

N, 7.18. Isolated yield, 230 mg (33%) of **7e** as an oil. $^1\text{H-NMR}$: δ 1.69 (d, 4 H, $J = 6.8$, CH_3CH and H_{7a}); 2.21 (d, 1 H, $J_{7a-7b} = 9.3$, H_{7a}); 3.15 (s broad, 1 H, H_4); 3.28 (s broad, 1 H, H_1); 4.33 (d, 1 H, $J_{3x-4} = 2.7$, H_{3x}); 5.96-6.06 (m, 1 H, CHCH_3); 6.36 (dd, 1 H, $J_{6-5} = 7.6$, $J_{6-1} = 2.9$, H_6); 6.48 (dd, 1 H, $J_{5-6} = 7.6$, $J_{5-4} = 2.9$, H_5); 6.63 (m, 1 H, NH); 6.83-8.13 (m, 12 H, arom.). $^{13}\text{C-NMR}$: δ 20.6 (CH_3); 46.0 (C_4); 47.7 (C_7); 48.2 (C_1); 54.3 (C_3); 54.9 (C_2); 74.0 (CH); 120.4 (CN); 122.4; 122.9; 125.2; 125.9; 126.7; 127.3; 127.9; 128.6; 128.7; 128.9; 130.9; 134.0; 137.4; 139.6 (arom.); 137.3 (C_6); 138.1 (C_5); 166.5 (CO). Anal. Calcd for $\text{C}_{27}\text{H}_{24}\text{N}_2\text{O}$: C, 82.62; H, 6.16; N, 7.14. Found: C, 82.66; H, 6.15; N, 7.10.

Synthesis of Enantiomerically Pure (1R,2R,3S,4S,5S,6S)-(+)-Iodolactone. (10). In an inert atmosphere, TiCl_4 (3 mL, 3 mmol) was added to a solution of **3d** (1.14 g, 4 mmol) in CH_2Cl_2 (40 mL). The solution was stirred at room temperature for 1 h and then cooled to -40°C , and freshly distilled cyclopentadiene (1.32 g, 20 mmol) was added. After being stirred for 24 h, the solution was quenched by the addition of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and filtered and the filtrate evaporated in vacuo. The oily residue was saponified with 10% KOH/EtOH (150 mL) and refluxed for 4 h, and the EtOH was removed in vacuo. Water (50 mL) was added and extracted with Et_2O (3×10 mL). The aqueous layer was acidified with HCl (12 N) and extracted with Et_2O (3×10 mL). The organic solution was evaporated in vacuo to yield an oily mixture of exo and endo carboxylic cycloadducts. This residue was dissolved in MeOH (5 mL), and the pH was adjusted to 8 with 5% aqueous NaHCO_3 . It was then treated with an excess of iodine stock solution (5 g of I_2 , 10 g of KI , 30 mL of water) and allowed to stand for 1 h. The precipitate was collected by filtration, washed with 5% aqueous $\text{Na}_2\text{S}_2\text{O}_3$ solution (3×5 mL), and dried to afford 870 mg (77%) of **10**. The iodo-

lactone was successively recrystallized from $\text{MeOH}/\text{H}_2\text{O}$ until constant α . Mp: $194-6^\circ\text{C}$. $[\alpha]_D^{25}$ ($c = 1.00 \times 10^{-2}$ g/mL, CHCl_3): $+87.5 \pm 0.2^\circ$. $^1\text{H-NMR}$: δ 2.66-2.68 (m, 2 H, H_{7a} and H_{7b}); 3.21 (s, 1 H, H_4); 3.81 (s, 1 H, H_{3n}); 3.90 (d, 1 H, $J_{1-6} = 5.1$, H_1); 4.40 (d, 1 H, $J_{5-7a} = 1.9$, H_{5n}); 5.40 (d, 1 H, $J_{6-1} = 5.1$, H_{6a}); 7.31-7.42 (m, 5 H, arom.). $^{13}\text{C-NMR}$: δ 27.2 (C_5); 36.7 (C_7); 50.0 (C_2); 50.8; 52.5; 56.0 (C_1 , C_3 and C_4); 87.8 (C_6); 114.3 (CN); 127.7; 128.6; 129.1; 135.6 (arom.); 170.8 (CO). Anal. Calcd for $\text{C}_{15}\text{H}_{12}\text{INO}_2$: C, 49.34; H, 3.31; N, 3.84; I, 34.75. Found: C, 49.29; H, 3.24; N, 3.77; I, 34.61.

Synthesis of Enantiomerically Pure (1S,2S,3R,4R,5R,6R)-(-)-Iodolactone. (11). (-)-Iodolactone **11** was obtained in a similar way, starting from the (*E*)-2-cyanocinnamate of (*S*)-ethyl lactate **3c** (1.09 g, 4 mmol). Isolated yield, 983 mg (87%). $[\alpha]_D^{25}$ ($c = 1.00 \times 10^{-2}$ g/mL, CHCl_3): $-87.5 \pm 0.2^\circ$. Anal. Calcd for $\text{C}_{15}\text{H}_{12}\text{INO}_2$: C, 49.34; H, 3.31; N, 3.84; I, 34.75. Found: C, 49.26; H, 3.26; N, 3.71; I, 34.67.

Acknowledgment. This research was supported by the Dirección General de Investigación Científica y Técnica (project number PB88-0038).

Supplementary Material Available: X-ray crystallographic and ORTEP data for **11**, Tables S1-S4 containing a summary of crystal data, structure determination details, and atom positional and thermal parameters, a full list of bond lengths, bond and torsional angles, and interatomic contacts (15 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

Insertion of Nitrogen Oxide and Nitrosonium Ion into the Cyclopropane Ring: A New Route to 2-Isoxazolines and Its Mechanistic Studies

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The 9,10-dicyanoanthracene (DCA)-sensitized photoreaction of 1,2-diarylcyclopropanes **1a-d** in nitrogen oxide (NO)-saturated CH_3CN afforded 3,5-diaryl-2-isoxazolines **2a-d** in excellent yields. The reaction of **1a-d** with NOBF_4 or with a mixture of NO and O_2 in CH_3CN also afforded **2a-d** or **2a-b**. These reactions proceed via the attack of NO on the radical cation of **1**, which is formed by electron transfer from **1** to $^1\text{DCA}^*$ or NO^+ . The reaction of 1-alkyl-2-arylcyclopropanes with NOBF_4 afforded mixtures of 3-alkyl-5-aryl-2-isoxazolines and 4-alkyl-5-aryl-2-isoxazolines via the direct attack of NO^+ on the cyclopropane rings. The reaction of 1,1,2,2-tetraphenylcyclopropane with NOBF_4 afforded 2,3,5,5-tetraphenyl-2-isoxazolinium tetrafluoroborate via the migration of the phenyl group to nitrogen.

Introduction

Nitrogen oxide (NO) has a radical character and can be used as a radical trapping agent.¹ An elegant use of this property of NO in organic synthesis is the photolysis of alkyl nitrites, in which an unactivated C-H group is nitrated regioselectively. This reaction occurs via the photolytic cleavage of an alkyl nitrite to generate an alkoxyl radical and NO. Intramolecular hydrogen abstraction from the alkyl group by the alkoxyl radical in a 1,5-hy-

drogen shift fashion, followed by the attack of NO on the resulting carbon radical, produces 4-nitroso 1-ols. The reaction has been utilized for the selective introduction of functionality into a steroid skeleton.^{1b} However, no information is yet available about the reactivity of NO toward radical cation species generated from organic compounds.

