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Catalytic Opening of the Diaziridine Fragment in 6-Aryl-1,5-diazabicyclo[3.1.0]hexanes

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Abstract—Treatment of 6-aryl-1,5-diazabicyclo[3.1.0]hexanes with Lewis acids [$BF_3 \cdot Et_2O$ or $In(OTf)_3$] promotes opening of the diaziridine ring, followed by formation of 1,3-dipolar cycloaddition products with *N*-arylmaleimides. The conversion of the initial diaziridine depends on the nature of the 6-aryl group. Diazabicyclohexanes with donor substituents react quantitatively to give (in the absence of dipolarophiles) the corresponding azomethine imine dimers, 1,2,4,5-tetrazine derivatives. The conversion of diazabicyclohexanes having acceptor substituents is poor; simultaneously, the fraction of the hydrolysis products increases. The stereoselectivity in the 1,3-dipolar cycloaddition, i.e. the ratio of the *cis*- and *trans*-adducts, depends on the catalyst and solvent. Azomethine imine dimers react with *N*-arylmaleimides in the presence of indium(III) trifluoromethanesulfonate to give the same 1,3-dipolar cycloaddition products as those obtained from parent 1,5-diazabicyclohexanes.

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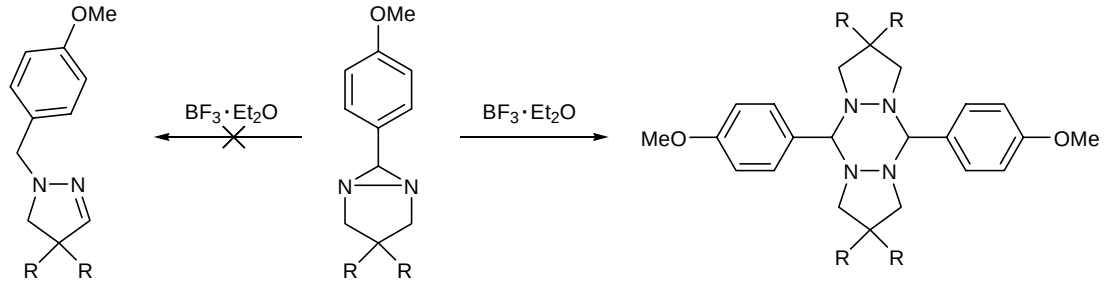
It is known that monocyclic diaziridines in the presence of mineral acids readily undergo hydrolysis with opening of the three-membered ring at the carbon–nitrogen or nitrogen–nitrogen bond [1–5]. Likewise, opening of the three-membered ring in aziridines at the carbon–nitrogen bond occurs by the action of, e.g., scandium trifluoromethanesulfonate; if the reaction is performed in the presence of activated alkenes, 1,3-dipolar cycloaddition products are formed [6, 7]. Catalytic opening of the diaziridine ring in 1,5-diazabicyclo[3.1.0]hexane in the presence of boron trifluoride–ether complex leads to the formation of a dimeric compound, 1,5,7,11-tetrahydrodipyrazolo-[1,2-*a*:1',2'-*d*][1,2,4,5]tetrazine [8].

We previously showed that thermolysis of 6-aryl-1,5-diazabicyclo[3.1.0]hexanes having a strained *cis*-N,N-disubstituted diaziridine fragment involves opening of the diaziridine ring at the carbon–nitrogen bond to give unstable azomethine imines. The latter are capable of undergoing either isomerization to the corresponding 1-arylmethyl-4,5-dihydropyrazoles or 1,3-dipolar cycloaddition to various dipolarophiles provided that they are present in the reaction mixture [9–12]. *N*-Arylmaleimides as dipolarophiles give rise mainly to adducts with *trans* arrangement of the aryl group and pyrrolidine ring [10, 11].

