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Asymmetric Syntheses with BINOL-Based Imidoyl Azide*

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Abstract—Thermal decomposition of 2,4,6-trimethylphenyl and (+)-2′-methoxy-1,1′-binaphthalen-2-yl (4-methylphenylsulfonyl)azidimidocarbonates in the presence of norbornene, methyl acrylate, and camphene was studied. (+)-2′-Methoxy-1,1′-binaphthalen-2-yl (4-methylphenylsulfonyl)azidimidocarbonate reacted with norbornene to give (+)-*exo*-2′-methoxy-1,1′-binaphthalen-2-yl *N*-(4-methylphenylsulfonyl)-3-azatricyclo-[3.2.1.$0^{2,4}$]octane-3-carboximidoate, while in the reaction with methyl acrylate a mixture of stereoisomeric methyl 1-[(2′-methoxy-1,1′-binaphthalen-2-yloxy)(4-methylphenylsulfonylimino)methyl]aziridine-2-carboxylates was obtained. The reaction of 2,4,6-trimethylphenyl (4-methylphenylsulfonyl)azidimidocarbonate with (–)-camphene involved insertion of intermediate nitrene into the exocyclic double bond with formation of 2,4,6-trimethylphenyl *N*-(3,3-dimethylbicyclo[2.2.1]heptan-2-ylidenemethyl)-*N*′-(4-methylphenylsulfonyl)-imidocarbamate as a 3 : 1 mixture of *E*,*E* and *E*,*Z* diastereoisomers in good yield.

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Despite the lack of tetrazole compounds in natural systems, the chemistry of these heterocycles has attracted increasing attention since early 1980s [1]. This interest stems from the ability of tetrazoles to mimic carboxy group, which stimulated incorporation of tetrazole fragment into biologically active molecules [2]. Another area of interest is therapeutic application of tetrazole derivatives some of which exhibit antihypertensive, antiallergic, and antibiotic activity [2]. Tetrazoles have found wide application in agriculture, biology [3], and preparation of explosives [4]. An important aspect of tetrazole chemistry that have not been considered carefully is the use of their isomeric imidoyl azide form as a source of azide in asymmetric synthesis. Imidoyl azides are used for the synthesis of aziridines, functionalization of aromatic compounds, and preparation of sulfoximines [5–7]. It is believed that imidoyl azides having electron-withdrawing groups are more stable and more selective than other organic azides [5, 6, 8, 9].

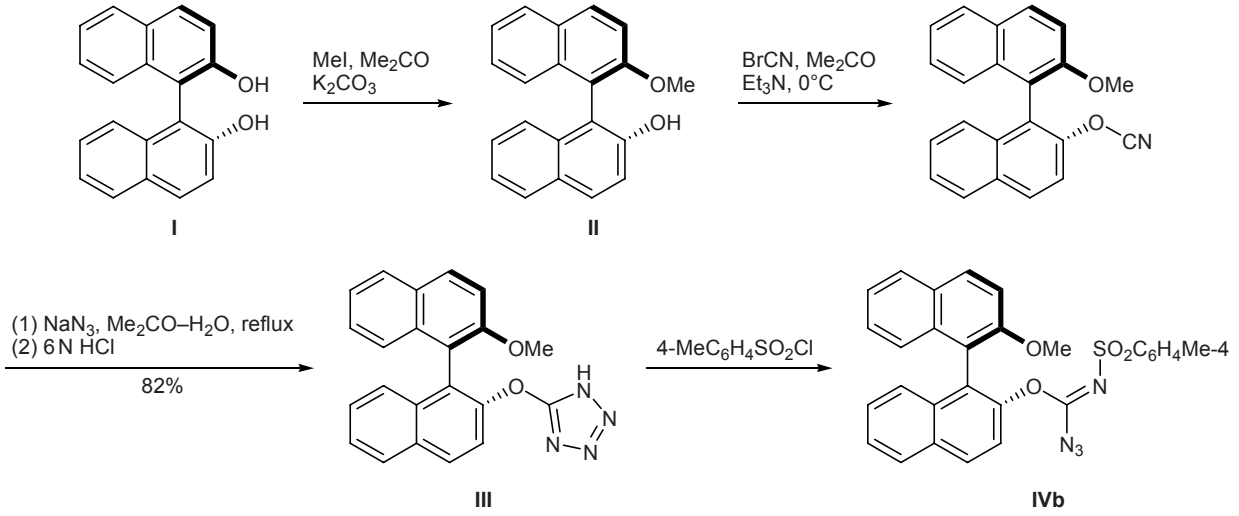
In continuation of our studies on the chemistry of tetrazoles and related compounds such as imidoyl azides and imidoyl nitrenes [7, 9–15], the present article reports on stereoselective asymmetric synthesis of a series of new compounds derived from tetrazoles. In addition, we describe here the synthesis of optically active (+)- or (–)-5-(2′-methoxy-1,1′-binaphthalen-2-yloxy)-1*H*-tetrazole (**IIIb**) as the first aryloxy tetrazole with axial chirality and a chiral pool for asymmetric synthesis.