Nitrosonium ion (NO^+), generated from nitrosonium salts such as NOBF_4 and NOPF_6 , acts as an electrophilic nitrosation reagent² and also as a strong one-electron ox-

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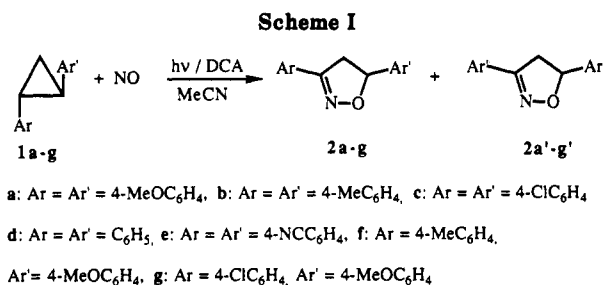
Table I. Insertion of NO into 1,2-Diarylcyclopropanes and 1-Alkyl-2-arylcyclopropanes

compd	$E_{1/2}^{ox}$ ^a (V)	$k_q^b \times 10^{10}$ (dm ³ mol ⁻¹ s ⁻¹)	method ^c	ΔG^d (kJ mol ⁻¹)	time (h)	product(s) ^e (%)	isomer ratio ^f
1a	0.55	1.83	A	-103.2	3.0	2a (91)	
			B	-33.7	0.5	2a (88)	
			C		0.1	2a (93)	
1b	0.90	1.55	A	-69.5	3.0	2b (55)	
			B	0	0.5	2b (80)	
			C		0.1	2b (70)	
1c	1.06	1.43	A	-54.0	3.0	2c (31)	
			B	15.4	0.5	2c (38)	
			C		0.1	0	
1d	1.14	1.41	A	-46.3	3.0	2d (17)	
			B	23.1	0.5	2d (26)	
			C		0.1	0	
1e	1.26	0.58	A	-34.7	6.0	0	
			B	34.7	1.0	0	
			C		0.1	0	
1f	0.82	1.80	A	-77.2	3.0	2f (45), 2f' (11)	8:2
			B	-7.7	0.5	2f (72), 2f' (18)	8:2
1g	0.82	1.70	A	-77.2	3.0	2g (24), 2g' (10)	7:3
			B	-7.7	0.5	2g (56), 2g' (24)	7:3
1h	0.97		A	-62.7	6.0	0	
			B	6.7	0.5	2h (41), 4h (27)	3:2
1i	1.35		A	-26.1	6.0	0	
			B	43.4	0.5	2i (34), 4i (23)	3:2
1j	0.95		A	-64.6	6.0	0	
			B	4.8	0.5	2j (7), 4j (34)	1:5

^a Oxidation potentials of cyclopropanes vs Ag/AgClO₄ in acetonitrile. ^b Rate constants for fluorescence quenching of DCA in aerated CH₃CN; [DCA] = 1 × 10⁻⁴ mol L⁻¹; τ (DCA, air) = τ (DCA, N₂) × I(DCA, air)/I(DCA, N₂) = 12.8 ns; τ (DCA, N₂) = 15.3 ns; see ref 22. ^c A: DCA-sensitized photoirradiation under NO. B: By use of NOBF₄. C: By use of NO and O₂. ^d The calculated free energy changes for the one-electron transfer process from cyclopropanes to ¹DCA* (method A) or NOBF₄ (method B). Reduction potentials are as follows: DCA -1.33 V, NOBF₄ -0.9 V. ^e Isolated yields. ^f Determined by 270-MHz ¹H NMR.

idizing reagent for organic compounds.^{3,4} Addition reactions of NO⁺ to alkenes⁵ and arylcyclopropanes^{4b,6} have been reported.

Previously, we have reported that the 9,10-dicyanoanthracene (DCA)-sensitized photooxygenation of 1,2-diarylcyclopropanes affords 3,5-diaryl-1,2-dioxolanes via their radical cations.⁷ This result implies that NO may attack the radical center of cyclopropane radical cations to form 2-isoxazolines. On the basis of this hypothesis, we have



studied reactions of NO and NO⁺ with arylcyclopropane derivatives. The following three reactions have been examined: (a) the photoreaction of arylcyclopropane derivatives with NO in the presence of DCA, (b) the reaction of arylcyclopropanes with NO⁺ (NOBF₄), and (c) the reaction of arylcyclopropanes with NO in the presence of molecular dioxygen. We have found that 1,2-diarylcyclopropanes can be converted to 2-isoxazoline derivatives by these methods. However, the reaction of 1,1,2,2-tetra-substituted cyclopropanes with NO⁺ afforded 2-isoxazolinium salts via the migration of a phenyl substituent onto nitrogen. In this paper, we report on the results obtained so far and on the mechanistic features of the reactions.

Results and Discussion

DCA-Sensitized Insertion of NO into a Cyclopropane Ring. Irradiation of an NO-saturated acetonitrile solution containing *trans*-1,2-bis(4-methoxyphenyl)cyclopropane (1a) (0.025 mol dm⁻³) and a catalytic amount of DCA (5 × 10⁻⁴ mol dm⁻³) with >400 nm light for 3 h gave 3,5-bis(4-methoxyphenyl)-2-isoxazoline (2a) in 91% isolated yield.^{8b} After the photoreaction, most of

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Table II. Substituent Effect on Insertion of NO into 1-(1-Naphthyl)-2-(4-substituted phenyl)cyclopropanes

compd	substituent	$E_{1/2}^{ox}$ (V)	σ^+	method ^b	2:2 ^c
1k	CH ₃ O	0.78	-0.778	A	85:15
				B	82:18
				C	80:20
1l	CH ₃	0.96	-0.311	A	44:56
				B	50:50
				C	50:50
1m	H	0.98	0.000	B	29:71
1n	Cl	0.99	0.114	B	23:77
1o	Br	1.00	0.150	B	22:78
1p	CN	1.04	0.659	A	d:>99
				B	d:>99
				C	d:d

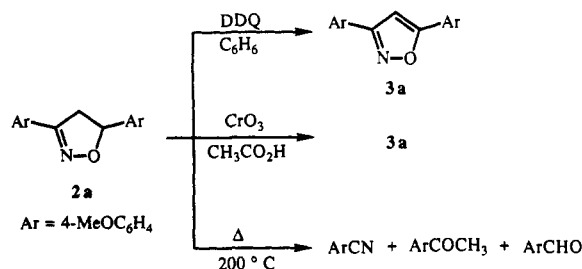
^a Oxidation potentials of cyclopropanes vs Ag/AgClO₄. ^b A: DCA-sensitized photoirradiation under NO. B: By use of NOBF₄. C: By use of NO and O₂. ^c Determined by 270-MHz ¹H NMR. ^d Not detected.

the DCA was recovered. The structure of **2a** was determined from its spectroscopic (¹H NMR, IR, MS) and analytical data and also from its chemical transformations. The pyrolysis of **2a** at 200 °C gave 4-methoxybenzoxazole, 4-methoxyacetophenone, and a small amount of 4-methoxybenzaldehyde.^{9a} The oxidation of **2a** with DDQ in benzene or with CrO₃ in acetic acid gave 3,5-bis(4-methoxyphenyl)isoxazole (**3a**) in nearly quantitative yield.