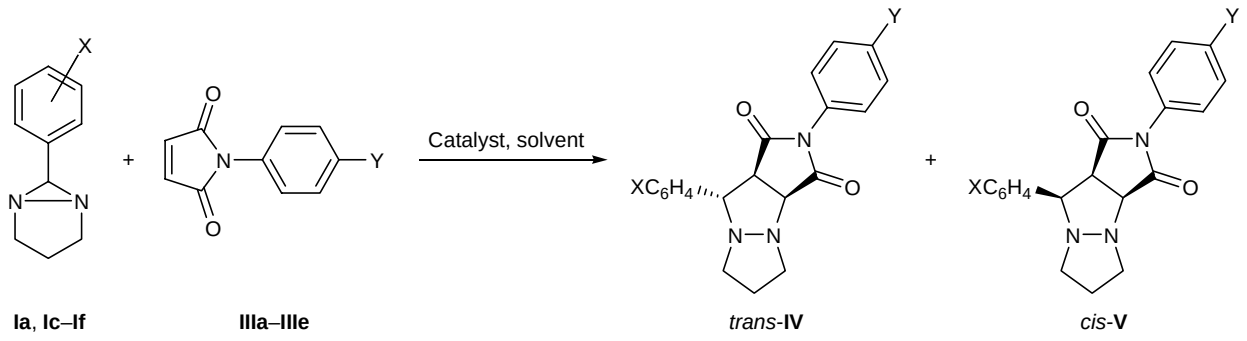
The present article reports on the behavior of 6-aryl-1,5-diazabicyclo[3.1.0]hexanes in reactions with

Scheme 1.



R = H (**a**), Me (**b**).

Scheme 2.



Ia, **Ic**–**If** **IIIa**–**IIIe** *trans*-**IV** *cis*-**V**

I, X = 4-MeO (**a**), 4-Me (**c**), H (**d**), 4-Cl (**e**), 3-O_2N (**f**); **III**, Y = MeO (**a**), Me (**b**), H (**c**), Br (**d**), O_2N (**e**).

Lewis acids [boron trifluoride–ether complex and indium(III) trifluoromethanesulfonate] in the presence and in the absence of *N*-arylmaleimides as reactive 1,3-dipolarophiles. It was presumed that catalytic opening of the diaziridine fragment, as in the thermal reaction, will generate unstable azomethine and that it will be stabilized via subsequent 1,3-dipolar cycloaddition.

However, unlike thermal reaction leading to 1-arylmethyl-4,5-dihydropyrazoles, treatment of 6-(4-methoxyphenyl)-1,5-diazabicyclo[3.1.0]hexane (**Ia**) and 6-(4-methoxyphenyl)-3,3-dimethyl-1,5-diazabicyclo[3.1.0]hexane (**Ib**) with $BF_3 \cdot Et_2O$ (~5 mol %) in acetonitrile or THF–diethyl ether in the absence of *N*-arylmaleimides resulted in the formation of 5,11-bis(4-methoxyphenyl)tetrahydrodipyrazolo[1,2-*a*:1',2'-*d*][1,2,4,5]tetrazine (**IIa**) and 5,11-bis(4-methoxyphenyl)-2,2,8,8-tetramethyltetrahydrodipyrazolo[1,2-*a*:1',2'-*d*][1,2,4,5]tetrazine (**IIb**), respectively (Scheme 1). Compounds **IIa** and **IIb** are dimerization products of the corresponding unstable intermediate azomethine imines.

