A New Approach for Oxygenation Using Nitric Oxide under the Influence of *N*-Hydroxyphthalimide

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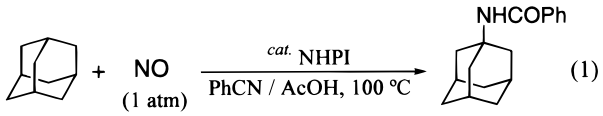
An approach for partial oxygenation through a carbocation as an intermediate was successfully developed by using nitric oxide under the influence of *N*-hydroxyphthalimide. Thus, a variety of benzylic ethers were converted into the corresponding partially oxidized compounds, which are difficult to prepare by conventional methods, in high yields. For example, the reaction of phthalane with NO in the presence of a catalytic amount of NHPI at 60 °C gave phthalaldehyde in 80% yield. The reaction was found to proceed through the formation of a hemiacetal, such as 1-hydroxyphthalane. In addition, 1,3-di-*tert*-butoxymethyl benzene afforded 1,3-benzenedicarbaldehyde in good yield. On the other hand, isochroman was converted into 1,1′-oxodiisochromane under these reaction conditions. The reaction of ethers with NO in the presence of a NHPI catalyst is thought to proceed via the formation of a carbocation as an intermediate. A possible reaction path was suggested.

Introduction

In recent years, much attention has been paid to nitric oxide (NO), which is known to have a free radical character,[1] in the fields of biochemistry[2] and medical science.[3] However, its application to synthetic organic chemistry is quite limited because of the sparse information available on the chemical behavior of NO and the difficulty incurred in controlling its reactivity. Recently, Mukaiyama has reported that nitric oxide could be used as a nitrogen source for the synthesis of nitrogen-containing compounds such as 2-nitrosocarboxamindes[4a] and nitroalkenes.[4b] In addition, it has been shown that amines,[5a,b] phenothiazines,[5c] and dienes[5d] react with nitric oxide in the presence or absence of dioxygen.

We have shown a novel methodology for the catalytic oxidation of alkanes with molecular oxygen using *N*-hydroxyphthalimide (NHPI), which serves as a radical catalyst.[6] In the course of our study to extend the NHPI-catalyzed reaction, our attention was directed toward the use of NO, which has a radical character analogous to molecular oxygen. In a previous paper, we have shown that the reaction of adamantane with NO in the presence of a catalytic amount of NHPI in a mixed solvent of benzonitrile and acetic acid at 100 °C afforded *N*-1-adamantylbenzamide (eq 1).[7] We demonstrate herein an

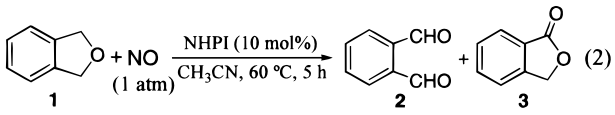


(1)

application of NO to an oxygenation reaction, which is rationally explained by assuming a carbocation as a transient intermediate, under the influence of NHPI as the catalyst.

Results

The reaction of phthalane (**1**) with NO in the presence of a catalytic amount of NHPI was carried out under several conditions. Surprisingly, the reaction of **1** under a NO atmosphere (1 atm) in the presence of NHPI (10 mol %) in acetonitrile at 60 °C for 5 h provided phthalaldehyde (**2**) in 80% yield along with phathalide (**3**) (12%) without formation of a carboxylic acid as well as nitrogen-containing compounds (eq 2, Table 1). The compound **2**



(2)

is an attractive starting material for the synthesis of various important pharmaceuticals. However, there have been few direct, practical synthetic methods so far despite their synthetic importance.[8] Of the various indirect procedures, the hydrolysis of α,α,α′,α′-tetrabromo-*o*-xy-

(1) (a)Walling, C. *Free Radicals in Solution*; Wiley: New York, 1957. (b) Kochi, J. K. *Free Radicals*; Wiley: New York, 1973; Vol. II. (c) Jones, K. *Comprehensive Inorganic Chemistry, Vol. 2;* Trotman-Dickenson, A. F., Ed.; Pergamon Press: Oxford, 1973.

(2) Culotta, E.; Koshland, D. E., Jr. *Science* **1992**, *258*, 1862.