(±)-2′-Methoxy-1,1′-binaphthalen-2-ol (**II**) was prepared by methylation of 1,1′-binaphthalene-2,2′-diol (BINOL) with methyl iodide in acetone and was purified by repeated crystallization from carbon tetrachloride. The reaction was performed at room temperature, and it was not accompanied by racemization. In the next step, (±)-**II** reacted with cyanogen bromide to produce (±)-2′-methoxy-1,1′-binaphthalen-2-yl cyanate, and the latter was treated with sodium azide in aqueous acetone. The conditions for the reaction of **II** with BrCN were optimized by varying the solvent and the amount of triethylamine. Under the optimal conditions (1 : 1 acetone–water mixture, 1.5 equiv of triethylamine) the yield of aryloxytetrazole (**IIIb**) reached 82% (Scheme 1).

Imidoyl azides are generally prepared by reaction of tetrazoles with such electrophiles as TsCl, BrCN, MsCl, etc., in THF [5, 6, 9]. Tetrazoles **IIIa** and **IIIb** were converted into the corresponding imidoyl azides **IVa** and **IVb** using different solvents at room temperature. The best yields were obtained in acetone (reaction time 2 h; see table). Azides **IVa** and **IVb** are

* The text was submitted by the authors in English.

Scheme 1.



stable for several days at room temperature, and they decompose at 104 and 96°C, respectively. Optically active azide (+)-**IVb** was synthesized under similar conditions.

Azides **IVa** and **IVb** were then brought into reactions with some unsaturated compounds, namely norbornene, methyl acrylate, and camphene. The reactions of **IVa** and **IVb** with norbornene gave aziridine derivatives **Va** and **Vb**, respectively (Scheme 2). It is generally accepted that the mechanism of formation of aziridine ring from organic azides involves thermal decomposition of the latter to give nitrene species which add at double bond of the substrate [5, 6, 9–11]. In the case of strained olefins, such as norbornene, the reaction at low temperature is presumed to produce 4,5-dihydro-1,2,3-triazole intermediate. This intermediate is stable in the cold with protection from light, but it decomposes above room temperature or on exposure to light with liberation of nitrogen and formation of the corresponding aziridine [8]. Wijnen et al. [16] succeeded in isolating intermediate dihydrotriazole in the reaction of norbornene with phenyl azide. The reactions of **IVa** and **IVb** with norbornene at room temperature were very slow, but at 40°C they were complete in 2 h (according to the TLC data). No intermediate dihydrotriazoles were detected in the reaction mixture.

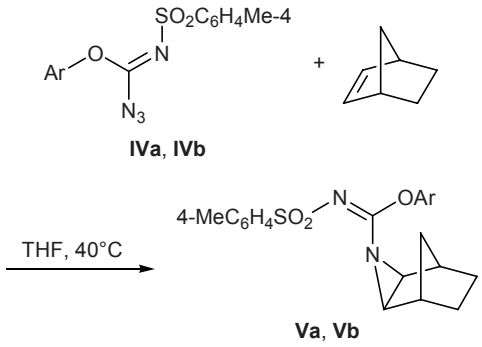
Transformation of tetrazole derivatives **IIIa** and **IIIb** into imidoyl azides **IVa** and **IVb**

Initial comp. no.	Solvent	Reaction time, h	Product no.	Yield, %
IIIa	Methylene chloride	12	**IVa**	40[a]
IIIa	Tetrahydrofuran	12	**IVa**	55[a]
IIIa	Ethyl acetate	6	**IVa**	75[a]
IIIa	Acetone	2	**IVa**	75[b]
IIIb	Ethyl acetate	5	**IVb**	62[a]
IIIb	Acetone	2	**IVb**	50[b]

[a] Yields (isolated) after purification through column chromatography.