The ^{1}H NMR spectra of the reaction mixtures obtained from 6-(4-methylphenyl)- and 6-(4-chlorophenyl)-1,5-diazabicyclo[3.1.0]hexanes **Ic** and **Ie** under analogous conditions also contained signals typical of protons in positions *5* and *11* of 1,2,4,5-tetrazine ring (δ 4.03 ppm for **IIc** and 4.16 ppm for **IIe**), as well as signals from protons in the trimethylene bridges (multiplets at δ 1.75–1.89, 2.35–2.48, and 2.60–2.70 ppm with equal intensities). However, the conversion of the initial diazabicyclohexanes did not exceed 5–8%; moreover, signals belonging to the hydrolysis products were also present in the spectra, especially when the reaction was carried out in acetonitrile. Likewise, in the ^{1}H NMR spectra of the reaction mixtures obtained from 6-phenyl-1,5-diazabicyclo[3.1.0]hexane (**Id**) and compound **Ie** in the presence of indium(III)

Table 1. Ratios of *trans*- and *cis* isomers **IV** and **V** in the thermal (Δ) and catalytic ($BF_3 \cdot Et_2O$) opening of the diaziridine ring in compounds **Ia** and **Ic**–**If** in the presence of *N*-arylmaleimides **IIIa**–**IIIe**[a]

N-Arylmaleimide	6-Aryl-1,5-diazabicyclo[3.1.0]hexane									
	Ia		**Ic**		**Id**[b]		**Ie**[b]		**If**[c]	
	Δ	$BF_3 \cdot Et_2O$	Δ	$BF_3 \cdot Et_2O$	Δ	$BF_3 \cdot Et_2O$	Δ	$BF_3 \cdot Et_2O$	Δ	$BF_3 \cdot Et_2O$
IIIa	1.8	0.5–0.7	2.1	0.71	2.0	0.74	2.0	0.67	1.7	0.56
IIIb	2.4	0.77	2.6	0.71	3.1	0.74	2.9	0.71	2.0	0.5
IIIc	1.9	0.5–0.6	2.2	0.56	2.0	0.5–0.7	2.0	0.5	1.5	0.59
IIId	1.7	0.4	1.8	0.43	2.0	0.5	1.9	0.48	1.5	0.48
IIIe	1.3	0.15–0.2[d]	1.5	0.4	1.7	0.29	1.5	0.29	1.3	0.2

[a] The thermolysis was carried out in toluene (~110°C), and the reaction in the presence of boron trifluoride–ether complex, in acetonitrile at room temperature.

[b] The conversion was less than 50–60%.

[c] The conversion was less than 30% (ratio of the addition/hydrolysis products ~0.7).

[d] For the reaction in toluene, the *trans*/*cis* ratio was ~0.7.

trfluoromethanesulfonate in THF, signals from protons in the 1,2,4,5-tetrazine ring and trimethylene bridges of the corresponding dimers were observed. Here, the conversion of the initial compounds did not exceed 50–60%, and the fraction of their hydrolysis products was ≥60%. Attempts to isolate azomethine imine dimers as individual substances were unsuccessful.

As well as in the thermal process, catalytic opening of the diaziridine ring in compounds **I** in the presence of *N*-arylmaleimides resulted in the formation of mixtures of diastereoisomeric adducts **IV** and **V** (Scheme 2), but the diastereoisomer ratio (i.e., stereoselectivity) depended on the solvent and catalyst. For example, compounds **Ia** and **Ic**–**If** reacted with maleimides **IIIa**–**IIIe** in acetonitrile in the presence of boron trifluoride–ether complex to give mainly the corresponding *cis*-adducts **V** (in contrast to the thermal reactions). In these cases, unlike the reactions performed in the absence of a 1,3-dipolarophile, the conversion of compounds **Ia** and **Ic** was complete (according to the ^{1}H NMR data), the conversion of **Id** and **Ie** did not exceed 50–60%, and the conversion of **If** was less than 30%; simultaneously, the fraction of the hydrolysis products increased (Table 1).

