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# Thermal Decomposition Products of Methyl 1,5-Diphenyl- and 5-Methyl-1-phenyl-2,3-diazabicyclo[3.1.0]hex-2-ene-6-*exo*-carboxylates

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**Abstract**—Methyl 1,5-diphenyl- and 5-methyl-1-phenyl-2,3-diazabicyclo[3.1.0]hex-2-ene-6-*exo*-carboxylates at 138°C undergo decomposition via elimination of nitrogen molecule with formation in each case of five products. Two products are methyl 1,3-diphenyl(or 1-methyl-3-phenyl)bicyclo[1.1.0]butane-2-*endo*- and -*exo*-carboxylates, and the three others are derivatives of buta-1,3-diene, methyl (*Z*)-2-benzylidene-3-phenyl(or 3-methyl)but-3-enoate and methyl (*E*)- and (*Z*)-3,4-diphenyl(or 4-methyl-3-phenyl)penta-2,4-dienoates. The formation of these products may be rationalized assuming intermediacy of substituted allylcarbene which undergoes both intramolecular cycloaddition and rearrangements involving 1,2-hydride and 1,2-vinyl shifts.

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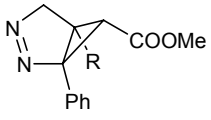
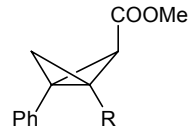
It is known [1–8] that substituted 2,3-diazabicyclo-[3.1.0]hex-2-enes undergo thermal decomposition with elimination of nitrogen molecule to produce buta-1,3-diene and bicyclo[1.1.0]butane derivatives. The ratio of the decomposition products, in particular the fraction of bicyclobutane derivative, strongly depends on the substitution pattern in the initial compound. In order to elucidate this dependence, it is necessary to study thermal decomposition of a wider series of substrates. As such substrates we selected methyl 1,5-diphenyl- and 5-methyl-1-phenyl-2,3-diazabicyclo-[3.1.0]hex-2-ene-6-*exo*-carboxylates **I** and **II** which were used by us previously as pyridazine precursors [9]. Komendantov et al. [2] reported that the thermolysis of ester **I** yields *endo*-bicyclobutane **IIIa** as the only product. We examined the thermal decomposition of **I** in more detail with a view to thoroughly determine the product composition and compare it with the composition of the thermolysis products of **II**.

It was found that the decomposition patterns of compounds **I** and **II** are essentially similar and much more complex than it was reported in [2]. In each case, two diastereoisomeric bicyclobutane derivatives (**IIIa** and **IIIb** from **I** and **IVa** and **IVb** from **II**) and three buta-1,3-diene derivatives (**Va**, **VIa**, and **VIb** from **I** and **VIIa**, **VIIIa**, and **VIIIb** from **II**) were formed. The compositions of the thermolysis products, determined by $^1$H NMR, are given in Table 1. All compounds were isolated as individual substances by chromatography. Bicyclobutane **IIIa** was described previously [2, 10];

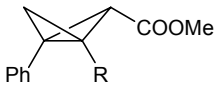



**I**, **II**

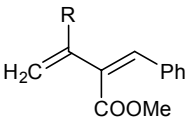
**I**, R = Ph; **II**, R = Me.



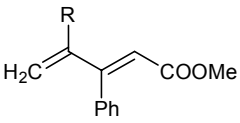

**IIIa**, **IVa**



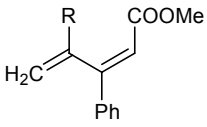

**IIIb**, **IVb**




**Va**, **VIIa**




**VIa**, **VIIIa**




**VIb**, **VIIIb**

**IIIa**, **IIIb**, **Va**, **VIa**, **VIb**, R = Ph; **IVa**, **IVb**, **VIIa**, **VIIIa**, **VIIIb**, R = Me.

compound **IIIb** was synthesized in [11] by thermal isomerization of **IIIa**. Bicyclobutanes **IVa** and **IVb** were identified on the basis of their $^1$H and $^{13}$C NMR spectra which were compared with the spectra of **IIIa** and **IIIb**. As expected, similarity in the chemical shifts of the 2-H and 4-H protons and $C^1$ ($C^3$), $C^2$, and $C^4$ carbon nuclei was observed for bicyclobutanes with similar configuration (**IIIa**/**IVa** and **IIIb**/**IVb**) (Table 2). The above proton signals of **IIIa** and **IIIb** were displaced downfield relative to the corresponding signals of **IVa** and **IVb**.