[b] Yield (isolated) after recrystallization.

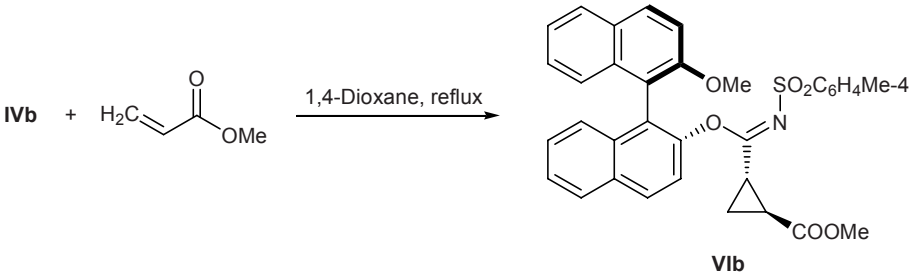
Scheme 2.



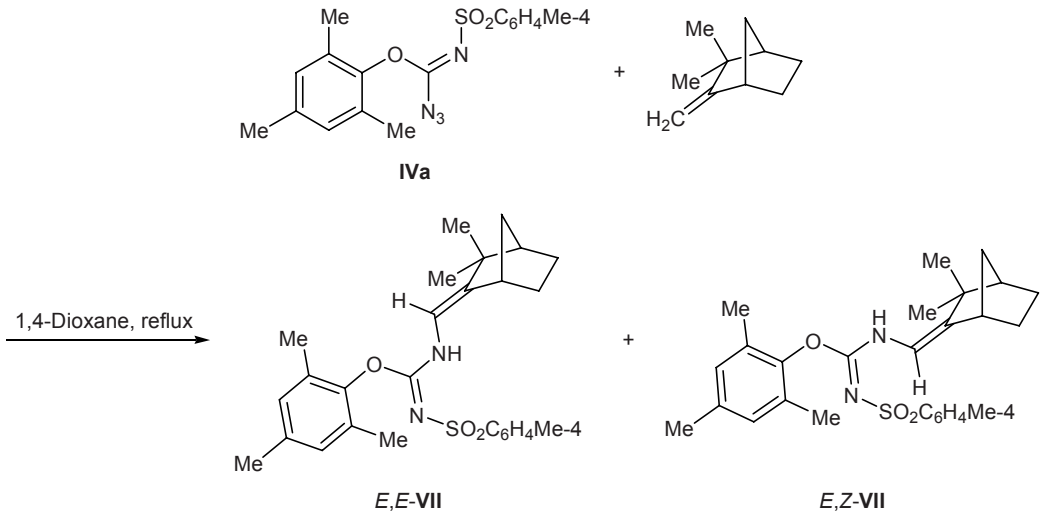
Ar = 2,4,6-$Me_3C_6H_2$ (**a**), 2′-methoxy-1,1′-binaphthalen-2-yl (**b**).

Azide **IVb** failed to react with methyl acrylate at room temperature over a period of 24 h, while heating the reactants in boiling 1,4-dioxane for 12 h gave a mixture of stereoisomeric methyl 1-[(2′-methoxy-1,1′-binaphthalen-2-yloxy)(4-methylphenylsulfonylimino)methyl]aziridine-2-carboxylates (**VIb**) in 35% yield (Scheme 3). We failed to separate this mixture into individual stereoisomers.

Scheme 3.



Scheme 4.



No reaction was observed between azide **IVa** and (–)-camphene in tetrahydrofuran at 40°C. The reaction in 1,4-dioxane under reflux was accompanied by evolution of nitrogen (formation of nitrene), and the product was 2,4,6-trimethylphenyl *N*-(3,3-dimethylbicyclo[2.2.1]heptan-2-ylidenemethyl)-*N*′-(4-methylphenylsulfonyl)imidocarbamate (**VII**) as a mixture of *E*,*E* and *E*,*Z* isomers at a ratio of 3:1 (overall yield 54%; Scheme 4).