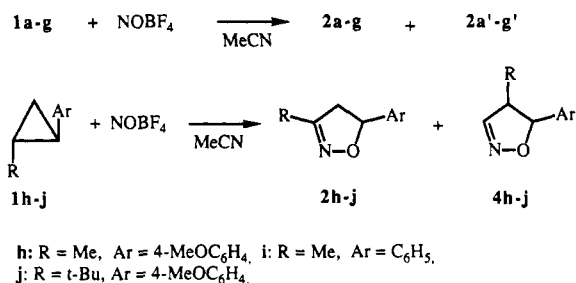
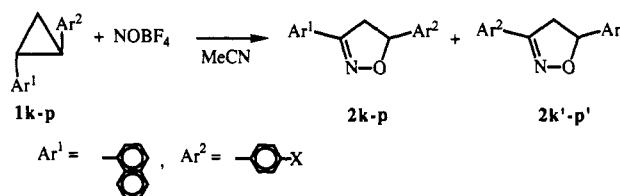
When triethylamine (0.01 mol dm⁻³) was added to the reaction system, the formation of **2a** was completely quenched. This photoinsertion of NO did not occur in less polar solvents such as benzene and dichloromethane.

Similar photoreaction of **1b-d** gave the corresponding 3,5-diaryl-2-isoxazolines **2b-d**. However, electron-poor cyclopropane **1e** did not undergo the photoinsertion of NO, and the starting material was recovered quantitatively. When unsymmetrically substituted 1,2-diarylcyclopropanes **1f-g** were used as substrates, positional isomers **2f-g** and **2f'-g'** were obtained. In these cases, formation of **2f-g** predominated. (Note that in **2f-g**, the oxygen atom is bonded to the carbon atom with the more electron-donating aromatic substituent.) However, for 1-alkyl-2-arylcyclopropanes **1h-j**, the photoinsertion of NO did not occur, even on prolonged irradiation. These results are summarized in Table I.

Insertion of NO into a Cyclopropane Ring by the Use of NOBF₄. The reaction of **1a** with 1.1–1.2 equiv of NOBF₄ in acetonitrile at room temperature under argon atmosphere gave **2a** in 88% yield. Similarly, the reaction



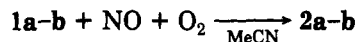
of **1b-d** with NOBF₄ gave **2b-d**.^{8a} The yields of 2-isoxazolines decreased when the oxidation potentials of the cyclopropanes increased. No reaction occurred with **1e**. Unsymmetrically substituted cyclopropanes **1f-g** gave

Scheme II**Scheme III**

mixtures of **2f-g** and **2f'-g'**, essentially in the same ratios as those obtained from the DCA-sensitized NO insertion. These results are given in Table I.

The reaction of 1-alkyl-2-arylcyclopropanes (**1h-j**) with NOBF₄ in a similar manner gave mixtures of two isomeric isoxazolines, 3- and 4-alkyl-5-aryl-2-isoxazolines (**2h-j** and **4h-j**), which were not obtained from the photoreaction. These results are also given in Table I.

Insertion of NO into the Cyclopropane Ring by the Use of NO and O₂. Nitrogen oxide (60 cm³ min⁻¹) and air (300 cm³ min⁻¹) were bubbled for 6 min into a room-temperature acetonitrile solution containing **1a**. The



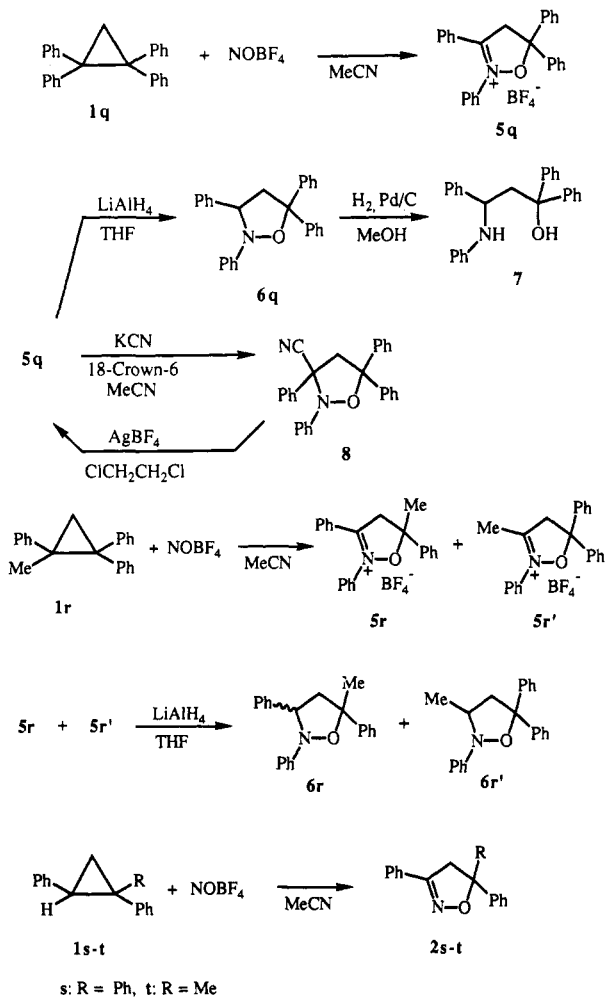
reaction mixture was extracted with benzene–water. From the organic layer, **2a** was obtained in 93% isolated yield. Similar treatment of **1b** gave **2b**. In this reaction also, the yields of 2-isoxazolines decreased when the oxidation potentials of the cyclopropanes increased. These results are also given in Table I.

Regioselectivity in the Formation of 2-Isxazolines. The regioselectivity of the NO insertion into unsymmetrically substituted cyclopropanes **1f-g** by the DCA-sensitized photoreaction and the reaction with NOBF₄ was essentially identical, and both the reactions gave the isomeric isoxazolines **2f-g** and **2f'-g'** in essentially the same ratios. To elucidate the mechanistic basis for the regioselectivity in the NO-insertion reaction, we studied the effect of different phenyl substituents on the NO-insertion reaction of 1-(1-naphthyl)-2-(4-substituted phenyl)cyclopropanes **1k-p** by employing three methods: the DCA-sensitized photoreaction, the reaction with NOBF₄, and the reaction with the NO–O₂ system. All of these reactions gave mixtures of 3-(1-naphthyl)-5-(4-substituted phenyl)-2-isoxazolines **2k-p** and 5-(1-naphthyl)-3-(4-substituted phenyl)-2-isoxazolines **2k'-p'**. The ratios of **2k-p** to **2k'-p'** were determined by ¹H NMR analysis of the reaction mixtures. These compounds were especially suitable for NMR analysis because the introduction of a naphthyl group caused a strong downfield shift of the proton signals on C₅ of **2k'-p'** and prevented overlapping of those signals with all of the proton signals of **2k-p**. The ratios of **2k-p** to **2k'-p'** increased when the electron-donating ability of the substituents increased. Furthermore, the product ratios obtained from the three types of reac-

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Scheme IV



tions for each substituent were in good agreement with each other, within experimental error. The results are summarized in Table II. A plot of $\log(2k-p/2k'-p')$ against the substituent constants (σ^+ values) exhibited a good linear relationship ($r = 0.999$).¹⁰ This strongly suggests that the three reactions have a common intermediate.

