Diels-Alder Reaction of (*E*)-4-(2-Nitroethenyl)-1*H*-imidazoles and Methyl (*E*)-3-(1-Imidazol-4-yl)propenoates

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Diels-Alder reaction of methyl (*E*)-3-(1*H*-imidazol-4-yl)propenoates **2**, **3a-c** and (*E*)-4-(2-nitroethenyl)-1*H*-imidazoles **3d,e** with 2,3-dimethyl-1,3-butadiene, cyclopentadiene, and cyclohexa-1,3-diene gave the corresponding cycloadducts **6-9**.

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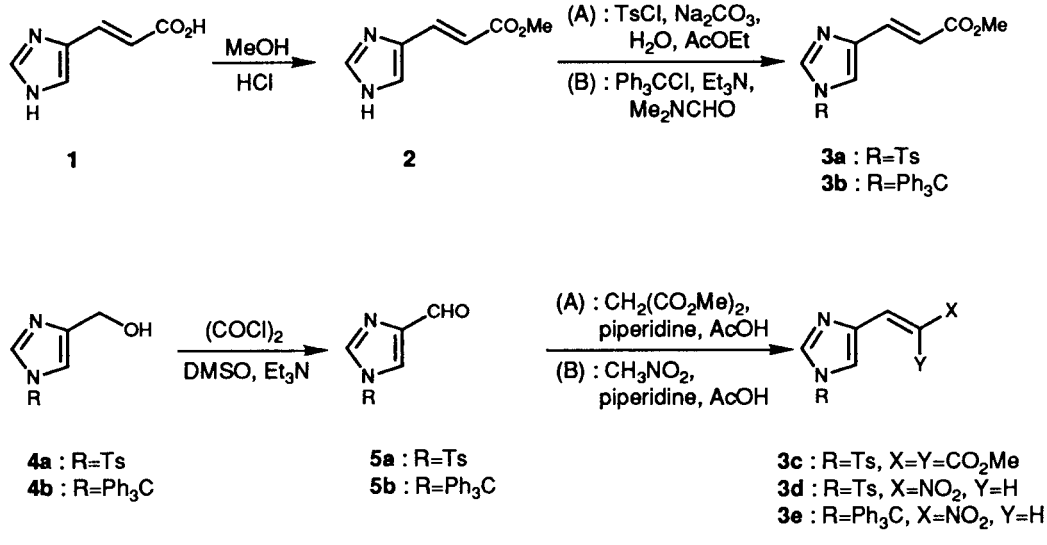
In view of the importance of naturally occurring imidazoles such as histamine and histidine in biological systems, it is not surprising to find that a large number of synthetic imidazoles have been prepared as potential pharmaceutical agents. During the course of our synthetic study on histamine agonists and antagonists [1], we were interested in a Diels-Alder reaction of 4-ethenyl-1*H*-imidazoles with some dienes. For this purpose we have prepared a series of the title compounds **2** and **3a-e** and examined their reactivities toward some dienes.

The compounds **2** and **3a,b** were easily obtained from (*E*)-3-(1*H*-imidazol-4-yl)propenoic acid (urocanic acid) (**1**) as illustrated in Scheme 1. The compounds **3c-e** were synthesized by Knoevenagel condensation of the 1*H*-imidazole-4-carboxaldehydes **5a,b**, which in turn were prepared conveniently by Swern oxidation of the corresponding alcohols **4a,b**, with dimethyl malonate and nitromethane.