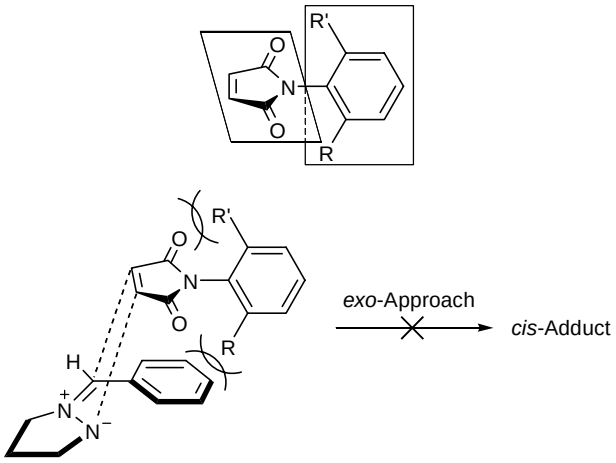
The stereoselectivity in the reaction of compound **Ia** with maleimides **IIIa**–**IIId** in the presence of indium(III) trifluoromethanesulfonate in tetrahydrofuran or toluene was approximately the same as in the thermal process (within experimental error, ±10–20%), while the reaction of **Ia** with *N*-(4-nitrophenyl)maleimide (**IIIe**) in toluene gave the corresponding *cis*-adduct as the major product. In the reactions of **If** with $In(OTf)_3$ in THF, the *cis*-adducts were mainly formed in all cases (Table 2). As in the reaction catalyzed by $BF_3 \cdot Et_2O$, the conversion of **If** did not exceed 30–40%, and a considerable amount of the hydrolysis product (3-nitrobenzaldehyde) was formed.

Table 2. Stereoselectivity (ratio of *trans*- and *cis*-adducts **IV** and **V**) in the cycloaddition of compounds **Ia** and **If** and **IIIa**–**IIIe** in THF and toluene in the presence of indium(III) trifluoromethanesulfonate

N-Arylmaleimide	6-Aryl-1,5-diazabicyclo[3.1.0]hexane		
	Ia		**If**
	THF	toluene	THF
IIIa	2.0	2.0	0.83
IIIb	2.8	3.0	0.67
IIIc	1.9	2.8	0.75
IIId	1.3	1.7	0.83
IIIe	1.0	0.83	0.4

We previously found that thermolysis of 6-aryl-1,5-diazabicyclo[3.1.0]hexanes in the presence of *N*-arylmaleimides having substituents in positions *2* and *6* of the aromatic ring, e.g., *N*-mesitylmaleimide (**IIIf**) and *N*-(2,6-dichlorophenyl)maleimide, gives exclusively the corresponding *trans*-adducts [11]. The observed 100% stereoselectivity is likely to be determined by spatial interactions between the aryl group in intermediate *Z*-azomethine imine and *ortho*-substituents in *N*-arylmaleimide which has a nonplanar structure. These interactions hamper *exo*-approach of the reactants, which could lead to the formation of *cis*-adduct (Scheme 3).

Scheme 3.



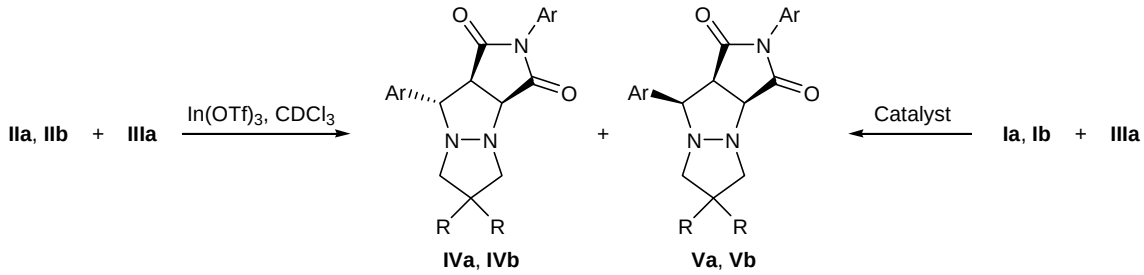
By the action of boron trifluoride–ether complex on diazabicyclohexane **Ia** in the presence of maleimide **IIIf** in acetonitrile or deuterochloroform, as well as in the thermal reaction, compound **VI** was obtained as the only product; i.e., no change in stereoselectivity was observed (Scheme 4).