Reaction of 1,1,2,2-Tetrasubstituted Cyclopropanes with NOBF_4 . The reaction of 1,1,2,2-tetraphenylcyclopropane (1q) with NOBF_4 gave 2,3,5,5-tetraphenyl-2-isoxazolium tetrafluoroborate (5q) in 89% yield.^{8c} The structure of 5q was determined from its spectroscopic properties (^1H NMR, ^{13}C NMR, IR, MS) and elemental analysis and also from its chemical conversions. The reduction of 5q with LiAlH_4 in THF gave 2,3,5,5-tetraphenylisoxazolidine (6q),^{11,12} which was further reduced by catalytic hydrogenation on Pd-C to give 1,1,3-triphenyl-3-(*N*-anilino)-1-propanol (7). The reaction of 5q with KCN in acetonitrile in the presence of a catalytic amount of 18-crown-6 afforded 3-cyano-2,3,5,5-tetraphenylisoxazolidine (8) in 79% yield. Treatment of 8 with AgBF_4 in 1,2-dichloroethane gave 5q in quantitative yield. When 1-methyl-1,2,2-triphenylcyclopropane (1r) was treated with NOBF_4 in a similar manner, two isomeric

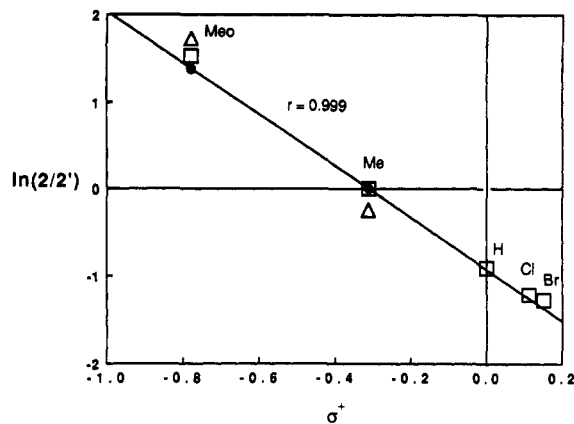
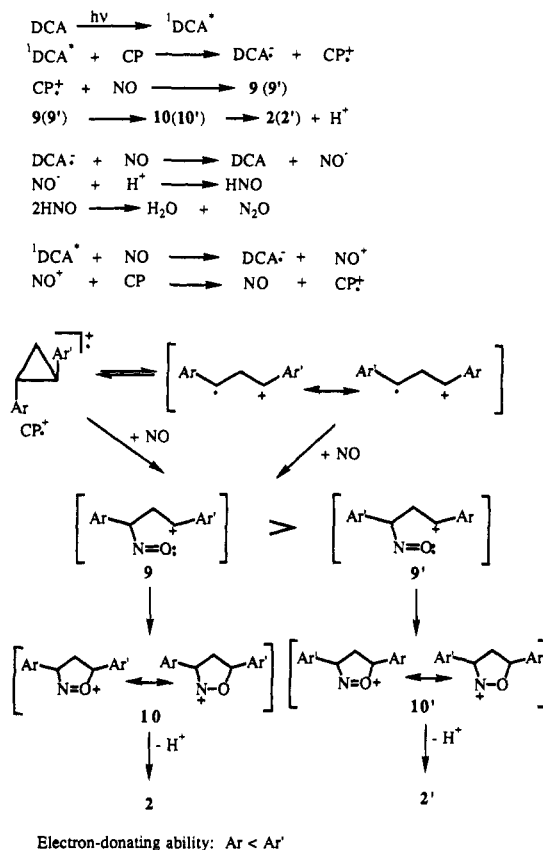


Figure 1. Plots of $\ln(2/2')$ vs Hammett σ^+ values of the phenyl substituents. $2/2'$: the product ratios for $2k-o/2k'-o'$ in the insertion of NO into cyclopropane ring of 1k-o. Key: (Δ) NO-DCA-*h* ν system, (\square) NOBF_4 system, (\bullet) NO- O_2 system.

Scheme V



2-isoxazolium salts 5r and 5r' were obtained in a 5:3 ratio in quantitative yield. They gave a mixture of two isomeric isoxazolidine derivatives 6r and 6r' upon treatment with LiAlH_4 . However, the reactions of 1,1,2-triphenylcyclopropane (1s) and 1-methyl-1,2-diphenylcyclopropane (1t) with NOBF_4 gave the 2-isoxazolines 2s-t in 72% and 84% yields, respectively.

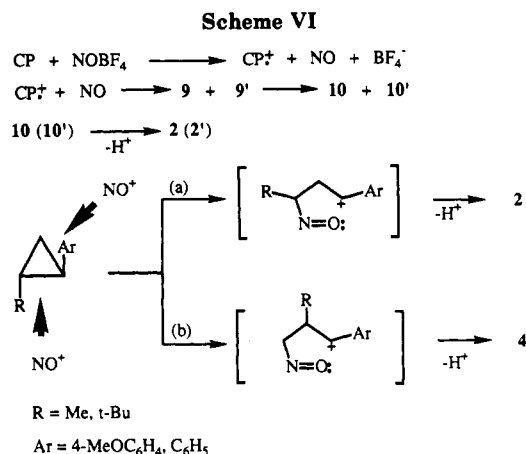
Mechanisms. The DCA-Sensitized NO Insertion. The fluorescence of DCA in acetonitrile was quenched by 1,2-diarylcyclopropanes 1a-d at a nearly diffusion-controlled rate. The calculated free energy changes (ΔG) for a one-electron transfer process from 1,2-diarylcyclopropanes to singlet excited $^1\text{DCA}^*$ in acetonitrile¹³ are negative (see Table I). On the basis of these results, we

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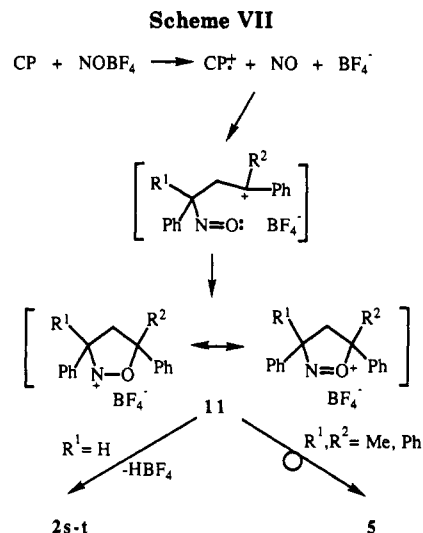


propose the electron-transfer mechanism outlined in Scheme V for the photochemical insertion of NO into 1,2-diarylcyclopropanes. One-electron transfer from 1,2-diarylcyclopropane (CP) to ¹DCA* generates radical ion CP^{•+} and DCA⁻. Radical cation CP^{•+} reacts with NO to give 1,3-diaryl-1-nitrosopropyl cation 9. The intramolecular ring closure of this cation gives isoxazolidinium cation 10, which, upon deprotonation, produces 2-isoxazoline 2. For unsymmetrically substituted cyclopropanes, two cations 9 and 9' could be formed. The regioselectivity in the NO insertion can be accounted for by the relative stability of these cation intermediates. Intermediate 9 would be stabilized by an electron-donating aromatic substituent much more effectively than 9' would. The linear relationship shown in Figure 1 supports this interpretation.

The quantitative recovery of DCA indicates that the oxidation of DCA⁻ to DCA is involved in the photoreaction. This oxidation may be accomplished by an NO molecule: the one-electron reduction of NO followed by protonation gives HNO, which disproportionates to H₂O and N₂O.¹⁴ The reaction mixture did indeed become acidic after irradiation.