The structure of **VII** was determined by NMR spectroscopy using NOE and differential NOE techniques. Irradiation of protons of the geminal methyl groups in the camphene fragment (δ 1.10 ppm) induced increase in the intensity of the doublet signal at δ 6.25 ppm from the C=CH proton of the major isomer (*E*,*E*) and decrease in the intensity of the corresponding signal of the minor isomer (*E*,*Z*, δ 6.46 ppm; Figs. 1–3).

EXPERIMENTAL

Potassium carbonate, norbornene, and solvents from Merck, methyl iodide from Riedel-Dehaen, and optically active 1,1′-binaphthalene-2,2′-diol from Fluka were used. Racemic 1,1′-binaphthalene-2,2′-diol was synthesized according to the procedure reported in [17]. The syntheses of 5-(2,4,6-trimethylphenyloxy)-1*H*-tetrazole (**IIIa**) and 2,4,6-trimethylphenyl (4-methylphenylsulfonyl)azidimidocarbonate (**IVa**) were described previously [13]. The IR spectra were recorded on a JASCO FT/IR-680 PLUS spectrometer. The NMR spectra were obtained on a Bruker Ultrashield spectrometer (500 MHz for ^{1}H) using $CDCl_3$ or DMSO-d_6 as solvent. The optical rotations were measured on a JASCO p-1030 polarimeter. The mass spectra were recorded on a Fisons Trio 1000 spectrometer (UK). Elemental analysis was performed at the Research Institute of Petroleum Industry (Pazhouheshgah Bld., Qom Road, Tehran, Iran). The melting points were determined on a Gallenkamp apparatus and are not corrected. Silica gel 60 (Merck, 40–63 μm) was used for flash chromatography. All solvents were purified by standard procedures prior to use.

5-(2′-Methoxy-1,1′-binaphthalen-2-yloxy)-1*H*-tetrazole (IIIb). Cyanogen bromide, 0.27 g (2.5 mmol), was added to a solution of 0.6 g (2.0 mmol) of 2′-methoxy-1,1′-binaphthalen-2-ol in acetone on cool-