The key point in the determination of configuration of stereoisomers **IIIa**/**IIIb** and **IVa**/**IVb** was splitting of the *exo*-2-H and *exo*-4-H proton signals in the spectra of **IIIa** and **IVa** due to long-range *W*-coupling [12] which is possible only in the *endo* isomers. An additional evidence in support of structure **IVb** may be similarity of the 2-H and $C^2$ chemical shifts with those characteristic of model dimethyl 1-methyl-3-phenylbicyclobutane-2-*exo*,4-*exo*-dicarboxylate (**IX**) (δ 1.68, $\delta_C$ 41.0 ppm). All signals from the ring protons in *exo* isomers **IIIb** and **IVb** and from *endo*-4-H in *endo* isomers **IIIa** and **IVa** appeared as singlets or weakly split doublets due to small (0–1.4 Hz) geminal coupling constant typical of bicyclobutanes and the lack of long-range spin–spin coupling.

There were no problems in the structure assignment of three known buta-1,3-diene derivatives **VIIa** [13, 14], **VIIIa** [13, 15], and **VIIIb** [13] which were identified by comparing their $^1$H NMR spectra with published data.

Dienes **Va**, **VIa**, and **VIb** were not reported previously. However, ethyl ester analog of **VIa** was described in [16], and compound **VIa** was identified on the basis of the observed similarity in the chemical shifts of 2-H and 5-H in these esters. The structure of diene **VIb** was determined by comparing its $^1$H NMR spectrum with those of related dimethyl (*E*,*E*)- and (*Z*,*Z*)-3,4-diphenylhexa-2,4-dienedioates **Xa** and **Xb**. The structure of **VIb** was also confirmed by similarity of the chemical shifts of 2-H in **Vb** (δ 6.47 ppm) and **Xb** (δ 6.60 ppm). Likewise, the chemical shifts of 2-H in **VIa** (δ 5.97 ppm) and **Xa** (δ 5.80 ppm) indicated their similar configurations.

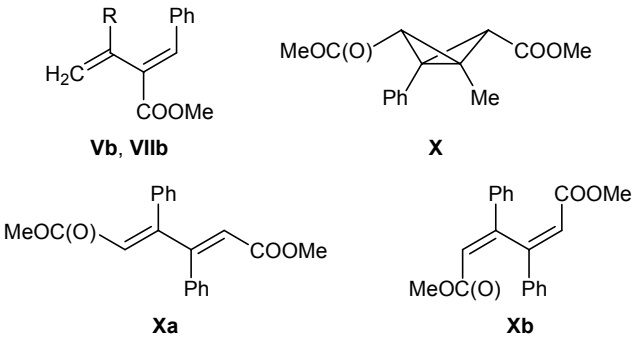
**Table 1.** Composition of the thermolysis products of compounds **I** and **II**

| Compound **I** | | Compound **II** | |
|---|---|---|---|
| product | fraction, % | product | fraction, % |
| **IIIa** | 71.2 | **IVa** | 53.5 |
| **IIIb** | 5.3 | **IVb** | 17.1 |
| **Va** | 5.9 | **VIIa** | 13.3 |
| **VIa** | 12.7 | **VIIIa** | 7.0 |
| **VIb** | 4.9 | **VIIIb** | 9.1 |

The structure of diene **Va** as stereoisomer of methyl (*E*)-2-benzylidene-3-phenylbut-3-enoate (**Vb**) reported in [13] followed from the position of the PhC**H**= proton signal (δ 6.69 ppm), which was similar to the position of the corresponding signal of model diene **VIIa** (δ 6.70 ppm) but appreciably different from that observed for isomer **Vb** (δ 7.67 ppm).