The fluorescence of DCA was efficiently quenched by NO in acetonitrile. The oxidation potential of NO in acetonitrile, E_{1/2}^{ox} = 0.9 V vs Ag/Ag⁺, indicates that the electron transfer from NO to ¹DCA* is an exothermic process. If NO⁺ is formed via this process, an electron transfer from cyclopropanes to NO⁺ or a direct attack of NO⁺ on the cyclopropane rings may occur to give 2-isoxazolines. However, in the DCA-sensitized NO insertion, participation of NO⁺ is unlikely because the DCA-sensitized NO insertion of 1-alkyl-2-aryl-cyclopropanes 1h-j does not afford 2-isoxazolines, whereas the reaction of 1h-j with NOBF₄ does give 2-isoxazolines (see below).

NO Insertion with NOBF₄. The proposed mechanism for the reaction of 1,2-diarylcyclopropanes with NOBF₄ is shown in Scheme VI. The first step is a one-electron transfer from 1,2-diarylcyclopropane (CP) to NOBF₄ to produce radical cation CP^{•+} and a neutral NO molecule. This process was rationalized from the following results. The yields of 2-isoxazolines decreased when the oxidation potentials of the cyclopropanes increased. In the case of 1a, a catalytic amount of NOBF₄ caused a rapid *cis*-*trans* isomerization, giving an equilibrium mixture containing *cis*-1a and *trans*-1a in a 5:95 ratio.¹⁵ The same equilibrium mixture was obtained from the DCA-sensitized photoisomerization of both *cis*-1a and *trans*-1a in acetonitrile, which proceeds via the radical cation of 1a.¹⁶ The gen-



eration of NO was recognized by the evolution of NO₂ when air was introduced into the reaction mixture.

The second step of the mechanism in Scheme VI is the attack of NO on the radical cation CP^{•+}. This step is reasonable because the ratios of 2k-p to 2k'-p' obtained from the reaction of 1k-p with NOBF₄ were essentially identical to those obtained from the DCA-sensitized NO insertions of 1k-p.

It is noteworthy that, although the DCA-sensitized photoreaction of 1-alkyl-2-aryl-cyclopropanes 1h-j with NO failed to give the NO-insertion products, the same cyclopropanes gave 2-isoxazolines 2h-j and 4h-j when treated with NOBF₄ in acetonitrile. It is likely that this reaction occurs via the direct attack of NO⁺ on the cyclopropane ring as shown in Scheme VI. This mechanism was supported by the fact that the ratios of 2h-j to 4h-j depended on the bulkiness of alkyl substituents. In fact, 4j was the predominant product in the reaction of 1j with NOBF₄. A reasonable explanation for the regioselectivity is that the attack of NO⁺ on the carbon-carbon bond of the cyclopropane ring of 1j occurs from the less-hindered side to give 4j (path b in Scheme VI). It is possible that, in the cases of less electron-rich 1,2-diarylcyclopropanes 1c-d, the direct attack of NO⁺ on the cyclopropane rings competes with the electron-transfer process.

Formation of 2-Isoxazolinium Salts. In the cases of 1,1,2,2-tetrasubstituted cyclopropanes 1q-r, 1-isoxazolinium or 2-isoxazolidinium intermediates 11, which are formed in the reaction with NOBF₄, do not have a hydrogen at C₃. Therefore, the phenyl group at that position migrates to the cationic nitrogen to give 2-phenyl-2-isoxazolinium salts 5. On the other hand, when hydrogen is present at C₃, as in the cases of 1s-t, deprotonation of 11 produces 2 (Scheme VII).

The NO Insertion by the Use of NO and O₂. In the presence of oxygen, NO is oxidized to NO₂, which is further converted into N₂O₃ and N₂O₄. These nitrogen oxides can be a NO⁺ source.¹⁷ Therefore, it is reasonable to suppose that NO⁺ is a reactive intermediate in the insertion of NO into 1,2-diarylcyclopropanes by the NO-O₂ reagent. The participation of NO⁺ is supported by the fact that the regioselectivity of the NO insertion into unsymmetrically

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substituted cyclopropanes **1k-l** with NOBF_4 is essentially identical to that observed for the reaction with the NO-O_2 system (Table II, Figure 1).

Experimental Section¹⁸

General Procedure for the DCA-Sensitized Photoreaction of Cyclopropane Derivatives under NO Atmosphere. An acetonitrile solution (8 mL) containing a cyclopropane derivative (0.2 mmol) and DCA (0.01 mmol) in a Pyrex tube (i.d. 10 mm) equipped with a two-way stopcock was deaerated by bubbling nitrogen into the solution for more than 15 min. A stream of NO was then introduced into the solution until the solution was saturated. This solution was irradiated for 3 h with >400 nm light from a 500-W high-pressure mercury lamp through a $\text{CuSO}_4\text{-NH}_3$ filter solution. After irradiation, nitrogen was bubbled through the solution for 10 min by means of a capillary to remove excess NO. The solvent was evaporated, and the residue was chromatographed on silica gel with benzene to give 3,5-diaryl-2-isoxazoline. For unsymmetrically substituted cyclopropanes, the ratios of **2k-p** to **2k'-p'** were determined by integration of the ^1H NMR signals of the protons on C_5 of **2k-p** and **2k'-p'**. No reaction occurred for 1-alkyl-2-arylcyclopropanes **1h-j**, and the starting materials were recovered quantitatively.

Pyrolysis of 2a. Compound **2a** (28 mg, 0.1 mmol) was placed in an NMR tube (i.d. 3 mm) and heated at 225 ± 5 °C for 10 min in an oil bath. The reaction mixture was dissolved in CDCl_3 and analyzed by ^1H NMR.

Oxidation of 2a with CrO_3 .^{9a} Compound **2a** (230 mg, 0.81 mmol) in acetic acid (10 mL) was added by portions to a stirred solution of CrO_3 (1 mol dm^{-3} , 2 mL) in acetic acid. The reaction mixture was heated at 80 °C for 15 min, cooled, diluted with water (20 mL), and then extracted with benzene (20 mL). The organic layer was evaporated under reduced pressure. Recrystallization of the residue from methanol gave **3a** (141 mg, 62%).

Oxidation of 2a with DDQ. A solution of **2a** (230 mg, 0.81 mmol) and 2 equiv of DDQ (367 mg) in benzene (10 mL) was refluxed for 6 h. The solvent was removed under reduced pressure. The residue was chromatographed on silica gel with benzene and then purified by recrystallization from methanol to give **3a** (225 mg, 99%).

3,5-Bis(4-methoxyphenyl)isoxazole (3a): mp 176–177 °C; ^1H NMR (270 MHz, CDCl_3) δ 3.86 (s, 6 H), 6.65 (s, 1 H), 7.39 (ABq, 4 H, $\Delta\nu = 230$ Hz, $J = 9.2$ Hz), 7.40 (ABq, 4 H, $\Delta\nu = 224$ Hz, $J = 9.2$ Hz); IR (KBr) 2844, 1613, 1516, 1437, 1303, 1251, 1178, 1029, 835 cm^{-1} ; MS (70 eV) m/z 281 (M^+). Anal. Calcd for $\text{C}_{17}\text{H}_{15}\text{O}_3\text{N}$: C, 72.58; H, 5.37; N, 4.98%. Found: C, 72.64; H, 5.29; N, 5.06%.