Opening of the strained *cis*-N,N-disubstituted diaziridine fragment in 6-aryl-1,5-diazabicyclo[3.1.0]-hexanes by the action of Lewis acids may be regarded as expected process. On the other hand, opening of the 1,2,4,5-tetrazine ring with formation of adducts **IVa**/**Va** and **IVb**/**Vb** in the $In(OTf)_3$-catalyzed reaction of dimers **IIa** and **IIb** in the presence of maleimide **IIIa** turned out to be surprising (Scheme 5); the same adducts can be obtained directly from diazabicyclohexanes **Ia** and **Ib**, respectively. Only thermal decomposition of the 1,2,4,5-tetrazine ring, leading to the corresponding azomethine imines, was reported previously (see, e.g., [13]). The *trans*/*cis* ratio (**IV**/**V**) was

Scheme 4.

IIIf **VI**

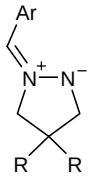
Scheme 5.



IVa, **IVb** **Va**, **Vb**

R = H (**a**), Me (**b**); Ar = 4-$MeOC_6H_4$.

2.0 in the reaction with dimer **IIa** and 1.7 for **IIb**. The ratio of adducts **IVa**/**Va** was similar to that observed in the thermal (in toluene) or catalytic [$In(OTf)_3$, THF] opening of the diaziridine fragment in diazabicyclohexane **Ia** in the presence of maleimide **IIIa** (Tables 1, 2). Presumably, all these reactions involve formation of a common intermediate, namely unstable azomethine imine **A**.



A

The different conversions of the initial diazabicyclohexanes in the catalytic processes performed in the absence and in the presence of a 1,3-dipolarophile (especially, in the reactions with compound **Ic**) may result from reversibility of the catalytic reaction. In the thermal process in the absence of a dipolarophile, intermediate azomethine imine is stabilized via irreversible isomerization to the corresponding 1-arylmethyl-4,5-dihydropyrazole [10, 14]. Electron-donor substituents in the aryl group partially stabilize intermediate azomethine imine and favor the equilibrium to be displaced toward its formation; therefore, the substrate conversion is complete. Solvent dependence of the ratio of the *cis*- and *trans*-adducts formed in the catalytic reaction in the presence of a dipolarophile may be rationalized in terms of solvation of intermediate azomethine imine and/or change of its configuration.

EXPERIMENTAL

The NMR spectra were recorded on a Bruker DPX-300 spectrometer at 300.130 and 75.468 MHz for ^1H and ^{13}C, respectively, using $CDCl_3$ as solvent. The chemical shifts were measured relative to the solvent signals ($CHCl_3$, δ 7.26 ppm; $CDCl_3$, δ_C 77.16 ppm) [15]. The solvents used were dried by standard procedures. Boron trifluoride–ether complex was distilled under argon prior to use. Indium(III) trifluoromethanesulfonate was commercial product (Acros Organics).

Initial 6-aryl-1,5-diazabicyclo[3.1.0]hexanes **Ia**–**If** were synthesized by condensation of the corresponding aromatic aldehyde with propane-1,3-diamine or 2,2-dimethylpropane-1,3-diamine, followed by oxidation of substituted hexahydropyrimidines with an aqueous solution of sodium hypochlorite according to the procedures described in [9–11].

Catalytic dimerization of 6-(4-methoxyphenyl)-1,5-diazabicyclo[3.1.0]hexanes. Compound **Ia** or **Ib**,

2–3 mmol, was dissolved in 5 ml of THF, ~3 drops (~18–20 mg, 0.1 mmol) of boron tifluoride–ether complex were added, and the mixture was stirred for 72 h at room temperature. The solvent was distilled off under reduced pressure on a rotary evaporator. In the reaction with compound **Ia**, the solid residue was shaken with warm acetone, the mixture was cooled, and the precipitate was filtered off and washed with acetone on a filter. In the reaction with **Ib**, the residue was recrystallized from acetone with addition of diethyl ether.

