaining always a highly minor compound also by changing some parameters of the reaction.

The structural identification of all bidiaziridine isomers achieved only through the use of 2D NMR techniques on purified amination compounds confirms the versatility of the multidimensional NMR spectroscopy.[22]

4. Experimental

4.1. General

IR spectra were recorded on a Perkin—Elmer 1600 FT/IR spectrophotometer in $CHCl_3$ as the solvent. ^1H NMR and ^{13}C NMR spectra were recorded by a Bruker Avance III 400 MHz NMR spectrometer. $CDCl_3$ was used as the solvent and $CHCl_3$ as the internal standard. 2D NMR techniques were recorded by a Bruker Avance III 400 MHz NMR spectrometer and used to assist in structure elucidation.[22b] HRMS analyses were performed using a Micromass Q-TOF spectrometer equipped with an ESI source and a syringe pump. The experiments were conducted in the positive ion mode. HPLC analyses were performed with a Varian 9002 instrument equipped with a Varian 9050 RI-4 differential refractometer. Eluants were mixtures of HPLC grade hexane and ethyl acetate. $NsONHCO_2Et$[23] and α-diimines **1**[4] were synthesized as previously described.

*4.1.1. N,N′-(1E,2E)-Ethane-1,2-diylidenebis(4-methylpentan-2-amine) [(±)-(E-s-trans-E)-**1c** and meso-(E-s-trans-E)-**1′c**].* Orange oil (150 mg, 67%). IR ($CHCl_3$) 1626 cm^{-1}. ^1H NMR ($CDCl_3$): δ=0.84 (d, *J*=6.5 Hz, 6H), 0.85 (d, *J*=6.5 Hz, 6H), 0.88 (d, *J*=6.4 Hz, 6H, **1′c**),

0.89 (d, J=6.5 Hz, 6H, **1′c**), 1.17 (d, J=6.6 Hz, 6H, **1′c**), 1.19 (d, J=6.6 Hz, 6H), 1.28–1.36 (m, 4H), 1.42–1.59 (m, 8H), 3.34–3.44 (m, 4H), 7.89 (s, 2H), 7.91 (s, 2H, **1′c**). ^{13}C NMR ($CDCl_3$): δ=21.4 (2C), 21.5 (2C), 21.6 (2C), 22.3 (2C), 23.0 (2C), 23.1 (2C), 24.3 (2C), 24.4 (2C), 46.5 (4C), 63.6 (4C), 159.7 (4C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{14}H_{29}N_2$: 225.2331; found: 225.2336.

*4.1.2. N,N′-(1E,2E)-Ethane-1,2-diylidenebis(3,3-dimethylbutan-2-amine) [(±)-(E-s-trans-E)-**1d** and meso-(E-s-trans-E)-**1′d**].* Brown solid (204 mg, 91%). IR ($CHCl_3$) 1619 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.79 (s, 18H, **1′d**), 0.81 (s, 18H), 1.05–1.13 (m, 12H), 2.82–2.89 (m, 4H), 7.82 (s, 2H, **1′d**), 7.84 (s, 2H). ^{13}C NMR ($CDCl_3$): δ=17.1 (2C, **1′d**), 17.2 (2C), 26.3 (6C, **1′d**), 26.4 (6C), 33.8 (2C, **1′d**), 34.0 (4C), 75.4 (2C), 75.6 (2C, **1′d**), 160.3 (2C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{14}H_{29}N_2$: 225.2331; found: 225.2333.

*4.1.3. N,N′-(1E,2E)-Ethane-1,2-diylidenebis(3-methylbutan-2-amine) [(±)-(E-s-trans-E)-**1e** and meso-(E-s-trans-E)-**1′e**].* Brown solid (151 mg, 77%). IR ($CHCl_3$) 1619 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.80 (d, J=6.8 Hz, 6H), 0.83 (d, J=6.7 Hz, 6H, **1′e**), 0.86 (d, J=6.8 Hz, 6H), 0.87 (d, J=6.7 Hz, 6H, **1′e**), 1.14 (d, J=6.4 Hz, 6H), 1.18 (d, J=6.4 Hz, 6H, **1′e**), 1.66–1.77 (m, 4H), 2.85–2.97 (m, 2H), 2.89–2.98 (m, 2H, **1′e**), 7.82 (s, 2H, **1′e**), 7.89 (s, 2H). ^{13}C NMR ($CDCl_3$): δ=18.6 (2C, **1′e**), 18.7 (4C), 19.0 (2C, **1′e**), 19.1 (2C), 19.2 (2C), 33.5 (4C), 71.8 (2C, **1′e**), 71.9 (2C), 159.8 (4C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{12}H_{25}N_2$: 197.2018; found: 197.2016.