General Procedure for the Reaction of Cyclopropane Derivatives with NOBF_4 . Argon was bubbled through an acetonitrile solution (8 mL) containing a cyclopropane derivative (0.2 mmol) for 10 min with vigorous stirring. NOBF_4 (1.1–1.2 equiv) was added to the solution. The resulting mixture was stirred for 30 min at room temperature, diluted with water, and then extracted with benzene (20 mL). The organic layer was dried over Na_2SO_4 and evaporated. Recrystallization of the residue gave a 2-isoxazoline derivative. For unsymmetrically substituted cyclopropanes, the ratios of **2k-p** to **2k'-p'** were determined by integration of the ^1H NMR signals of the protons on C_5 of **2k-p** and **2k'-p'**. For 1-alkyl-2-arylcyclopropanes **1h-j**, mixtures of **2h-j** and **4h-j** were obtained. They were separated by column chromatography on silica gel.

General Procedure for the Reaction of Cyclopropane Derivatives with NO and O_2 . Nitrogen oxide (60 mL min^{-1}) and air (300 mL min^{-1}) were bubbled through an acetonitrile solution (8 cm^3) containing a cyclopropane derivative (0.2 mmol) for 6 min. The reaction mixture was then diluted with water and extracted with benzene. The organic layer was dried over Na_2SO_4 and evaporated to give a 2-isoxazoline derivative.

Physical Properties of 2-Isoxazolines. **3,5-Bis(4-methoxyphenyl)-2-isoxazoline (2a):** mp 141–142 °C; ^1H NMR (270 MHz, CDCl_3) δ 3.30 (dd, 1 H, $J = 8.6$ and 17.2 Hz), 3.71 (dd, 1 H, $J = 10.9$ and 17.2 Hz), 3.81 (s, 3 H), 3.84 (s, 3 H), 5.66 (dd, 1 H), 7.11 (ABq, 4 H, $\Delta\nu = 109.7$ Hz, $J = 8.5$ Hz), 7.28 (ABq, 4 H, $\Delta\nu = 195.0$ Hz, $J = 9.3$ Hz); IR (KBr) 1580, 1230, 1170, 1010,

890, 820 cm^{-1} ; MS (70 eV) m/z 283 (M^+). Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{O}_3\text{N}$: C, 72.06; H, 6.05; N, 4.94. Found: C, 72.13; H, 6.13; N, 4.96.

3,5-Bis(4-methylphenyl)-2-isoxazoline (2b): mp 106–107 °C; ^1H NMR (270 MHz, CDCl_3) δ 2.35 (s, 3 H), 2.38 (s, 3 H), 3.31 (dd, 1 H, $J = 8.2$ and 16.8 Hz), 3.73 (dd, 1 H, $J = 10.2$ and 16.8 Hz), 5.68 (dd, 1 H), 7.23 (ABq, 4 H, $\Delta\nu = 20.5$ Hz, $J = 8.5$ Hz), 7.41 (ABq, 4 H, $\Delta\nu = 92.0$ Hz, $J = 8.5$ Hz); MS (70 eV) m/z 251 (M^+). Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{ON}$: C, 81.24; H, 6.82; N, 5.57. Found: C, 81.36; H, 6.80; N, 5.65.

3,5-Bis(4-chlorophenyl)-2-isoxazoline (2c): mp 96–97 °C; ^1H NMR (270 MHz, CDCl_3) δ 3.27 (dd, 1 H, $J = 8.2$ and 16.8 Hz), 3.76 (dd, 1 H, $J = 11.0$ and 16.8 Hz), 5.73 (dd, 1 H), 7.34 (ABq, 4 H, $\Delta\nu = 13.0$ Hz, $J = 10.4$ Hz), 7.50 (ABq, 4 H, $\Delta\nu = 68.2$ Hz, $J = 8.5$ Hz); IR (KBr) 1590, 1490, 1220, 880, 800 cm^{-1} ; MS (70 eV) m/z 295, 293, 291 (M^+). Anal. Calcd for $\text{C}_{15}\text{H}_{11}\text{ONCl}_2$: C, 61.66; H, 3.79; N, 4.79. Found: C, 61.52; H, 3.66; N, 4.86.

3,5-Diphenyl-2-isoxazoline (2d): mp 73–74 °C (lit.¹⁹ 74–75 °C).

3-(4-Methylphenyl)-5-(4-methoxyphenyl)-2-isoxazoline (2f): mp 121.5–123 °C; ^1H NMR (270 MHz, CDCl_3) δ 2.38 (s, 3 H), 3.31 (dd, 1 H, $J = 8.5$ and 16.6 Hz), 3.72 (dd, 1 H, $J = 10.6$ and 16.6 Hz), 3.81 (s, 3 H), 5.66 (dd, 1 H), 7.12 (ABq, 4 H, $\Delta\nu = 115.0$ Hz, $J = 9.2$ Hz), 7.40 (ABq, 4 H, $\Delta\nu = 101.2$ Hz, $J = 8.0$ Hz); IR (KBr) 2940, 1615, 1518, 1253, 1181, 1033, 905, 825, 538 cm^{-1} ; MS (70 eV) m/z 267 (M^+). Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{O}_2\text{N}$: C, 76.38; H, 6.41; N, 5.24. Found: C, 76.24; H, 6.45; N, 5.25.

3-(4-Methoxyphenyl)-5-(4-methylphenyl)-2-isoxazoline (2f'): ^1H NMR (270 MHz, CDCl_3) δ 2.34 (s, 3 H), 3.30 (dd, 1 H, $J = 8.5$ and 16.6 Hz), 3.73 (dd, 1 H, $J = 10.8$ and 16.6 Hz), 3.83 (s, 3 H), 5.66 (dd, 1 H), aromatic protons could not be assigned because the signals overlapped with those of the isomer.

3-(4-Chlorophenyl)-5-(4-methoxyphenyl)-2-isoxazoline (2g): mp 157.5–158.5 °C; ^1H NMR (270 MHz, CDCl_3) δ 3.30 (dd, 1 H, $J = 8.6$ and 16.7 Hz), 3.70 (dd, 1 H, $J = 10.9$ and 16.7 Hz), 3.81 (s, 3 H), 5.70 (dd, 1 H), 7.11 (ABq, 4 H, $\Delta\nu = 108.5$ Hz, $J = 8.6$ Hz), 7.51 (ABq, 4 H, $\Delta\nu = 64.7$ Hz, $J = 8.9$ Hz); IR (KBr) 2938, 1613, 1518, 1493, 1257, 1181, 1033, 907, 832 cm^{-1} ; MS (70 eV) m/z 289, 287 (M^+). Anal. Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2\text{NCl}$: C, 66.78; H, 4.90; N, 4.86. Found: C, 66.33; H, 4.71; N, 4.76.

3-(4-Methoxyphenyl)-5-(4-chlorophenyl)-2-isoxazoline (2g'): ^1H NMR (270 MHz, CDCl_3) δ 3.38 (dd, 1 H, $J = 8.8$ and 17.0 Hz), 3.73 (dd, 1 H, $J = 10.7$ and 17.0 Hz), 3.83 (s, 3 H), 5.74 (dd, 1 H), aromatic protons could not be assigned because the signals overlapped with those of the isomer.