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(4) (a) Kato, K.; Mukaiyama, T. *Chem. Lett.* **1990**, 1395. (b) Hata, E.; Yamada, T.; Mukaiyama, T. *Bull. Chem. Soc. Jpn.* **1995**, *68*, 3629 and references therein.

(5) (a) Nagano, T.; Takizawa, H.; Hirobe, M. *Tetrahedron Lett.* **1995**, *36*, 8239. (b) Itoh, T.; Nagata, K.; Matsuya, Y.; Miyazaki, M.; Ohsawa, A. *J. Org. Chem.* **1997**, *62*, 3582 and references therein. (c) Bosch, E.; Kochi, J. K. *J. Chem. Soc., Perkin Tarans. 1* **1995**, 1057. (d) Park, J. S. B.; Walton, J. C. *J. Chem. Soc., Perkin Trans. 2* **1997**, 2579.

(6) (a) Sakaguchi, S.; Takase, T.; Iwahama, T.; Ishii, Y. *Chem. Commun.* **1998**, 2037. (b) Sakaguchi, S.; Kato, S.; Iwahama, T.; Ishii, Y. *Bull. Chem. Soc. Jpn.* **1998**, *71*, 1, 1237. (c) Yoshino, Y.; Hayashi, Y.; Iwahama, T.; Sakaguchi, S.; Ishii, Y. *J. Org. Chem.* **1997**, *62*, 6810. (d) Ishii, Y.; Iwahama, T.; Sakaguchi, S.; Nakayama, K.; Nishiyama, Y. *J. Org. Chem.* **1996**, *61*, 4520.

(7) Sakaguchi, S.; Eikawa, M.; Ishii, Y. *Teatrahedron Lett.* **1997**, *38*, 7075.

(8) (a) Cha, J. S.; Lee, K. D.; Kwon, O. O.; Kim, J. M.; Lee, H. S. *Bull. Korean. Chem. Soc.* **1995**, *16*, 561. (b) Corriu, R. J. P.; Lanneau, G. F.; Perrot, M. *Tetrahedron Lett.* **1988**, *29*, 1271. (c) Firouzabadi, H.; Iranpoor, N.; Kiaeezadeh, F.; Toofan, J. *Tetrahedron* **1986**, *42*, 719. (d) Green, G.; Griffith, A. P.; Hollinshead, D. M.; Ley, S. V.; Schršder, M. *J. Chem. Soc., Perkin Trans. 1* **1984**, 681. (e) Ariyoshi, K.; Aso, Y.; Otsubo, T.; Ogura, F. *Chem. Lett.* **1984**, 891.

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Table 1. Reaction of 1 with NO under Selected Conditions[a]

run	catalyst	(mol %)	conv (%)	selectivity (%) 2	3	4
1	NHPI	(10)	100	80	12	<1
2[b]	NHPI	(10)	95	44	11	35
3	NHPI	(0)	no reaction			
4[c]	NHPI	(10)	100	52	15	24
5	NHMI	(10)	100	76	10	<1
6	NHTCPI	(10)	94	53	7	39
7	NHSI	(10)	32	63	9	<1
8	PI	(10)	no reaction			

[a] **1** (1 mmol) was allowed to react in the presence of NHPI under NO atmosphere (1 atm) in acetonitrile (5 cm^3) at 60 °C for 5 h. [b] Reaction was carried out at 40 °C. [c] Reaction was carried out in absolute acetonitrile. NHMI, *N*-hydroxymaleimide; NHTCPI, *N*-hydroxytetrachlorophthalimide; NHSI, *N*-hydroxysuccinimide; PI, phthalimide.

lene is thought to be the best method,[9] although α,α,α′,α′-tetrabromo-*o*-xylene must be prepared independently by bromination of *o*-xylene using excess bromine. Therefore, the present NHPI-catalyzed reaction of **1** with NO is considered to be a very convenient direct route to **2**.