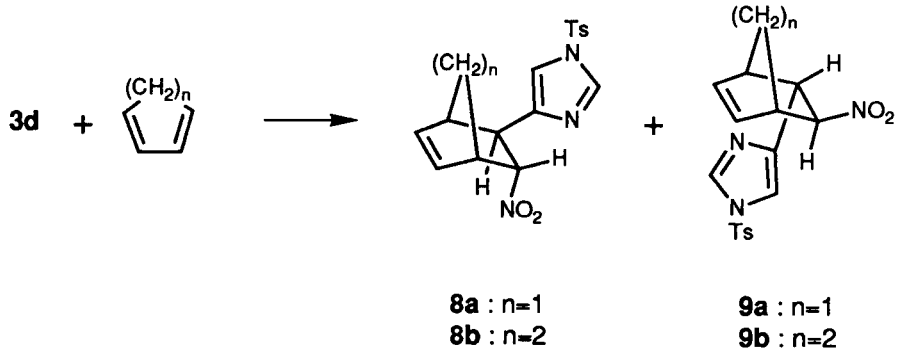
In general, the cycloaddition was carried out by heating a solution of **2** or **3** and a large excess of 2,3-dimethyl-1,3-butadiene in benzene in a sealed tube at 130-170° for several days. After evaporation of the solvent, the crude material was purified by silica gel chromatography. Compound **3b** (R = triphenylmethyl, X = CO_2Me, Y = H) gave an inseparable mixture of the cycloadduct **7b** and the starting material even after heating for 12 days. The stereochemistry of the cycloadducts **7a,d,e** was assigned by examining the coupling constants between the protons adjacent to the nitro (or ester) group and the imidazole ring. The observed large coupling constants (J = 10.6-10.8 Hz) clearly indicate that these cycloadducts **7** should be trans as expected. The structure of **2** was confirmed by conversion into **7a**. The results summarized in Table 1 indicate that (1) the nitroethenes **3d,e** are more reactive than the propenoates **3a,b**, and (2) introduction of the electron-withdrawing *p*-toluenesulfonyl group at the imidazole ring enhanced the reactivity.

We then examined the Diels-Alder reaction of the most reactive **3d** with some cyclic dienes. When a mixture of **3d** and cyclopentadiene was refluxed in toluene for 17 hours, two cycloadducts **8a** and **9a** were obtained in 91 and 7% yields, respectively. The structure and stereochemistry of the cycloadducts were determined by a comparison of their ^1H nmr spectra with those of the nitro-substituted bi-

Scheme 1



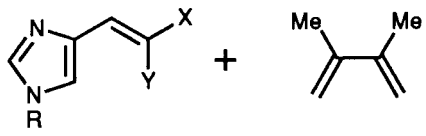
Scheme 2



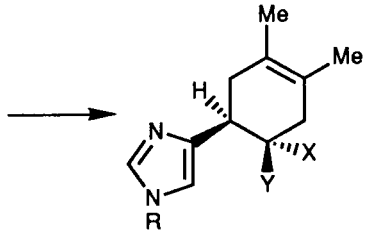
8a : n=1
8b : n=2

9a : n=1
9b : n=2

cyclo[2.2.1]heptenes [2,3]. The major adduct **8a** showed the signals for the α-protons to the nitro group and the imidazole ring at δ 5.24 (t, J = 3.9 Hz) and 3.28 (dd, J = 3.9 and 2.0 Hz), respectively, suggesting the nitro-endo and imidazole-exo structure. On the other hand, in the ^{1}H nmr spectrum of the minor adduct **9a** the corresponding signals appeared at δ 4.49 (dd, J = 4.1 and 1.5 Hz) and 3.82 (ddd, J = 4.1, 3.5, 1.0 Hz) in agreement with the nitro-exo and imidazole-endo structure. The reaction of **3d** with less reactive cyclohexa-1,3-diene was carried out in benzene in a sealed tube at 125-155° for 6 days to give the cycloadduct **8b** in 44% yield. The other isomer **9b** was obtained as an inseparable mixture with **8b** and could not be obtained in a pure state. The stereochemistry of **8b** was again assigned on the basis of the ^{1}H nmr spectroscopy. The observed coupling pattern of the α-proton to the nitro group [4] at δ 4.97 (dd, J = 5.1 and 2.4 Hz) was in good agreement with the reported values for the nitro-endo structure [2]. Since both the cycloadducts **8a** and **9a** were found to be stable in boiling toluene, they are assumed to be the kinetically controlled products. Thus, the stereochemistry of the cycloaddition is governed mainly by the nitro group, in accordance with the results of other workers [2,3].