5,11-Bis(4-methoxyphenyl)hexahydrodipyrazolo-[1,2-*a*:1',2'-*d*][1,2,4,5]tetrazine (IIa). Yield 81%, mp >152°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 1.75–1.89 (4H), 2.42 pseudoquartet (4H, *J* = 8.8 Hz), 2.60–2.70 (4H), 3.83 s (6H), 3.99 s (2H), 6.89 d (4H, *J* = 8.0 Hz), 7.49 d (4H, *J* 8.0 Hz). ^{13}C NMR spectrum (DEPT), δ_C, ppm: 22.9 (CH_2), 49.0 ($2CH_2$), 55.4 (OCH_3), 88.9 (NCHN), 113.7 (2CH), 130.1 (2CH), 130.2 (C), 160.0 (C). Found, %: C 69.45, 69.47; H 7.37, 7.34; N 14.88, 14.95. $C_{22}H_{28}N_4O_2$. Calculated, %: C 69.45; H 7.42; N 14.72.

5,11-Bis(4-Methoxyphenyl)-2,2,8,8-tetramethyl-hexahydrodipyrazolo[1,2-*a*:1',2'-*d*][1,2,4,5]tetrazine (IIb). Yield 73%, mp 158°C (decomp.). ^{1}H NMR spectrum, δ, ppm: 1.02 s (12H), 2.29 d (4H, *J* = 13.8 Hz), 2.33 d (4H, *J* = 13.8 Hz), 3.83 s (6H), 3.93 s (2H), 6.88 d (4H, *J* = 8.0 Hz), 7.46 br.d (4H, *J* = 8.0 Hz). ^{13}C NMR spectrum (DEPT), δ_C, ppm: 28.9 ($2CH_3$), 37.2 (C), 55.3 (OCH_3), 64.4 ($2CH_2$), 89.3 (NCHN), 113.6 (2CH), 130.0 (2CH), 130.1 (C), 160.0 (C). Found, %: C 71.52, 71.42; H 8.27, 8.34; N 12.94, 12.93. $C_{26}H_{36}N_4O_2$. Calculated, %: C 71.53; H 8.31; N 12.83.

Determination of the product ratio in the catalytic opening of the diaziridine ring in 6-aryl-1,5-diazabicyclo[3.1.0]hexanes in the presence of *N*-arylmaleimides. *a*. A mixture of 0.2 mmol of 6-aryl-1,5-diazabicyclo[3.1.0]hexane and 0.2 mmol of *N*-arylmaleimide in 1–2 ml of a solvent was purged with argon, and 1–2 drops (7–12 mg, 25–50 mol %) of freshly distilled boron trifluoride–ether complex were added. The mixture was stirred for 72 h at room temperature and evaporated under reduced pressure at a temperature not exceeding 40°C, and the residue was dissolved in DMSO-d_6 or $CDCl_3$. The ratio of *trans*- and *cis*-adducts **IV** and **V** was determined from the intensities of the broadened signal from the ArC**H** proton of the *trans* isomer and two downfield signals from the corresponding proton of the *cis* isomer [10]. The physical constants and spectral parameters of adducts **IV** and **V** were reported in [10], and those of compound **VI**, in [11].

b. A mixture of 0.2 mmol of 6-aryl-1,5-diazabicyclo-[3.1.0]hexane and 0.2 mmol of *N*-arylmaleimide was purged with argon, 6–7 mg (5–6 mol %) of indium(III) trifluoromethanesulfonate was added, and 2 ml of appropriate solvent (preliminarily purged with argon) was then added. The mixture (homogeneous in THF and heterogeneous in toluene) was stirred for 72 h at room temperature and evaporated under reduced pressure at a temperature not exceeding 40°C, and the residue was dissolved in DMSO-d_6 or $CDCl_3$.