The reaction took place even at 40 °C to give **2** (44%) and 1-hydroxyphthalane (**4**)[10] (35%) (run 2). Needless to say, no reaction took place in the absence of NHPI (run 3). Since the water content in the solvent is thought to be a dominant factor, the reaction was examined in acetonitrile dehydrated with CaH_2, although thorough removal of water from acetonitrile is difficult.[11] As predicted, the formation of **2** was restricted, and **4** was obtained in 24% yield (run 4). Several *N*-hydroxyimide derivatives analogous to NHPI were examined in the reaction of **1** with NO. *N*-Hydroxymaleimide and *N*-hydroxytetrachlorophthalimide were found to promote the present reaction, giving **2** and **4** in good yields (runs 5 and 6). However, *N*-hydroxysuccinimide resulted in **2** in a low conversion (run 7).

Figure 1 shows the time-dependence curves for the reaction of **1** with a NO/NHPI system. At an earlier stage

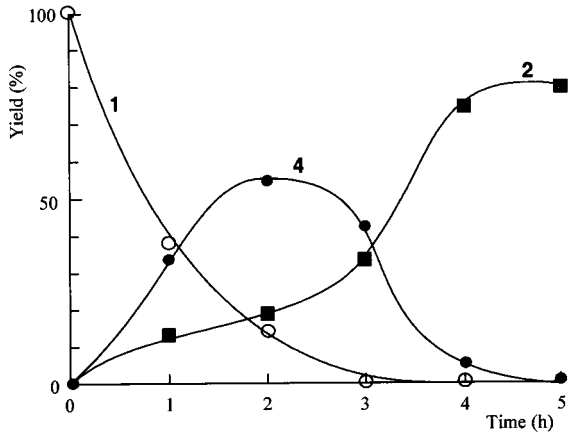


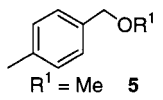
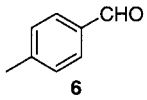
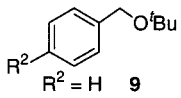
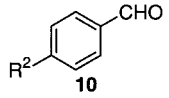
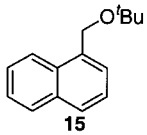
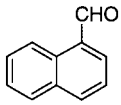
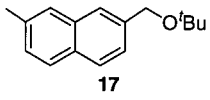
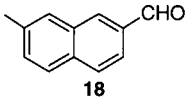
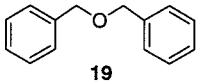
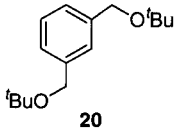
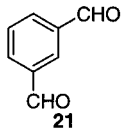
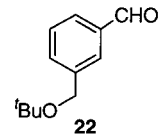
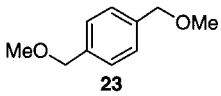
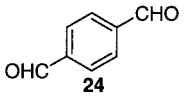
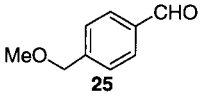
Figure 1. Time-dependence curves for the reaction of **1** with NO catalyzed by NHPI in CH_3CN at 60 °C.

(9) Bill, J. C.; Tarbell, D. S. *Organic Syntheses, Collect. Vol. IV*; Wiley: New York, 1963; p 807.

(10) **4** was found to exist in equilibrium with cyclic hemiacetal form 2-(hydroxymethyl)benzaldehyde by NMR measurement. Harron, J.; McClellnad, R. A.; Thankachan, C.; Tidwell, T. T. *J. Org. Chem.* **1981**, *46*, 903.

(11) Absolute acetonitrile containing water (<0.005%) was purchased from Wako Pure Chemical Industries, Ltd.

Table 2. Reaction of Benzyl Ethers by NO/NHPI System[a]

Run	Substrate	Product	Conv. (%)	Yield (%)
1	 R^1 = Me **5**	 **6**	56	50
2	= Et **7**	**6**	70	60
3	= ^{t}Bu **8**	**6**	90	72
4	 R^2 = H **9**	 **10**	80	70
5	= ^{t}Bu **11**	**12**	95	84
6	= Cl **13**	**14**	83	74
7	 **15**	 **16**	87	80
8	 **17**	 **18**	93	85
9[b]	 **19**	**10**	82	64
10	 **20**	 **21**	99	75
		 **22**		10
11	 **23**	 **24**	96	53
		 **25**		20

[a] Substrate (1 mmol) was allowed to react in the presence of NHPI (10 mol %) under NO atmosphere (1 atm) in acetonitrile (5 cm^3) at 60 °C for 10 h. [b] Benzyl alcohol (30%) was obtained.