2, 3a-e



6, 7a-e

Table 1

Diels-Alder Reaction of **2** and **3a-e** with 2,3-Dimethyl-1,3-butadiene

	Product R	X	Y	Conditions (°C/day)	Yield (%)
6	H	COOMe	H	140-170/6	16 [a]
7a	Ts	COOMe	H	140-170/6	58
7b	Ph_3C	COOMe	H	140-170/12	35 [b]
7c	Ts	COOMe	COOMe	140-170/6	86
7d	Ts	NO_2	H	125-155/1.5	92
7e	PH_3C	NO_2	H	125-155/6	63

[a] The unreacted starting material **2** (53%) was recovered. [b] Obtained as an inseparable mixture of the adduct **7b** and the starting material **3b**.

Unfortunately, the nitroethene **3d** gave no cycloadduct with 1,3-butadiene (3-sulfolene was used as the precursor) under the reaction conditions we employed (at 140-170° for 3 days): only decomposition of **3d** took place.

In summary, the Diels-Alder reaction of some 4-vinylimidazoles with dienes opens up a new route to some 4-substituted imidazoles which may serve as intermediates in the synthesis of histamine analogues.

EXPERIMENTAL

All melting points are uncorrected. The ir spectra were recorded with a JASCO-IRA-1 spectrophotometer. The ^{1}H nmr spectra were determined on a JEOL JNM-PMX 60 (60 MHz) or a Varian XL-300 (300 MHz) spectrometer using solutions in deuteriochlorofrom and tetramethylsilane as an internal standard.

Materials.

3-(1*H*-Imidazol-4-yl)propenoic acid (urocanic acid) (**1**) was obtained from the Aldrich Chemical Co., Inc. Methyl (*E*)-3-(1*H*-imidazol-4-yl)propenoate (**2**) [5], 1-(*p*-toluenesulfonyl)-1*H*-imidazole-4(or 5)-methanol (**4a**) [6] and 1-(triphenylmethyl)-1*H*-imidazole-4-methanol (**4b**) [7-11] were prepared according to the literatures.

Methyl (*E*)-3-[1-(*p*-Toluenesulfonyl)-1*H*-imidazol-4(or 5)-yl]propenoate (**3a**).

To a solution of **2** (1.00 g, 6.57 mmoles) and sodium carbonate (2.79 g, 26.3 mmoles) in water (13 ml) was added dropwise, over a 40 minute period, a solution of *p*-toluenesulfonyl chloride (1.5 g, 7.89 mmoles) in ethyl acetate (10 ml) with vigorous stirring at room temperature and the reaction mixture was stirred overnight. The organic layer was separated and the aqueous layer was further extracted with dichloromethane. The combined extracts were washed with saturated sodium bicarbonate solution, dried over magnesium sulfate, and concentrated to give **3a** (2.01 g, quantitative), which was essentially pure for the next step, mp 160-161° (2-propanol); ir (potassium bromide): 1720 cm^{-1}; ^1H nmr (60 MHz): δ 2.43 (s, CH_3, 3H), 3.73 (s, OMe, 3H), 6.56 (d, olefinic proton, 1H, J = 16 Hz), 7.23 (d, 2H, J = 8 Hz), 7.34 (brs, 1H), 7.40 (d, olefinic proton, 1H, J = 16 Hz), 7.77 (d, 2H, J = 8 Hz), 7.89 (brs, 1H).

Anal. Calcd. for $C_{14}H_{14}N_2O_4S$: C, 54.89; H, 4.61; N, 9.14. Found: C, 54.63; H, 4.52; N, 8.96.

Methyl (*E*)-3-(1-Triphenylmethyl-1*H*-imidazol-4-yl)propenoate (**3b**).

To a solution of **2** (761 mg, 5.00 mmoles) and triethylamine (1.59 ml, 11.4 mmoles) in dimethylformamide (5 ml) was added dropwise a solution of triphenylmethyl chloride (1.34 g, 4.80 mmoles) in dimethylformamide (10 ml) at room temperature. After stirring at room temperature for 30 minutes, the reaction mixture was poured into ice water (95 g). The precipitates were collected and dried to give **3b** (1.97 g, quantitative), which was essentially pure for the next step, mp 207-208° (hexane-ethyl acetate); ir (potassium bromide): 1705 cm^{-1}; ^1H nmr (60 MHz): δ 3.70 (s, OMe, 3H), 6.50 (d, olefinic proton, 1H, J = 16 Hz), 6.9-7.7 (m, 18H).