Thermolysis of 6-aryl-1,5-diazabicyclo[3.1.0]-hexanes Ia and Ic–If in toluene. A solution of 0.2 mmol of 6-aryl-1,5-diazabicyclo[3.1.0]hexane **Ia** or **Ic**–**If** and 0.2 mmol of the corresponding substituted *N*-arylmaleimide in 2 ml of toluene was stirred for 2.5 h at a bath temperature of 110°C. The solvent was evaporated, the residue was dissolved in $CDCl_3$, and the product ratio was determined from the ^{1}H NMR spectra.

Reaction of azomethine imine dimers IIa and IIb with *N*-(4-methoxyphenyl)maleimide in the presence of indium(III) trifluoromethanesulfonate. A mixture of 0.1 mmol of dimer **IIa** or **IIb** and 0.2 mmol of *N*-(4-methoxyphenyl)maleimide was purged with argon, and ~6 mg (0.01 mmol, 5 mol %) of $In(OTf)_3$ and 1.5 ml of $CDCl_3$ were added in succession. The resulting heterogeneous mixture [indium(III) trifluoromethanesulfonate did not dissolve completely] was stirred for 72 h at room temperature. The product ratio was determined from the ^{1}H NMR spectrum. In the reaction of **IIa** with maleimide **IIIa**, a mixture of adducts **IVa** and **Va** at a ratio of 2.0:1 was obtained. The spectral parameters of **IVa** and **Va** coincided with those given in [10]. From dimer **IIb** and maleimide **IIIa**, a mixture of adducts **IVb** and **Vb** was formed at a ratio of 1.7:1 (^{1}H NMR).

***rel*-(3a*R*,9*R*,9a*R*)-2,9-Bis(4-methoxyphenyl)-6,6-dimethylperhydropyrazolo[1,2-*a*]pyrrolo[3,4-*c*]-pyrazole-1,3-dione (IVb).** ^{1}H NMR spectrum, δ, ppm: 1.20 s (3H, CH_3), 1.23 s (3H, CH_3), 2.32 d (1H, *J* = 9.3 Hz), 2.53 d (1H, *J* = 9.3 Hz), 2.88 d (1H, *J* = 9.3 Hz), 3.18 d (1H, *J* = 9.3 Hz), 3.83 s (6H, OCH_3), 4.01–4.08 m (2H), 4.37 br.s (1H), 6.93 d (2H, *J* = 8.4 Hz), 6.98 d (2H, *J* = 8.4 Hz), 7.25 d (2H, *J* = 8.4 Hz), 7.48 d (2H, *J* = 8.4 Hz).

***rel*-(3a*R*,9*S*,9a*R*)-2,9-Bis(4-methoxyphenyl)-6,6-dimethylperhydropyrazolo[1,2-*a*]pyrrolo[3,4-*c*]-**

pyrazole-1,3-dione (Vb). An analytical sample was isolated from a solution of the reaction mixture in acetone–diethyl ether. mp 202–204°C. ^{1}H NMR spectrum, δ, ppm: 1.29 s (3H, CH_3), 1.31 s (3H, CH_3), 2.62 d (1H, *J* = 11.0 Hz), 3.00 d (1H, *J* = 9.3 Hz), 3.02 d (1H, *J* = 11.0 Hz), 3.17 d (1H, *J* = 9.3 Hz), 3.80 s (6H, OCH_3), 3.88 d.d (1H, *J* = 8.7, 7.3 Hz), 4.34 d (1H, *J* = 7.3 Hz), 4.59 d (1H, *J* = 8.7 Hz), 6.88 d (2H, *J* = 8.5 Hz), 6.92 d (2H, *J* = 8.5 Hz), 7.08 d (2H, *J* = 8.5 Hz), 7.34 d (2H, *J* = 8.5 Hz). Found, %: C 68.12; H 6.31; N 10.08. $C_{24}H_{27}N_3O_4$. Calculated, %: C 68.39; H 6.46; N 9.97.

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