4.2. General for the synthesis of bidiaziridines

To a stirred solution of α-diimines **1a**,**b** and **1**, **1′c**–**e** (1 mmol) in CH_2Cl_2 (3 mL), CaO (3 mmol) and $NsONHCO_2Et$ (2 mmol) were added at room temperature. After the reactions were completed (^{1}H NMR), water was added. This operation is repeated twice, the organic layers were combined, dried over sodium sulfate. and the solvent was removed in vacuo. Bidiaziridines **2a**,**b**, **3a**,**b**, **2d**–**e**, **3d**–**e**, and **4d**–**e** were separated by HPLC (hexane/ethyl acetate=90:10, 1.3 mL/min, RI; **2a**:[4] 176 mg, 44%; **2b**:[4] 236 mg, 63%; **2d**:[4]123 mg, 32%; **2e**:[4] 134 mg, 36%).

*4.2.1. Diethyl 2,2′-dicyclohexyl-3,3′-bidiaziridine-1,1′-dicarboxylate (**3a**).* Brown oil (12 mg, 3%). IR ($CHCl_3$) 1736 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=1.12–1.14 (m, 4H), 1.16–1.18 (m, 2H), 1.25 (t, J=7.1 Hz, 6H), 1.37–1.40 (m, 2H), 1.42–1.44 (m, 2H), 1.50–1.54 (m, 2H), 1.70–1.72 (m, 4H), 1.73–1.77 (m, 2H), 1.77–1.81 (m, 2H), 1.86–1.90 (m, 2H), 2.78 (s, 2H), 4.10–4.25 (m, 4H). ^{13}C NMR ($CDCl_3$): δ=13.2 (2C), 22.9 (2C), 23.1 (2C), 24.6 (2C), 28.3 (2C), 30.2 (2C), 60.9 (2C), 62.4 (2C), 66.9 (2C), 160.8 (2C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{20}H_{35}N_4O_4$: 395.2658; found: 395.2656.

*4.2.2. Diethyl 2,2′-dipentyl-3,3′-bidiaziridine-1,1′-dicarboxylate (**3b**).* Yellow oil (15 mg, 4%). IR ($CHCl_3$) 1734 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.83 (t, J=6.7 Hz, 6H), 1.22–1.31 (m, 8H), 1.26 (t, J=7.1 Hz, 6H), 1.55–1.60 (m, 4H), 2.05–2.15 (m, 2H), 2.60–2.70 (m, 2H), 3.02 (s, 2H), 4.10–4.25 (m, 4H). ^{13}C NMR ($CDCl_3$): δ=12.9 (2C), 13.2 (2C), 21.4 (2C), 26.9 (2C), 27.8 (2C), 58.3 (2C), 59.6 (2C), 62.2 (2C), 160.9 (2C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{18}H_{35}N_4O_4$: 371.2658; found: 371.2660.

*4.2.3. Diethyl 2,2′-bis(4-methylpentan-2-yl)-3,3′-bidiaziridine-1,1′-dicarboxylate [(±)-**2c**, (±)-**3c**; (±)-**4c**].* Orange oil (259 mg, 65%). IR ($CHCl_3$) 1739 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.73 (d, J=6.6 Hz, 6H), 0.74 (d, J=6.6 Hz, 6H), 0.78 (d, J=6.5 Hz, 6H), 0.82 (d, J=6.5 Hz, 12H), 0.85 (d, J=6.4 Hz, 6H), 1.01 (d, J=6.7 Hz, 6H), 1.02 (d, J=6.7 Hz, 6H), 1.18 (d, J=6.5 Hz, 6H), 1.30–1.40 (m, 6H); 1.59–1.65 (m, 6H), 1.69–1.72 (m, 6H), 1.88–1.96 (m, 6H), 2.54 (s, 2H, **2c**), 2.55 (d, J=8.3 Hz, 1H, **4c**), 2.59 (d, J=8.3 Hz, 1H, **4c**), 2.61 (s, 2H, **3c**), 4.12–4.29 (m, 12H). ^{13}C NMR ($CDCl_3$): δ=12.5 (2C), 13.5 (4C), 15.8 (2C), 16.5 (2C), 19.0 (10C), 20.0 (4C), 30.8 (2C), 31.4 (4C), 41.9 (2C), 42.5 (2C), 42.8 (2C), 59.6 (2C), 61.7 (2C), 62.7 (2C), 63.1 (3C), 63.5 (3C), 69.4 (2C), 71.4 (2C), 71.8 (2C), 160.8 (2C), 161.4 (4C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{20}H_{39}N_4O_4$: 399.2971; found: 399.2964.