Anal. Calcd. for $C_{26}H_{22}N_2O_2$: C, 79.17; H, 5.62; N, 7.10. Found: C, 78.88; H, 5.65; N, 6.85.

1-(*p*-Toluenesulfonyl)-1*H*-imidazole-4(or 5)-carboxaldehyde (**5a**).

To a solution of an oxidizing reagent [prepared by adding a solution of dimethyl sulfoxide (2.0 ml, 28.6 mmoles) in dry dichloromethane (36 ml) to a solution of oxalyl chloride (1.2 ml, 13.7 mmoles) in dry dichloromethane (24 ml) at −70° under a nitrogen atmosphere] was added dropwise a solution of the alcohol **4a** (3.0 g, 11.9 mmoles) in dry dichloromethane (36 ml) at −70° and the reaction mixture was stirred for 20 minutes. Triethylamine (8.3 ml, 59.5 mmoles) was added to the mixture and the whole was allowed to warm up to room temperature. Water (50 ml) was added to the reaction mixture and the products were extracted with dichloromethane. The extract was washed with brine, dried over magnesium sulfate, and concentrated. The residue was chromatographed on silica gel under pressure (hexane-ethyl acetate, 4:1) to give the aldehyde **5a** (2.85 g, 96%), mp 101.5-102.5° (hexane-ethyl acetate) [lit [12] mp 98°].

1-(Triphenylmethyl)-1*H*-imidazole-4-carboxaldehyde (**5b**).

Using a similar procedure to that described above for the preparation of **5a**, the aldehyde **5b** (0.82 g, 82%) was obtained from the alcohol **4b** (1.00 g, 2.94 mmoles), mp 209-213° (chloroform-hexane) [lit [7,8] mp 197-199°].

4(or 5)-(*E*)-(2-Nitroethenyl)-1-(*p*-toluenesulfonyl)-1*H*-imidazole (**3d**).

To a solution of the aldehyde **5a** (2.00 g, 7.99 mmoles) in nitromethane (30 ml) was added piperidine (1 drop) and acetic acid (1 drop). The reaction mixture was stirred at room temperature overnight, then concentrated. The residue was chromatographed on silica gel under pressure (dichloromethane) to give **3d** (1.81 g, 77%), mp 157-158° (benzene) [lit [12] mp 157-158°].

Anal. Calcd. for $C_{12}H_{11}N_3O_4S$: C, 49.15; H, 3.75; N, 14.33. Found: C, 49.08; H, 3.68; N, 14.07.

4-(*E*)-(2-Nitroethenyl)-1-(triphenylmethyl)-1*H*-imidazole (**3e**).

Using a similar procedure to that described above for the preparation of **3d**, **3e** (747 mg, 91%) was obtained from the aldehyde **5b** (731 mg, 2.16 mmoles) and nitromethane (10 ml), mp 222-223° (hexane-ethyl acetate); ^1H nmr (60 MHz): δ 6.97-7.56 (m, 17H), 7.64 (d, olefinic proton, 1H, J = 16 Hz), 7.74 (s, 1H).

Anal. Calcd. for $C_{24}H_{19}N_3O_2$: C, 75.57; H, 5.02; N, 11.02. Found: C, 75.70; H, 5.06; N, 10.77.

Dimethyl 1-(*p*-Toluenesulfonyl)-1*H*-imidazol-4(or 5)-ylmethylenemalonate (**3c**).

To a solution of the aldehyde **5a** (300 mg, 1.20 mmoles) in benzene (15 ml) was added dimethyl malonate (321 mg, 2.40 mmoles), piperidine (1 drop) and acetic acid (1 drop), and the mixture was stirred at 40° for 3 hours. The solvent was evaporated off and the residue was chromatographed on silica gel (hexane-ethyl acetate, 2:1) to give **3c** (438 mg, quantitative), mp 138-140° (hexane-ethyl acetate); ir (potassium bromide): 1720, 1710 cm^{-1}; ^1H nmr (60 MHz): δ 2.43 (s, CH_3, 3H), 3.79, 3.86 (2 x s, 2 x OMe, 3H each), 7.34 (d, 2H, J = 8 Hz), 7.47 (s, 1H), 7.54 (brs, 1H), 7.81 (d, 2H, J = 8 Hz), 7.94 (brs, 1H).