It should be noted that neither diene **Vb** nor its analog **VIIb** [17] (which are characterized by anomalously large chemical shift of the olefinic proton in the benzylidene fragment, δ 7.67 and 7.47 ppm, respectively) was detected among the thermolysis products of compounds **I** and **II**.

Some authors believe [1, 3, 18] that thermal decomposition of 2,3-diazabicyclo[3.1.0]hex-2-enes involves intermediate formation of diazo compound **A** which generates allylcarbene **B**. Isomerization of the latter may follow either intramolecular cycloaddition pattern with formation of bicyclobutane structure or




**V**, R = Ph; **VII**, R = Me.

**Scheme 1.**

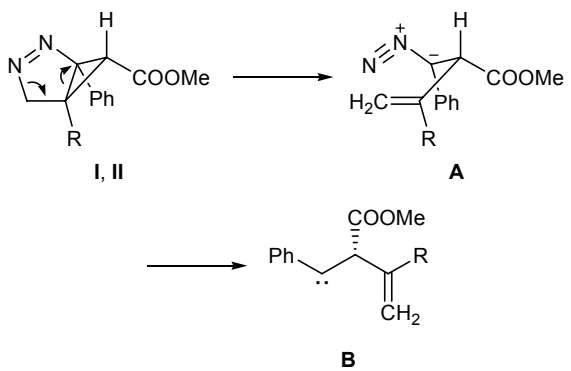


**Table 2.** Chemical shifts of selected protons and carbon nuclei in bicyclobutanes **IIIa**, **IIIb**, **IVa**, and **IVb**

| Compound no. | Chemical shifts δ, ppm (*J*, Hz) | | | | | |
|---|---|---|---|---|---|---|
| | 2-H | *exo*-4-H | *endo*-4-H | $C^1$, $C^3$ | $C^2$ | $C^4$ |
| **IIIa** | 4.04 d (4.3) | 2.96 d.d (4.3, 1.4) | 2.70 d (1.4) | 31.4 | 46.7 | 35.1 |
| **IVa** | 3.26 d (4.3) | 2.18 d.d (4.3, 1.0) | 2.32 br.s | 24.6 | 49.1 | 33.7 |
| **IIIb** | 2.21 s | 2.12 s | 1.32 s | 28.8 | 44.9 | 33.2 |
| **IVb** | 1.78 s | 1.59 s | 0.92 s | 22.7 | 43.4 | 32.5 |

1,2-hydride and/or 1,2-vinyl shifts with formation of buta-1,3-diene derivatives (Scheme 1). As applied to our case, the above mechanism rationalizes formation of all detected thermolysis products of compounds **I** and **II**. However, the absence of dienes **Vb** and **VIIb** among the products and hence the statement of strict stereoselectivity of the 1,2-hydride shift in carbene **B**, in contrast to non-stereoselective 1,2-vinyl shift, requires special discussion.

The large fraction of bicyclobutane derivatives (76.5 and 70.6%, respectively) in the thermolysis products of compounds **I** and **II** is a specific feature of the substitution pattern in these 2,3-diazabicyclo-[3.1.0]hex-2-ene derivatives bearing a phenyl group in the bridgehead position ($C^1$) and *exo*-oriented methoxycarbonyl group on $C^6$. Presumably, substituted phenyl(allyl)carbene **B** appears to be fairly stable, which ensures intramolecular cyclopropanation. On the other hand, the presence of a methoxycarbonyl group in the α-position with respect to the carbene center in intermediate **B** favors its rearrangement to butadiene derivatives. The latter assumption is supported by the data of [7], according to which thermal decomposition of an analog of **I** with a different substituent on $C^6$, 1,5-diphenyl-6-*exo*-(phenylselanylmethyl)-2,3-diazabicyclo[3.1.0]hex-2-ene, yields only the corresponding bicyclobutane derivative. The ratio of stereoisomers **IIIa** and **IIIb** formed by thermolysis of 2,3-diazabicyclo[3.1.0]hex-2-ene (**I**) corresponds to the thermodynamic equilibrium (~13.5:1) [11], whereas the ratio **IVa**/**IVb** (3:1) in the transformation of 2,3-diazabicyclo[3.1.0]hex-2-ene **II** is determined by the kinetic factor, for compounds **IVa** and **IVb**, unlike their analogs **IIIa** and **IIIb**, are not interconvertible up to 170°C.