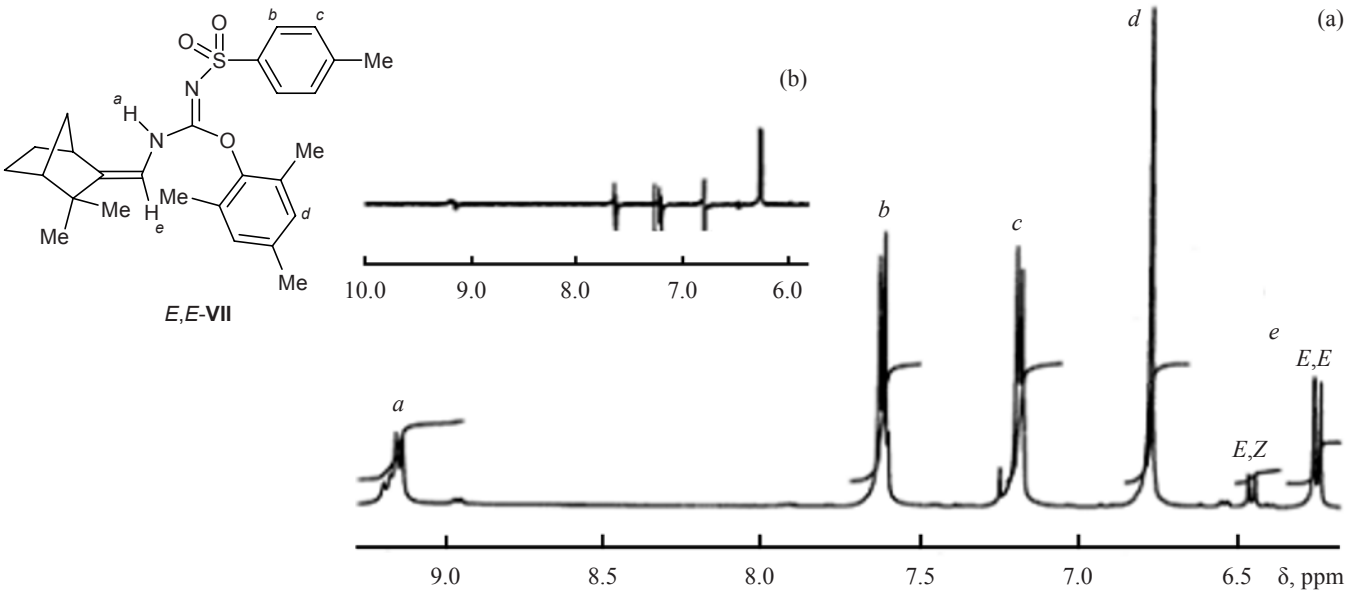


Fig. l. (a) A fragment of the ^{1}H NMR spectrum of 2,4,6-trimethylphenyl *N*-(3,3-dimethylbicyclo[2.2.1]heptan-2-ylidenemethyl)-*N*′-(4-methylphenylsulfonyl)imidocarbamate (**VII**, a mixture of *E*,*E* and *E*,*Z* isomers at a ratio of 3:1) in $CDCl_3$ and (b) its differential NOE spectrum (irradiation at δ 1.10 ppm).

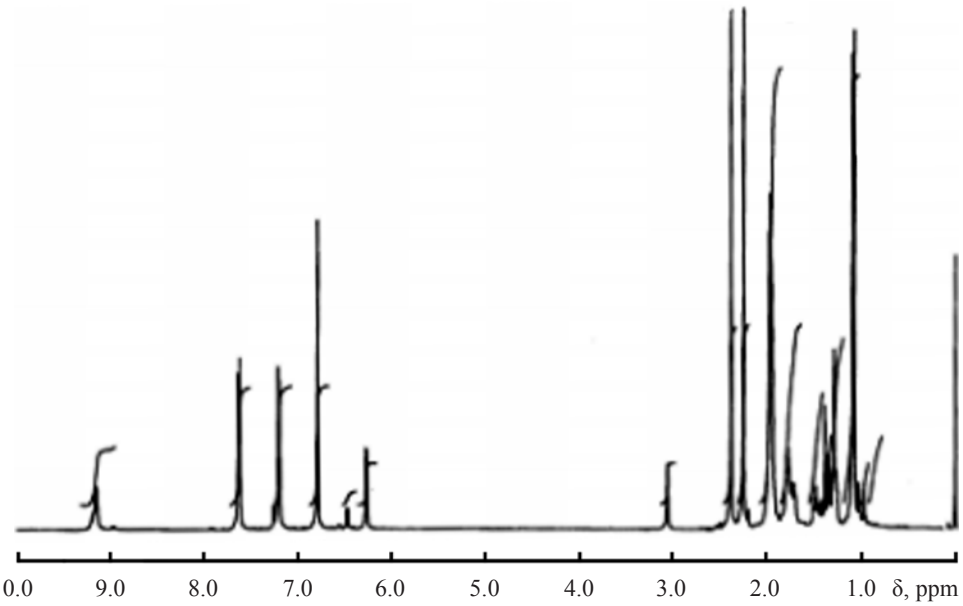


Fig. 2. ^{1}H NMR spectrum of 2,4,6-trimethylphenyl *N*-(3,3-dimethylbicyclo[2.2.1]heptan-2-ylidenemethyl)-*N*′-(4-methylphenylsulfonyl)imidocarbamate (**VII**, a mixture of *E*,*E* and *E*,*Z* isomers at a ratio of 3:1) in $CDCl_3$.

ing to 0°C. Triethylamine, 0.4 ml (3.0 mmol), was then added dropwise under stirring over a period of 0.5 h, the mixture was stirred for 0.5 h, the precipitate of triethylamine hydrochloride was filtered off, and a solution of 0.16 g (2.5 mmol) of sodium azide in aqueous acetone (50:50) was added to the filtrate. The mixture was stirred for 0.5 h at room temperature and heated for 2 h under reflux, cooled, and acidified with 6 N hydrochloric acid, and the precipitate of (±)-**IIIb** was filtered off and recrystallized from aqueous methanol (50:50). Yield 0.61 g (82%), colorless crystals, mp 236–238°C. IR spectrum (KBr), ν, cm^{-1}: 3053, 2731, 2626, 1619, 1590, 1506, 1458, 1261, 1053, 817. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.61 s (3H), 7.01–8.16 m (12H), 16.50 br.s (1H). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 55.9, 113.7, 115.8, 119.9, 123.3, 123.5, 124.1, 125.4, 125.7, 126.6, 127.0, 128.0, 128.2, 128.5, 129.8, 130.3, 131.3, 133.0, 133.1, 149.4,