Anal. Calcd. for $C_{16}H_{16}N_2O_6S$: C, 52.71; H, 4.39; N, 7.69. Found: C, 52.60; H, 4.38; N, 7.59.

Diels-Alder Reaction of **2** or **3** with 2,3-Dimethyl-1,3-butadiene. General Procedure.

A solution of **2** or **3** (1.70 mmoles), 2,3-dimethyl-1,3-butadiene (0.96 ml, 8.50 mmoles), and hydroquinone (10 mg) in dry benzene (25 ml) was heated in a sealed tube at 130-170° for several days. After removal of the solvent, the residue was chromatographed on silica gel (hexane-ethyl acetate, 4:1). The reaction conditions and yields are summarized in Table 1. The following compounds were prepared.

Methyl *trans*-4,5-Dimethyl-2-[1*H*-imidazol-4-yl]cyclohex-4-ene-1-carboxylate (**6**).

Compound **6** was an oil; ^1H nmr (60 MHz): δ 1.62 (s, 2 x CH_3, 6H), 2.08-2.43 (m, 4H), 2.50-3.32 (m, 2H), 3.48 (s, OCH_3, 3H), 6.73 (br s, 1H), 7.46 (br s, 1H), 7.57 (br s, NH, 1H). The structure of **6** was confirmed by conversion into **7a**, using a similar procedure to that described for the preparation of **3a**.

Methyl *trans*-4,5-Dimethyl-2-[1-(*p*-toluenesulfonyl)-1*H*-imidazol-4(or 5)-yl]cyclohex-4-ene-1-carboxylate (**7a**).

Compound **7a** had mp 108-110° (hexane-ethyl acetate); ir (potassium bromide): 1720 cm^{-1}; ^1H nmr (300 MHz): δ 1.61 (s, 2 x CH_3, 6H), 2.07-2.40 (m, 4H), 2.43 (s, CH_3, 3H), 2.80 (dt, H-2, 1H, J = 5.8, 10.6 Hz), 3.04 (dt, H-1, 1H, J = 5.8, 10.6 Hz), 3.38 (s, OCH_3, 3H), 7.01 (br s, 1H), 7.35 (d, 2H, J = 8.3 Hz), 7.79 (d, 2H, J = 8.3 Hz), 7.91 (d, 1H, J = 1.2 Hz).

Anal. Calcd. for $C_{17}H_{17}N_3O_4S$: C, 56.81; H, 4.77; N, 11.69. Found: C, 56.88; H, 4.76; N, 11.56.

Dimethyl 4,5-Dimethyl-2-[1-(*p*-toluenesulfonyl)-1*H*-imidazol-4(or 5)-yl]cyclohex-4-ene-1,1-dicarboxylate (**7c**).

This compound had mp 119-120° (hexane-ethyl acetate); ir (potassium bromide): 1730 cm^{-1}; ^1H nmr (60 MHz): δ 1.63 (s, 2 x CH_3, 6H), 2.46 (s, CH_3, 3H), 2.06-2.89 (m, 4H), 3.56, 3.63 (2 x s, 2 x OCH_3, 3H each), 3.06-4.00 (m, 1H), 7.06 (s, 1H), 7.33 (d, 2H, J = 8 Hz), 7.76 (d, 2H, J = 8 Hz), 7.83 (s, 1H).

Anal. Calcd. for $C_{22}H_{26}N_2O_6S$: C, 59.18; H, 5.87; N, 6.27. Found: C, 59.13; H, 5.89; N, 6.26.

trans-4(or 5)-(4,5-Dimethyl-2-nitrocyclohex-4-en-1-yl)-1-(*p*-toluenesulfonyl)-1*H*-imidazole (**7d**).