## EXPERIMENTAL

The elemental compositions were determined on a Hewlett Packard HP-185B CHN analyzer. The $^1$H and $^{13}$C NMR spectra were measured on a Bruker DPX-300 spectrometer at 300.130 and 75.468 MHz, respectively, using $CDCl_3$ as solvent. Analytical thin-layer chromatography was performed on Silufol UV-254 plates using petroleum ether–diethyl ether (4:1) as eluent; spots were developed by treatment with iodine vapor. Silica gel L (40–100 μm) was used for column chromatography; eluent petroleum ether–diethyl ether. Compounds **I** and **II** were synthesized according to the procedure described in [9].

**Thermolysis of methyl 1,5-diphenyl-2,3-diazabicyclo[3.1.0]hex-2-ene-6-carboxylate (I).** *a. Preparative experiment*. Ester **I**, 1.0 g, was added in one portion to 5 ml of boiling *p*-xylene (bp 138°C), and the solution was heated for 15 min under reflux in an argon atmosphere. The mixture was cooled to 20°C, the solvent was removed under reduced pressure, and the residue, 910 mg of a light yellow viscous oily material, was dissolved on heating in 10 ml of petroleum ether–diethyl ether (5:1). The solution was kept for a long time at –5°C, and 140 mg of crystalline bicyclobutane **IIIa** was filtered off. The mother liquor was subjected to column chromatography on silica gel to isolate an additional portion (343 mg) of **IIIa** and compounds **IIIb**, **Va**, **VIa**, and **VIb**.

**Methyl 1,3-diphenylbicyclobutane-2-*endo*-carboxylate (IIIa).** Yield 483 mg (53%), $R_f$ 0.25, mp 74–75°C (from hexane); published data [2]: mp 75°C. The $^1$H and $^{13}$C NMR spectra of **IIIa** were identical to those reported in [10] (Table 2).

**Methyl 1,3-diphenylbicyclobutane-2-*exo*-carboxylate (IIIb).** Yield 35 mg (4%), colorless oily substance, $R_f$ 0.30. The $^1$H and $^{13}$C NMR spectra of **IIIb** were identical to those reported in [10] (Table 2).

**Methyl (*Z*)-2-benzylidene-3-phenylbut-3-enoate (Va).** Yield 38 mg (4%), colorless oily substance, $R_f$ 0.28. $^1$H NMR spectrum, δ, ppm: 3.75 s (3H, $CO_2CH_3$), 5.42 s and 5.45 s (1H each, $=CH_2$), 6.69 s (1H, =CH), 7.24–7.36 m (10H, Ph). $^{13}$C NMR spectrum, $\delta_C$, ppm: 52.1, 116.8, 127.9, 128.2, 128.3, 128.35, 128.4, 128.45, 133.8, 135.3, 135.6, 139.6, 146.7, 167.6. Found, %: C 81.59; H 6.33. $C_{18}H_{16}O_2$. Calculated, %: C 81.79; H 6.10.

**Methyl (*E*)-3,4-diphenylpenta-2,4-dienoate (VIa).** Yield 73 mg (8%), colorless oily substance, $R_f$ 0.26. $^1$H NMR spectrum, δ, ppm: 3.57 s (3H, $CO_2CH_3$), 5.23 s and 5.56 s (1H each, $=CH_2$), 5.97 s (1H, =CH), 7.31–7.49 m (10H, Ph). $^{13}$C NMR spectrum, $\delta_C$, ppm: 51.2, 120.1, 122.9, 127.75, 127.8, 127.9, 128.3, 128.6, 128.7, 137.9, 140.0, 151.0. 156.5, 166.6. Found, %: C 81.62; H 6.29. $C_{18}H_{16}O_2$. Calculated, %: C 81.79; H 6.10.