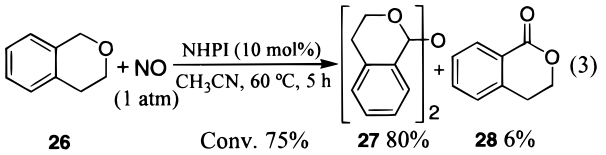
of the reaction, **4** was produced as the major product. After 2 h, **2** was obtained as the major product, and the yield of **4** decreased. This indicates that **2** was produced through the formation of **4**.

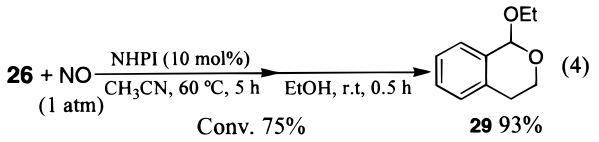
Since the NHPI-catalyzed reaction of **1** with NO was found to provide an interesting route of benzylic ethers to aldehydes and/or their synthetic equivalents, hemiacetals, which are difficult to prepare by conventional methods, the present reaction was extended to various ethers (Table 2). A variety of benzyl ethers used were converted successfully into the coresponding aldehydes in moderate to good yields. The reaction of 4-methoxymethyl toluene (**5**) catalyzed by NHPI (10 mol %) under NO atmosphere for 5 h afforded *p*-tolualdehyde (**6**) in 50% yield at 56% conversion (run 1). To improve the yield of **6**, several benzyl ethers were prepared, and 4-*tert*-

butoxymethyl toluene (**8**) was found to be transformed into **6** in 72% yield (run 3). Thus, *tert*-butoxymethyl benzene derivatives were allowed to react with NO in the presence of the NHPI catalyst. The reaction of 1-*tert*-butoxymethyl-4-*tert*-butylbenzene (**11**) gave 4-*tert*-butylbenzaldehyde in 84% yield, while 1-*tert*-butoxymethyl-4-chlorobenzene (**13**) resulted in 4-chlorobenzaldehyde in somewhat lower yield (74%) under these reaction conditions (runs 5 and 6).

The reaction of dibenzyl ether (**19**) also proceeded smoothly to form benzaldehyde (**10**) in 64% yield together with benzyl alcohol (30%) without formation of benzoic acid. It is interesting to note that benzenedicarbaldehyde derivatives, which are difficult to prepare up to now, were synthesized by the use of the present NO/NHPI system. The reaction of 1,3-di-*tert*-butoxymethyl benzene (**20**) afforded 1,3-benzenedicarbaldehyde (**21**) in 75% yield (run 10). 4-Terephthalaldehyde (**24**) was obtained from the reaction of 1,4-dimethoxymethyl benzene (**23**), along with a small amount of 4-methoxymethyl benzaldehyde (**25**).

On the other hand, the reaction of isochroman (**26**) (see eqs 3 and 4) catalyzed by NHPI (10 mol %) under NO





atmosphere afforded 1,1′-oxodiisochromane (**27**) in 80% yield together with 1-isochromanone (**28**) (6%). To our best knowledge, there is only one report on the conversion of **26** to **27** using a stoichiometric amount of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ).[12] The product **27** was isolated as a single crystal by recyclization with diethyl ether and was analyzed by X-ray measurement (Figure 2). Interestingly, 1-ethoxyisochromane (**29**) was obtained in excellent yield (93%) by allowing **27** to react with ethanol (see Experimental Section). The compound **29** is known to be an important precursor of antitumor active compounds, such as isochromanyltroplones.[13]

Discussions

Mechanistically, the present reaction is rationally explained by considering the formation of a carbocation as a transient intermediate (Scheme 1). In a previous paper, we showed that phthalimide-*N*-oxyl (PINO) is generated by exposing NHPI to nitric oxide.[7] Janzen et al. have also reported that NO can abstract the phenolic hydrogen atom from phenol to form a phenoxy radical.[14]