This compound had mp 133-135° (hexane-ethyl acetate); ^1H nmr (300 MHz): δ 1.63, 1.66 (2 x s, 2 x CH_3, 3H each), 2.20-2.32 (m, 1H), 2.39-2.57 (m, 2H), 2.44 (s, CH_3, 3H), 2.63-2.76 (m, 1H), 3.41 (dt, H-2, 1H, J = 6.1, 10.7 Hz), 4.92 (ddd, H-1, 1H, J = 10.8, 10.3, 5.8 Hz), 7.09 (d, 1H, J = 1.3 Hz), 7.36 (d, 2H, J = 8.5 Hz), 7.77 (d, 2H, J = 8.5 Hz), 7.90 (d, 1H, J = 1.3 Hz).

Anal. Calcd. for $C_{18}H_{21}N_3O_4S$: C, 57.59; H, 5.64; N, 11.19. Found: C, 57.42; H, 5.62; N, 10.96.

4-(4,5-Dimethyl-2-nitrocyclohex-4-en-1-yl)-1-(triphenylmethyl)-1*H*-imidazole (**7e**).

This compound had mp 190-193° (hexane-ethyl acetate); ^1H nmr (300 MHz): 1.63, 1.65 (2 x s, 2 x CH_3, 3H each), 2.23-2.37, 2.45-2.79 (m, 4H), 3.35 (dt, H-1, 1H, J = 5.9, 10.8 Hz), 4.95 (dt, H-2, 1H, J = 5.9, 10.8 Hz), 6.58 (d, 1H, J = 1.3 Hz), 7.05-7.16 (m, 6H), 7.29-7.42 (m, 9H), 7.35 (d, 1H, J = 1.3 Hz).

Anal. Calcd. for $C_{30}H_{29}N_3O_2$: C, 77.73; H, 6.31; N, 9.06. Found: C, 77.56; H, 6.16; N, 8.98.

2-*endo*-Nitro-3-*exo*-[1-(*p*-toluenesulfonyl)-1*H*-imidazol-4(or 5)-yl]-bicyclo[2.2.1]hept-5-ene (**8a**) and 2-*exo*-Nitro-3-*endo*-[1-(*p*-toluenesulfonyl)-1*H*-imidazol-4(or 5)-yl]bicyclo[2.2.1]hept-5-ene (**9a**).

A solution of **3d** (500 mg, 1.70 mmoles) and freshly distilled cyclopentadiene (1.0 ml) in toluene (20 ml) was refluxed for 17 hours. After removal of the solvent, the residue was chromatographed on silica gel (hexane-ethyl acetate, 6:1) to give **8a** (566 mg, 91%) and **9a** (44 mg, 7%).

Compound **8a** had mp 114-115° (hexane-ethyl acetate); ^1H nmr (300 MHz): δ 1.63 (qd, 1H, J = 2.0, 9.5 Hz), 2.00 (br d, 1H, J = 9.5 Hz), 2.45 (s, CH_3, 3H), 2.94-2.98 (br, 1H), 3.28 (dd, H-3, 1H, J = 3.9, 2.0 Hz), 3.56-3.62 (br, 1H), 5.24 (t, H-2, 1H, J = 3.9 Hz), 6.08 (dd, 1H, J = 5.9, 3.0 Hz), 6.52 (dd, 1H, J = 5.9, 3.2 Hz), 7.19 (dd, 1H, J = 1.5, 0.8 Hz), 7.39 (d, 2H, J = 8.4 Hz), 7.85 (d, 2H, J = 8.4 Hz), 7.97 (d, 1H, J = 8.4 Hz).

Anal. Calcd. for $C_{17}H_{17}N_3O_4S$: C, 56.81; H, 4.77; N, 11.69. Found: C, 56.88; H, 4.76; N, 11.56.

Compound **9a** had mp 116-117° (hexane-ethyl acetate); ^1H nmr (300 MHz): δ 1.77 (qd, 1H, J = 1.5, 9.3 Hz), 2.07 (br d, 1H, J = 9.3 Hz), 2.45 (s, CH_3, 3H), 3.21-3.26 (m, 1H), 3.43-3.47 (m, 1H), 3.82 (ddd, H-3, 1H, J = 4.1, 3.5, 1.0 Hz), 4.49 (dd, H-2, 1H, J = 4.1, 1.5 Hz), 6.16 (dd, 1H, J = 5.6, 2.7 Hz), 6.20 (dd, 1H, J = 5.6, 3.4 Hz), 7.02 (dd, 1H, J = 1.4, 1.0 Hz), 7.37 (d, 2H, J = 8.5 Hz), 7.81 (d, 2H, J = 8.5 Hz), 7.89 (d, 1H, J = 1.4 Hz).