**Methyl (*Z*)-3,4-diphenylpenta-2,4-dienoate (VIb).** Yield 26 mg (3%), colorless oily substance, $R_f$ 0.24. $^1$H NMR spectrum, δ, ppm: 3.65 s (3H, $CO_2CH_3$), 5.25 s and 5.92 s (1H each, $=CH_2$), 6.47 s (1H, =CH), 7.26–7.34 m (3H), 7.36–7.42 m (3H), 7.44–7.52 m (2H), 7.57–7.66 m (2H). $^{13}$C NMR spectrum, $\delta_C$, ppm: 51.2, 115.2, 118.0, 126.3, 127.5, 127.7, 128.4, 128.6, 129.5, 138.6, 138.7. 146.2, 156.0, 166.3. Found, %: C 81.69; H 6.24. $C_{18}H_{16}O_2$. Calculated, %: C 81.79; H 6.10.

*b*. *Analytical experiment*. Compound **I**, 50 mg, was added in one portion to 1.0 ml of boiling *p*-xylene, and the mixture was heated for 15 min under reflux in an argon atmosphere. The solvent was removed under reduced pressure to leave 45 mg of a light yellow viscous oily material which was analyzed by $^1$H NMR spectroscopy to reveal five compounds at a ratio indicated in Table 1.

**Thermolysis of methyl 5-methyl-1-phenyl-2,3-diazabicyclo[3.1.0]hex-2-ene-6-carboxylate (II).** *a*. *Preparative experiment*. Ester **II**, 750 mg, was added in one portion to 7.5 ml of boiling *p*-xylene. The solution was heated for 30 min under reflux, the solvent was removed under reduced pressure, and the light yellow oily residue, 660 mg, was subjected to chromatographic separation on silica gel to isolate individual compounds **IVa**, **IVb**, and **VIIIb**. In addition, a fraction containing dienes **VIIa** and **VIIIa** at a ratio of ~2:1 was isolated [$R_f$ 0.34, colorless oily substance, yield 102 mg (16%). Found, %: C 78.19; H 5.64. $C_{13}H_{11}O_2$. Calculated, %: C 78.37; H 5.57]. Signals of each components were identified in the $^1$H NMR spectrum of that fraction.

**Methyl 1-methyl-3-phenylbicyclobutane-2-*endo*-carboxylate (IVa).** Yield 245 mg (37%), colorless oily substance, $R_f$ 0.33**.** $^1$H NMR spectrum, δ, ppm: 1.40 s (3H, $CH_3$), 2.18 d.d (1H, *exo*-4-H, $^2J$ = 1.0, $^4J$ = 4.3 Hz), 2.32 br.s (1H, *endo*-4-H), 3.26 d (1H, *exo*-2-H, $J$ = 4.3 Hz), 3.71 s (3H, $CO_2CH_3$), 7.20–7.30 m (2H, Ph), 7.32–7.42 m (3H, Ph). $^{13}$C NMR spectrum, $\delta_C$, ppm: 12.3 ($CH_3$), 20.5 ($C^1$), 24.6 ($C^3$), 33.7 ($C^4$), 49.1 ($C^2$), 51.5 ($OCH_3$), 125.3 (2C), 125.6, 128.3 (2C), 136.7, 171.7 (C=O). Found, %: C 78.09; H 5.64. $C_{13}H_{11}O_2$. Calculated, %: C 78.37; H 5.57.

**Methyl 1-methyl-3-phenylbicyclobutane-2-*exo*-carboxylate (IVb).** Yield 79 mg (12%), colorless oily substance, $R_f$ 0.36. $^1$H NMR spectrum, δ, ppm: 0.92 s (1H, *endo*-4-H), 1.59 s (1H, *exo*-4-H), 1.78 s (1H, *endo*-2-H), 1.79 s (3H, $CH_3$), 3.69 s (3H, $CO_2CH_3$), 7.20–7.27 m (3H) and 7.28–7.36 m (2H) (Ph). $^{13}$C NMR spectrum, $\delta_C$, ppm: 7.3 ($CH_3$), 22.5 ($C^1$), 22.7 ($C^3$), 32.5 ($C^4$), 43.4 ($C^2$), 51.3 ($OCH_3$), 126.1, 127.7 (2C), 128.1 (2C), 134.2, 168.2 (C=O). Found, %: C 78.31; H 5.59. $C_{13}H_{11}O_2$. Calculated, %: C 78.37; H 5.57.