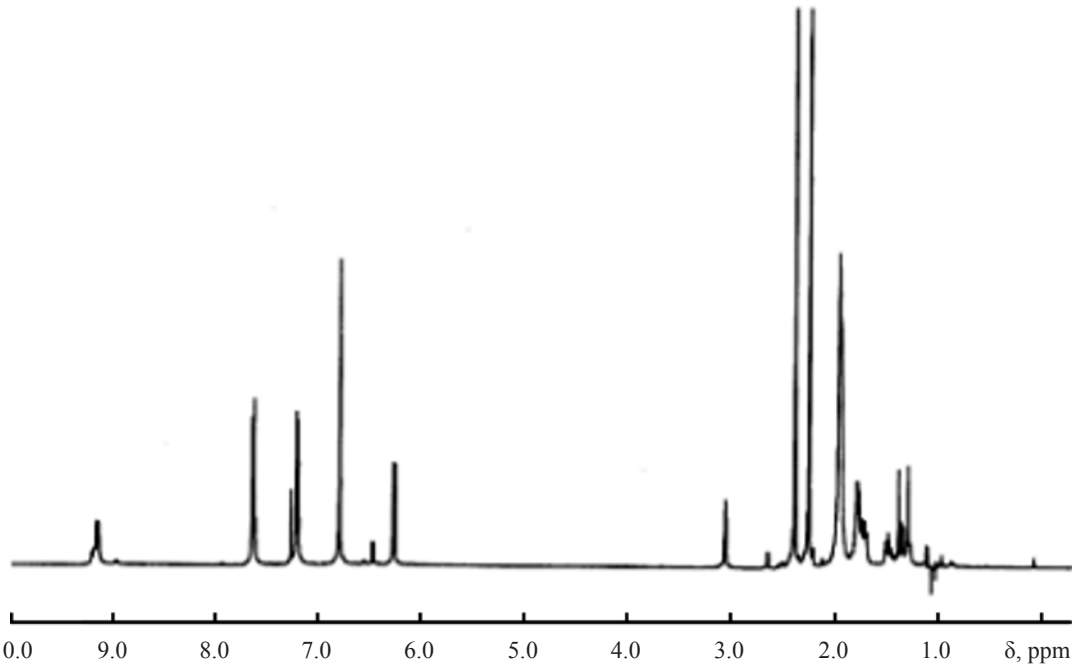


Fig. 3. NOE ^{1}H NMR spectrum of 2,4,6-trimethylphenyl *N*-(3,3-dimethylbicyclo[2.2.1]heptan-2-ylidenemethyl)-*N*′-(4-methylphenylsulfonyl)imidocarbamate (**VII**, a mixture of *E*,*E* and *E*,*Z* isomers at a ratio of 3:1) in $CDCl_3$ (irradiation at δ 1.10 ppm).

154.7. Mass spectrum, *m*/*z*: 368 $[M]^+$, 354, 311, 297, 286, 268, 239, 226, 134, 120, 43. Found, %: C 71.70; H 4.40; N 14.80. $C_{22}H_{16}N_4O_2$. Calculated, %: C 71.73; H 4.38; N 15.21.

Following an analogous procedure, from chiral 2′-methoxy-1,1′-binaphthalen-2-ols ($[\alpha]_D^{25}$ = +38.7 and –38.2°; published data [18]: $[\alpha]_D^{25}$ = +38.7 and –39.2°) we obtained optically active compounds (+)-**IIIb** and (–)-**IIIb**; $[\alpha]_D^{25}$ = +25.1 and –24.9 (acetone, *c* = 0.1), mp 128–130 and 130–132°C, respectively.

2′-Methoxy-1,1′-binaphthalen-2-yl (4-methylphenylsulfonyl)azidimidocarbonate (IVb). 4-Methylbenzenesulfonyl chloride, 0.19 g (1.0 mmol), was added at room temperature to a solution of 0.37 g (1.0 mmol) of tetrazole **IIIb** in acetone. Triethylamine, 0.15 ml (1.1 mmol), was then added dropwise over a period of 20 min. When the reaction was complete (2 h according to the TLC data), triethylamine hydrochloride was filtered off, the filtrate was evaporated under reduced pressure, and the residue was recrystallized from chloroform–hexane (9:1). Yield 75%, mp 96°C (decomp.). IR spectrum (KBr), ν, cm^{-1}: 3053, 2731, 2626, 1619, 1590, 1506, 1458, 1261, 1053, 817. Mass spectrum, *m*/*z*: 494 $[M - N_2]^+$, 339, 300, 268, 239, 155, 91.