*4.2.4. Diethyl 2,2′-bis(1,2,2-trimethylpropyl)-3,3′-bidiaziridine-1,1′-dicarboxylate [(±)-**3d**].* Orange oil (27 mg, 7%). IR ($CHCl_3$) 1735 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.91 (d, J=6.7 Hz, 6H), 0.96 (s, 18H), 1.26 (t, J=7.1 Hz, 6H), 1.72 (q, J=6.7 Hz, 2H), 2.69 (s, 2H), 4.01–4.12 (m, 4H). ^{13}C NMR ($CDCl_3$): δ=11.1 (2C), 12.8 (2C), 26.1 (6C), 33.5 (2C), 62.8 (2C), 63.2 (2C), 72.1 (2C), 160.8 (2C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{20}H_{39}N_4O_4$: 399.2971; found: 399.2974.

*4.2.5. Diethyl 2,2′-bis(1,2-dimethylpropyl)-3,3′-bidiaziridine-1,1′-dicarboxylate [(±)-**3e**].* Orange oil (22 mg, 6%). IR ($CHCl_3$) 1735 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.84 (d, J=6.9 Hz, 6H), 0.88 (d, J=6.6 Hz, 6H), 1.24 (d, J=6.8 Hz, 6H), 1.34 (t, J=7.1 Hz, 6H), 1.59–1.72 (m, 2H), 1.92–1.98 (m, 2H), 2.67 (s, 2H), 4.22–4.32 (m, 4H). ^{13}C NMR ($CDCl_3$): δ=12.5 (2C), 15.8 (2C), 19.0 (4C), 31.4 (2C), 62.7 (2C), 63.1 (2C), 71.4 (2C), 160.8 (2C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{18}H_{35}N_4O_4$: 371.2658; found: 371.2651.

*4.2.6. Diethyl 2,2′-bis(1,2,2-trimethylpropyl)-3,3′-bidiaziridine-1,1′-dicarboxylate [(±)-**4d**].* Orange oil (139 mg, 35%). IR ($CHCl_3$) 1734 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.91 (d, J=6.6 Hz, 6H), 0.95 (s, 18H), 1.23–1.29 (m, 6H), 1.58–1.69 (m, 2H), 2.47 (d, J=8.5 Hz, 1H), 2.63 (d, J=8.5 Hz, 1H), 4.15–4.24 (m, 4H). ^{13}C NMR ($CDCl_3$): δ=13.3 (2C), 13.9 (2C), 25.7 (6C), 33.4 (2C), 59.3 (2C), 62.2, 63.6, 72.8 (2C), 160.6 (2C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{20}H_{39}N_4O_4$: 399.2971; found: 399.2972.

*4.2.7. Diethyl 2,2′-bis(1,2-dimethylpropyl)-3,3′-bidiaziridine-1,1′-dicarboxylate [(±)-**4e**].* Orange oil (126 mg, 34%). IR ($CHCl_3$) 1733 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ=0.80 (d, J=6.9 Hz, 6H), 0.92 (d, J=6.6 Hz, 6H), 1.10 (d, J=6.8 Hz, 6H), 1.34 (t, J=7.1 Hz, 6H), 1.70–1.82 (m, 2H), 1.92–2.00 (m, 2H), 2.60 (d, J=8.3 Hz, 1H), 2.65 (d, J=8.3 Hz, 1H), 4.22–4.38 (m, 4H). ^{13}C NMR ($CDCl_3$): δ=13.5 (2C), 16.4 (2C), 20.0 (4C), 31.4 (2C), 59.6 (2C), 62.5, 63.5, 71.8 (2C), 161.4 (2C). HRMS-ESI (m/z) $[M+H]^+$ calcd for $C_{18}H_{35}N_4O_4$: 371.2658; found: 371.2656.

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Supplementary data

Supplementary data associated with this article can be found in the online version, at http://dx.doi.org/10.1016/j.tet.2013.08.017.

References and notes

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