**Methyl (*Z*)-2-benzylidene-3-methylbut-3-enoate (VIIa).** $^1$H NMR spectrum, δ, ppm: 2.07 s (3H, $CH_3$), 3.76 s (3H, $CO_2Me$), 5.09 s and 5.22 s (1H each, $=CH_2$), 6.70 s (1H, =CH), 7.25–7.37 m (5H, Ph).

**Methyl (*Z*)-4-methyl-3-phenylpenta-2,4-dienoate (VIIIa).** $^1$H NMR spectrum, δ, ppm: 2.06 s (3H, $CH_3$), 3.56 s (3H, $CO_2Me$), 4.88 s and 5.37 s (1H each, $=CH_2$), 6.09 s (1H, =CH), 7.21–7.46 m (5H, Ph).

**Methyl (*E*)-4-methyl-3-phenylpenta-2,4-dienoate (VIIIb).** Yield 42 mg (6.4%), colorless oily substance, $R_f$ 0.31. $^1$H NMR spectrum, δ, ppm: 1.97 s (3H, $CH_3$), 3.76 s (3H, $CO_2Me$), 4.92 s and 5.27 s (1H each, $=CH_2$), 6.15 s (1H, =CH), 7.27–7.56 m (5H, Ph). Found, %: C 78.49; H 5.34. $C_{13}H_{11}O_2$. Calculated, %: C 78.37; H 5.57.

*b*. *Analytical experiment*. Compound **II**, 50 mg, was added in one portion to 1.0 ml of boiling *p*-xylene, and the solution was heated for 30 min under reflux in an argon atmosphere. The solvent was removed under reduced pressure, and the light yellow viscous oily residue was analyzed by $^1$H NMR (Table 1).

**Dimethyl 1-methyl-3-phenylbicyclo[1.1.0]-butane-2,4-dicarboxylate (IX)** was synthesized according to the procedure described in [19]. $^1$H NMR spectrum, δ, ppm: 1.68 s (2H, *endo*-2-H, *endo*-4-H), 1.98 s (3H, $CH_3$), 3.60 s (6H, $CO_2CH_3$), 7.28–7.40 m (3H, Ph), 7.48–7.58 (2H, Ph). $^{13}$C NMR spectrum, $\delta_C$, ppm: 4.2 ($CH_3$), 24.3 ($C^1$), 29.2 ($C^3$), 41.0 ($C^2$, $C^4$), 51.4 ($OCH_3$), 127.8 (2C), 127.85 (2C), 129.6, 131.0, 167.4.

**Dimethyl (*E*,*E*)-3,4-diphenylhexa-2,4-dienedioate (IXa)** was synthesized according to the procedure described in [20]. $^1$H NMR spectrum, δ, ppm: 3.50 s (6H, $CO_2CH_3$), 5.80 s (2H, 2-H, 5-H), 7.22–7.30 (4H) and 7.40–7.50 (6H) (Ph). $^{13}$C NMR spectrum, $\delta_C$, ppm: 51.3, 125.3, 128.2, 128.24, 128.6, 136.7, 155.9, 166.0.

**Dimethyl (*Z*,*Z*)-3,4-diphenylhexa-2,4-dienedioate (IXb)** was synthesized as described in [21]. $^1$H NMR spectrum, δ, ppm: 3.64 s (6H, $CO_2CH_3$), 6.60 s (2H, 2-H, 5-H), 7.33–7.40 (6H) and 7.53–7.60 (4H) (Ph). $^{13}$C NMR spectrum, $\delta_C$, ppm: 51.3, 116.7, 127.0, 128.7, 129.6, 137.6, 154.4, 165.4.

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