2,4,6-Trimethylphenyl *N*-(4-methylphenylsulfonyl)-*exo*-3-azatricyclo[3.2.1.0^{2,4}]octane-3-carboximidoate (Va). A mixture of 0.36 g (1.0 mmol) of 2,4,5-trimethylphenyl (4-methylphenylsulfonyl)-azidimidocarbonate (**IVa**) and 0.18 g (2.0 mmol) of norbornene in 50 ml of anhydrous THF was heated for 3 h at 40°C under dry nitrogen. The mixture was cooled to room temperature and evaporated under reduced pressure, and the residue was passed through a column charged with silica gel using cyclohexane–ethyl acetate (8:2) as eluent. The product was additionally purified by recrystallization from chloroform–hexane. Yield 0.22 g (50%), mp 126–128°C. IR spectrum (KBr), ν, cm^{-1}: 2995, 2966, 1556, 1383, 1201, 1053, 817. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.75 d (1H, *J* = 10.0 Hz), 1.12 d (1H, *J* = 10.0 Hz), 1.18 d (2H, *J* = 8.18 Hz), 1.45 d (2H, *J* = 8.2 Hz), 1.98 s (6H), 2.25 s (3H), 2.37 s (3H), 2.95 s (2H), 6.80 s (2H), 7.19 d (2H, *J* = 8.0 Hz), 7.71 d (2H, *J* = 8.0 Hz). Mass spectrum, *m*/*z*: 424 $[M]^+$, 360, 343, 319, 269, 227, 155, 91. Found, %: C 70.1; H 6.70; N 6.9. $C_{24}H_{28}N_2O_3S$. Calculated, %: C 67.90; H 6.65; N 6.65.

2′-Methoxy-1,1′-binaphthalen-2-yl *N*-(4-methylphenylsulfonyl)-*exo*-3-azatricyclo[3.2.1.0^{2,4}]octane-3-carboximidoate (Vb). A mixture of 0.52 g (1.0 mmol) of compound **IVb** and 0.18 g (2.0 mmol) of norbornene in 50 ml of anhydrous THF was stirred for 2 h at 40°C under dry nitrogen. The mixture was cooled to room temperature, the solvent was removed under reduced pressure, and the residue was passed

through a short silica gel column using cyclohexane–ethyl acetate (8:2) as eluent. Yield 0.39 g (65%), colorless crystals, mp 164–166°C. IR spectrum (KBr), ν, cm^{-1}: 2995, 2966, 1556, 1383, 1183, 1147, 747. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.45 d (1H, *J* = 10.0 Hz), 0.74 d (1H, *J* = 10.0 Hz), 0.87 m (2H), 1.2 m (2H), 1.98 d (1H, *J* = 5 Hz), 2.10 s (1H), 2.15 d (1H, *J* = 5 Hz), 2.20 s (1H), 2.33 s (3H), 3.70 s (3H), 7.09–7.80 m (17H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 161.2, 155.1, 147.2, 139.9, 133.7, 133.6, 131.8, 130.0, 128.9, 128.8, 128.1, 127.8, 126.9, 126.5, 126.3, 125.7, 125.4, 125.1, 123.7, 121.6, 116.9, 113.5, 56.5, 43.22, 42.67, 36.0, 35.9, 27.6, 25.2,25.1, 21.4. Mass spectrum, *m*/*z*: 588 $[M]^+$, 454, 434, 300, 282, 268, 239, 155, 119, 91. Found, %: C 74.2; H 5.7; N 3.6. $C_{36}H_{32}N_2O_4S$. Calculated, %: C 73.5; H 5.5; N 4.7.

Following the above procedure but on a smaller scale, from chiral substrate (+)-**IVb** we obtained optically active compound **Vb** with $[\alpha]_D^{25}$ = +26.8° (acetone, *c* = 0.1).