Anal. Calcd. for $C_{17}H_{17}N_3O_4S$: C, 56.81; H, 4.77; N, 11.69. Found: C, 56.89; H, 4.79; N, 11.37.

2-*endo*-Nitro-3-*exo*-[1-(*p*-toluenesulfonyl)-1*H*-imidazol-4(or 5)-yl]-bicyclo[2.2.2]oct-5-ene (**8b**).

Using a procedure similar to that described for the preparation of **7**, a solution of **3d** (200 mg, 0.682 mmole) and cyclohexa-1,3-diene (0.32 ml, 3.41 mmoles) in benzene (7 ml) was heated in a sealed tube at 140-170° for 6 days. Column chromatography of silica gel (hexane-ethyl acetate, 6:1) of the crude material gave **8b** (113 mg, 44%) and a mixture of **8b** and **9b** (15 mg).

Compound **8b** had mp 141-143° (hexane-ethyl acetate); ^1H nmr (300 MHz): δ 0.98-1.10 (m, 1H), 1.43 (tt, 1H, J = 12.2, 3.7 Hz), 1.54-1.64 (m, 1H), 1.66-1.76 (m, 1H), 2.45 (s, CH_3, 3H), 2.75-2.81 (m, 1H), 3.35-3.42 (m, 2H), 4.97 (dd, H-2, 1H, J = 5.1, 2.4 Hz), 6.18 (dd, 1H, J = 6.3, 8.1 Hz), 6.53 (ddd, 1H, J = 8.1, 6.9, 1.1 Hz), 7.15 (dd, 1H, J = 1.3, 0.9 Hz), 7.38 (d, 2H, J = 8.5 Hz), 7.83 (d, 2H, J = 8.5 Hz), 7.94 (d, 1H, J = 1.3 Hz).

Anal. Calcd. for $C_{18}H_{19}N_3O_4S$: C, 57.90; H, 5.13; N, 11.25. Found: C, 57.74; H, 5.05; N, 11.17.

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REFERENCES AND NOTES

[1] P. G. Cooper, R. C. Young, G. J. Durant and C. R. Ganellin in Comprehensive Medicinal Chemistry, Vol **3**, C. Hansch, P. G. Sammes and J. B. Taylor eds, Pergamon Press, Oxford, 1990, pp 323-421.

[2] N. Ono, A. Kamimura and A. Kaji, *J. Org. Chem.*, **53**, 251 (1988).

[3] J. Bourguignon, G. Le Nard and G. Queguiner, *Can. J. Chem.*, **63**, 2354 (1985).

[4] The signal for the α-proton to the imidazole ring was masked by that of one of the bridgehead protons.

[5] W. Schenck, *Arch. Pharm. (Weinheim)*, **307**, 517 (1974).

[6] K. Matsumoto, T. Miyahara, M. Suzuki and M. Miyoshi, *Agric. Biol. Chem.*, **38**, 1097 (1974).

[7] M. Bernabé and A. Burger, *J. Med. Chem.*, **14**, 883 (1971).

[8] J. L. Kelly, C. A. Miller and E. W. McLean, *J. Med. Chem.*, **20**, 721 (1977).

[9] J. M. Kokosa, R. A. Szafasz and E. Tagupa, *J. Org. Chem.*, **48**, 3605 (1983).

[10] D. P. Davis, K. L. Kirk and L. A. Cohen, *J. Heterocyclic Chem.*, **19**, 253 (1982).

[11] K. L. Kirk, *J. Heterocyclic Chem.*, **22**, 57 (1985).

[12] A. M. Rozhkov, U. G. Kolmakova and V. P. Mamaev, *Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim. Nauk*, 144 (1972); *Chem. Abstr.*, **77**, 139895 (1972).