3-Methyl-5-(4-methoxyphenyl)-2-isoxazoline (2h): mp 72.0–72.8 °C; ^1H NMR (270 MHz, CDCl_3) δ 2.02 (s, 3 H), 2.88 (dd, 1 H, $J = 8.7$ and 17.3 Hz), 3.30 (dd, 1 H, $J = 10.7$ and 17.3 Hz), 3.80 (s, 3 H), 5.50 (dd, 1 H), 7.07 (ABq, 4 H, $\Delta\nu = 102.6$ Hz, $J = 8.9$ Hz); ^{13}C NMR δ 13.2, 46.7, 55.3, 81.4, 114.1, 127.2, 133.1, 154.9, 159.5; IR (KBr) 1600, 1508, 1430, 1250, 1010, 810 cm^{-1} . Anal. Calcd for $\text{C}_{11}\text{H}_{13}\text{O}_2\text{N}$: C, 69.09; H, 6.85; N, 7.33. Found: C, 68.86; H, 7.03; N, 7.29.

4-Methyl-5-(4-methoxyphenyl)-2-isoxazoline (4h): ^1H NMR (270 MHz, CDCl_3) δ 1.28 (d, 3 H, $J = 7.3$ Hz), 3.15–3.36 (m, 1 H), 3.78 (s, 3 H), 4.91 (d, 1 H, $J = 8.5$ Hz), 6.95 (m, 4 H), 7.09 (d, 1 H, $J = 1.6$ Hz).

3-Methyl-5-phenyl-2-isoxazoline (2i):⁶ ^1H NMR (60 MHz, CDCl_3) δ 2.03 (s, 3 H), 2.84–3.30 (m, 2 H), 5.47 (dd, 1 H, $J = 8$ and 12 Hz), 7.35 (s, 5 H).

4-Methyl-5-phenyl-2-isoxazoline (4i):⁶ ^1H NMR (60 MHz, CDCl_3) δ 1.30 (d, 3 H, $J = 8$ Hz), 2.84–3.50 (m, 1 H), 4.91 (d, 1 H, $J = 8$ Hz), 7.35 (m, 6 H); MS (70 eV) m/z 161 (M^+).

3-tert-Butyl-5-(4-methoxyphenyl)-2-isoxazoline (2j): ^1H NMR (270 MHz, CDCl_3) δ 1.56 (s, 9 H), 2.89 (dd, 1 H, $J = 8.5$ and 17.1 Hz), 3.31 (dd, 1 H, $J = 10.5$ and 17.1 Hz), 3.81 (s, 3 H), 5.51 (dd, 1 H), 7.26 (ABq, 4 H, $\Delta\nu = 101.1$ Hz, $J = 8.7$ Hz); ^{13}C NMR δ 55.4, 81.5, 114.1, 127.2, 133.2, 164.0, 165.4.

4-tert-Butyl-5-(4-methoxyphenyl)-2-isoxazoline (4j): ^1H NMR (270 MHz, CDCl_3) δ 3.00 (s, 9 H), 3.01 (dd, 1 H, $J = 1.8$ and 6.0 Hz), 3.79 (s, 3 H), 5.21 (d, 1 H, $J = 6.0$ Hz), 7.03 (ABq, 4 H, $\Delta\nu = 67.5$ Hz, $J = 9.2$ Hz), 7.11 (d, 1 H, $J = 1.8$ Hz); ^{13}C NMR δ 27.5 (q), 33.0 (s), 55.3 (q), 68.9 (d), 81.8 (d), 114.1 (d), 126.9 (d),

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134.3 (s), 147.1 (s), 154.4 (s); IR (KBr) 2964, 1613, 1516, 1249, 1178, 1035, 824 cm⁻¹; MS (70 eV) *m/z* 161 (M⁺).

3-(1-Naphthyl)-5-(4-methoxyphenyl)-2-isoxazoline (2k): ¹H NMR (270 MHz, CDCl₃) δ 3.53 (dd, 1 H, *J* = 8.5 and 16.5 Hz), 3.81 (s, 3 H), 3.93 (dd, 1 H, *J* = 10.9 and 16.5 Hz), 5.72 (dd, 1 H), 7.15 (ABq, 4 H, Δ*ν* = 124.6 Hz, *J* = 8.9 Hz), 7.44–7.65 (m, 4 H), 7.87–7.91 (m, 2 H), 9.07 (d, 1 H, *J* = 8.5 Hz); ¹³C NMR δ 45.9, 55.4, 81.4, 114.2, 124.8, 126.5, 126.7, 127.2, 127.4, 127.6, 127.7, 128.6, 130.7, 130.9, 132.9, 134.1, 156.9, 159.7; IR (KBr) 3050, 1613, 1516, 1249, 1178, 1033, 901, 830, 803, 775 cm⁻¹; MS (70 eV) *m/z* 303 (M⁺).

3-(4-Methoxyphenyl)-5-(1-naphthyl)-2-isoxazoline (2k′): ¹H NMR (270 MHz, CDCl₃) δ 3.28 (dd, 1 H, *J* = 8.1 and 16.5 Hz), 3.82 (s, 3 H), 3.86 (dd, 1 H, *J* = 11.1 and 16.5 Hz), 6.30 (dd, 1 H), aromatic protons could not be assigned because the signals overlapped with those of the isomer.

3,5,5-Triphenyl-2-isoxazoline (2s): mp 141–142 °C (lit.²⁰ 140–141.5 °C); ¹H NMR (270 MHz, CDCl₃) δ 3.99 (s, 2 H), 7.23–7.74 (m, 15 H); ¹³C NMR δ 48.3, 92.0, 126.1, 126.7, 127.7, 128.5, 128.8, 129.7, 130.2, 144.1, 156.3; IR (KBr) 1493, 1450, 1361, 907, 750, 696, 559 cm⁻¹; MS (70 eV) *m/z* 299 (M⁺). Anal. Calcd for C₂₁H₁₇ON: C, 84.25; H, 5.72; N, 4.68. Found: C, 84.18; H, 5.59; N, 4.60.

5-Methyl-3,5-diphenyl-2-isoxazoline (2t): mp 71–73 °C; ¹H NMR (270 MHz, CDCl₃) δ 1.88 (s, 3 H), 3.92 (ABq, 2 H, Δ*ν* = 13.5 Hz, *J* = 20.3 Hz), 7.10–7.81 (m, 15 H); IR (KBr) 1480, 1430, 1350, 1260, 1050, 890, 820, 740, 680 cm⁻¹. Anal. Calcd for C₁₆H₁₅ON: C, 80.98; H, 6.37; N, 5.90. Found: C, 80.71; H, 6.37; N, 5.65.

General Procedure for the Reaction of 1,1,2,2-Tetra-substituted Cyclopropanes with NOBF₄. NOBF₄ (70 mg, 1.2 equiv) was added to an acetonitrile solution (20 mL) containing **1q** (173 mg, 0.5 mmol) under argon atmosphere at room temperature. The solution immediately turned dark purple and then gradually turned yellow within 20–30 min. The solvent was evaporated under reduced pressure. The residue was triturated with dry THF and filtered to give 2,3,5,5-tetraphenyl-2-isoxazolinium tetrafluoroborate (**5q**, 206 mg, 89%) as a yellow solid: 136–139 °C dec; ¹H NMR (270 MHz, CDCl₃) δ 5.15 (s, 2 H), 7.30–7.80 (m, 20 H); ¹³C NMR δ 50.8 (t), 95.4 (s), 122.4 (s), 126.0 (d), 126.9 (d), 129.2 (d), 129.8 (d), 129.9 (d), 131.0 (d), 131.1 (d), 133.7 (d), 133.9 (d), 136.0 (d), 137.8 (s), 166.0 (s); IR (KBr) 3000, 1580, 1430, 1360, 1180, 1020, 870, 830, 750, 670 cm⁻¹; MS (20 eV) *m/z* 375 (M⁺ – HBF₄). Anal. Calcd for C₂₇H₂₂ONBF₄: C, 70.00; H, 4.78; N, 3.02. Found: C, 69.58; H, 4.63; N, 2.99.