Methyl 1-[(2′-methoxy-1,1′-binaphthalen-2-yloxy)(4-methylphenylsulfonylimino)methyl]aziridine-2-carboxylate (VIb). A solution of 0.52 g (1.0 mmol) of tetrazole **IVb** and 1.8 ml (15 mmol) of methyl acrylate in 50 ml of anhydrous 1,4-dioxane was heated for 12 h under reflux in a nitrogen atmosphere. The mixture was cooled to room temperature and evaporated under reduced pressure, and the residue was passed through a column charged with silica gel using cyclohexane–ethyl acetate (8:2) as eluent. The product was additionally purified by recrystallization from chloroform–hexane. Yield 0.2 g (35%), colorless crystals, mp 150–154°C, $[\alpha]_D^{25}$ = +17.1 (acetone, *c* = 0.05). IR spectrum (KBr), ν, cm^{-1}: 2955, 2922, 2847, 1749, 1569, 1502, 1208, 812. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.5–1.64 m (1H), 1.7–2.1 m (1H), 2.35 s (3H), 2.50–2.61 m (1H), 3.70 s (3H), 3.74 s (3H), 6.90–8.10 m (16H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 25.5, 28.0, 36.4, 50.1, 56.9, 113.9, 117.3, 122.0, 124.2, 125.5, 126.1, 126.8, 126.9, 127.3, 128.2, 128.5, 129.2, 129.3, 130.4, 132.4, 134.0, 140.2, 144.5, 147.6, 155.5, 161.6, 163.5, 171.7. Mass spectrum, *m*/*z*: 580 $[M]^+$), 565, 454, 425, 396, 376, 339, 325, 300, 268, 91. Found, %: C 68.1; H 4.9; N 5.1. $C_{33}H_{28}N_2O_6S$. Calculated, %: C 68.26; H 4.86; N 4.82.

2,4,6-Trimethylphenyl *N*-(3,3-dimethylbicyclo[2.2.1]heptan-2-ylidenemethyl)-*N*′-(4-methylphenylsulfonyl)imidocarbamate (VII, a mixture of *E*,*E* and *E*,*Z* isomers at a ratio of 3:1**).** A solution of 0.36 g (1.0 mmol) of 2,4,5-trimethylphenyl (4-methylphenylsulfonyl)azidimidocarbonate (**IVa**) and 0.15 g (1.1 mmol) of (–)-camphene ($[\alpha]_D^{25}$ = –45.9°; acetone, *c* = 0.05) in 50 ml of anhydrous 1,4-dioxane was heated for 10 h under reflux in a dry nitrogen atmosphere. The mixture was cooled to room temperature and evaporated under reduced pressure, and the residue was passed through a column charged with silica gel using cyclohexane–ethyl acetate (8:2) as eluent. The product was additionally purified by recrystallization from chloroform–hexane. Yield 0.25 g (54%), mp 140–148°C, $[\alpha]_D^{25}$ = –26.3 (acetone, *c* = 0.05). We failed to separate the mixture into individual stereoisomers. IR spectrum (KBr), ν, cm^{-1}: 3316, 3283, 3241, 2962, 2933, 2853, 1597, 1385, 1030, 848. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.0 m (1H), 1.1 d (6H), 1.2–1.5 m (6H), 1.6–1.8 m (3H), 1.9 br (7H), 2.2 s (3H), 2.4 s (3H), 6.25 d (*J* = 8 Hz), 6.46 d (*J* = 8 Hz), 6.77 s (2H), 7.19 d (2H, *J* = 8 Hz), 7.6 d (2H, *J* = 8 Hz), 9.1 m (1H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 37.4, 37.5, 40.7, 40.8, 41.5, 44.3, 45.6, 48.1, 50.5, 114.2, 116.2, 120.3, 123.7, 123.9, 124.6, 125.6, 126.1, 126.3, 127.0, 127.4, 128.5, 128.5, 128.7, 128.9, 130.2, 130.7, 131.7, 133.4, 133.5, 147.2, 155.1, 161.2. Mass spectrum, *m*/*z*: 466 $[M]^+$, 451, 397, 330, 311, 262, 175, 155, 136, 91. Found, %: C 69.50; H 7.34; N 6.00. $C_{27}H_{34}N_2O_3S$. Calculated, %: C 69.14; H 7.30; N 5.97.

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