Similar treatment of **1r** gave **5r** and **5r′** quantitatively in a ratio of 5:3 as a dark, oily mixture.

5-Methyl-2,3,5-triphenyl-2-isoxazolinium tetrafluoroborate (5r): ¹H NMR (270 MHz, CDCl₃) δ 2.11 (s, 3 H), 4.54 (d, 1 H, *J* = 19.3 Hz), 4.96 (d, 1 H, *J* = 19.3 Hz), 7.13–7.72 (m, 15 H).

3-Methyl-2,5,5-triphenyl-2-isoxazolinium tetrafluoroborate (5r′): ¹H NMR (270 MHz, CDCl₃) δ 2.61 (s, 3 H), 4.72 (s, 2 H), 7.13–7.72 (m, 15 H), aromatic protons could not be assigned because the signals overlapped with those of the isomer.

Reduction of 5 with LiAlH₄. LiAlH₄ (15 mg, 0.4 mmol) was added by portions over 20 min to the stirred suspension of **5q** (41 mg, 0.11 mmol) in dry THF (10 mL). The resulting mixture was extracted with benzene–ether (1:1; 50 mL) and washed with water. The organic layer was dried over Na₂SO₄ and evaporated under reduced pressure. The ¹H NMR spectrum showed the formation of **6q** in quantitative yield. Treatment of a mixture of **5r** and **5r′** with LiAlH₄ in THF in a similar manner gave a mixture of **6r** and **6r′**.

Synthesis of 6q.¹² A mixture of *N*,*α*-diphenylnitron (1.49 g, 7.6 mmol) and 1,1-diphenylethene (1.46 g, 8.7 mmol) was heated

at 85 °C for 24 h under an argon atmosphere. Recrystallization of the reaction mixture from dichloromethane–methanol (1:2) gave **6q** as colorless crystals.

2,3,5,5-Tetraphenylisoxazolidine (6q): mp 115–116 °C (lit.¹² mp 113–115 °C); ¹H NMR (270 MHz, CDCl₃) δ 3.16 (dd, 1 H, *J* = 8.1 and 12.5 Hz), 3.51 (dd, 1 H, *J* = 8.1 and 12.5 Hz), 4.62 (t, 1 H, *J* = 8.1 Hz), 6.84–7.53 (m, 20 H); ¹³C NMR δ 53.1, 69.0, 87.0, 115.7, 121.5, 126.2, 126.4, 126.8, 127.2, 127.3, 127.5, 128.2, 128.3, 128.4, 128.8, 141.3, 144.1, 144.8, 150.2; IR (KBr) 3030, 2874, 1601, 1491, 1450, 1251, 1029, 994, 845 cm⁻¹; MS (20 eV) *m/z* 377 (M⁺). Anal. Calcd for C₂₇H₂₃ON: C, 85.91; H, 6.14; N, 3.71. Found: C, 85.70; H, 6.00; N, 3.56.

5-Methyl-2,3,5-triphenylisoxazolidine (6r): ¹H NMR (270 MHz, CDCl₃) δ 1.79 (s, 3 H), 3.33 (m, 1 H), 3.70 (m, 1 H), 4.35 (t, 1 H, *J* = 7.3 Hz), 6.85–7.60 (m, 15 H).

3-Methyl-2,5,5-triphenylisoxazolidine (6r′): ¹H NMR (270 MHz, CDCl₃) δ 1.82 (d, 3 H, *J* = 7.8 Hz), 3.30–3.42 (m, 1 H), 3.57–3.71 (m, 1 H), 3.97 (m, 1 H), 6.85–7.60 (m, 15 H).

Hydrogenation of 6q. Compound **5q** (42 mg, 0.09 mmol) was reduced with LiAlH₄ in THF (10 mL) as described above. Methanol (10 mL) and a catalytic amount of Pd on C (5%) were added to the reaction mixture. The resulting mixture was stirred for 12 h under hydrogen atmosphere and extracted with benzene. After evaporation of the solvent, the residue was analyzed by ¹H NMR. The ¹H NMR spectrum showed the formation of 1,1,3-triphenyl-3-(*N*-anilino)-1-propanol (**7**) in quantitative yield: oil; ¹H NMR (270 MHz, CDCl₃) δ 2.64 (dd, 1 H, *J* = 10.9 and 14.9 Hz), 2.83 (dd, 1 H, *J* = 2.4 and 14.9), 4.43 (dd, 1 H, *J* = 2.4 and 10.9 Hz), 4.55–5.20 (br s, 2 H), 6.42 (dd, 2 H, *J* = 0.8 and 7.7 Hz), 6.71 (dt, 1 H, *J* = 0.8 and 7.2 Hz), 7.03 (dd, 2 H, *J* = 7.2 and 7.7 Hz), 7.12–7.58 (m, 15 H).

Reaction of 5q with KCN. An acetonitrile solution (10 mL) containing **5q** (111 mg, 0.24 mmol), KCN (65 mg, 1 mmol), and 18-crown-6 (26 mg, 0.1 mmol) was stirred for 20 h at room temperature. The resulting mixture was extracted with ether–benzene (1:2, 60 mL). The organic layer was washed with water, dried (Na₂SO₄), and evaporated. Recrystallization of the residue from methanol gave 3-cyano-2,3,5,5-tetraphenylisoxazolidine (**8**, 76 mg, 79%) as yellowish crystals: 159–164 °C dec; ¹H NMR (270 MHz, CDCl₃) δ 3.72 (d, 1 H, *J* = 13.3 Hz), 3.96 (d, 1 H), 6.90–7.60 (m, 20 H); ¹³C NMR δ 62.1, 72.7, 86.2, 118.4, 118.5, 124.7, 126.3, 126.7, 126.9, 127.5, 128.1, 128.5, 128.5, 128.6, 129.3, 136.6, 142.4, 144.8, 145.4; IR (KBr) 3064, 2252, 1599, 1493, 1450, 1027, 911, 735, 696 cm⁻¹; MS (20 eV) *m/z* 402 (M⁺). Anal. Calcd for C₂₈H₂₂ON₂: C, 83.55; H, 5.51; N, 6.96. Found: C, 83.08; H, 5.30; N, 6.88.

Decyanation of 8 with AgBF₄.²¹ A mixture of **8** (26 mg, 0.064 mmol) and AgBF₄ (31 mg, 0.2 mmol) in dry 1,2-dichloroethane was stirred for 2 h under argon atmosphere. Precipitates were filtered off, and the filtrate was evaporated under reduced pressure. The residue was analyzed by ¹H NMR. The ¹H NMR spectrum showed the formation of **5q** in quantitative yield.

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Supplementary Material Available: Spectroscopic data, elemental analyses, and melting points for **1e**, **1k–p**, **2l–o**, and **2l′–p′** (4 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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