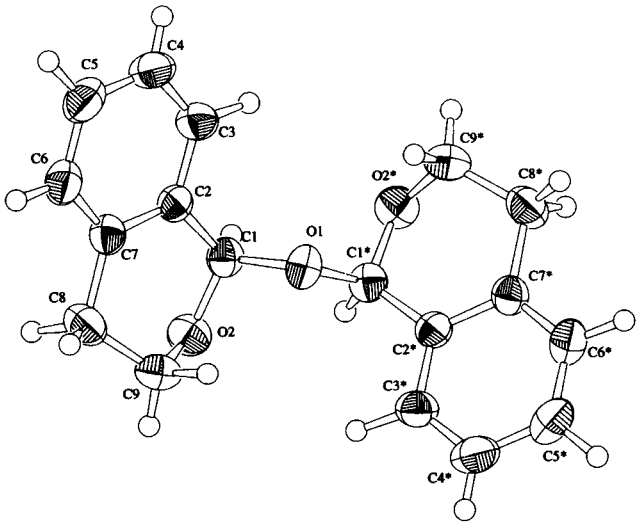
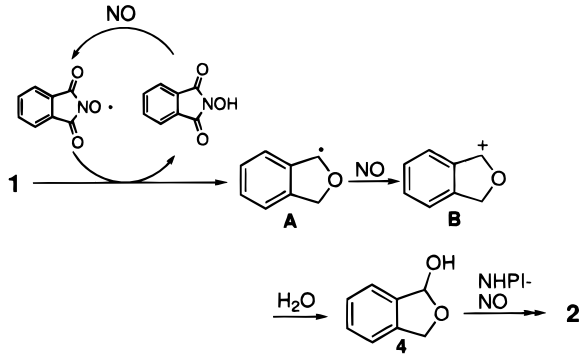


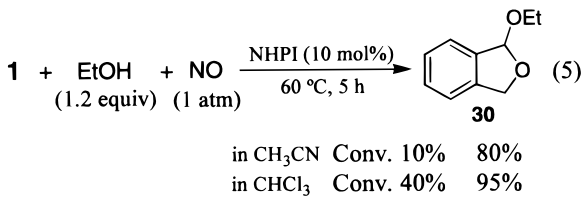
Figure 2. X-ray structure of **27**.

Scheme 1. A Possible Reaction Path for the Reaction of 1 by the NO/NHPI System



Hence, PINO derived from NHPI and NO can abstract the benzylic hydrogen atom from **1** to generate a radical **A** that is then reacted with NO to give a cation **B**. Suzuki has suggested the formation of a cationic species through a diazonium nitrate in the nitration of alkenes with NO.[15] Nucleophilic attack of the water to the cationic species **B** would result in **2** through 1-hydroxyphthalane **4**.

According to the above reaction sequence, the **B** may be trapped by a nucleophile, such as alcohol. Thus, we tried the NHPI-catalyzed reaction of **1** with NO in the presence of ethanol. The reaction of **1** catalyzed by NHPI in the presence of 1.2 equiv of ethanol with respect to **1** under NO in acetonitrile at 60 °C for 5 h gave 1-ethoxyphthalane (**30**) (80%) captured by ethanol, although the conversion was low (10%) (eq 5). However, when the

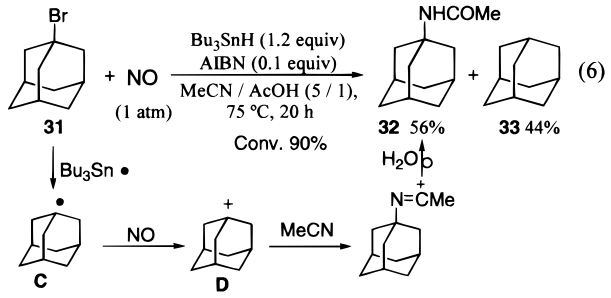


reaction was carried out in chloroform containing 1.2 equiv of ethanol, **30** was obtained in 95% selectivity at 40% conversion.

To obtain further information on the generation of an alkyl cation from an alkyl radical under the influence of NO, the transformation of an adamantyl radical to an

(12) Xu, Y.-C.; Lebeau, E.; Gillard, J. W.; Attardo, G. *Tetrahedron Lett.* **1993**, *34*, 3841.
(13) Yamato, M.; Hashigaki, K.; Yasumoto, Y.; Sakai, J.; Luduena, R. F.; Banerjee, A.; Tsukagoshi, S.; Tashiro, T.; Tsuruo, T. *J. Med. Chem.* **1987**, *30*, 1897 and references therein.
(14) Wilcox, A. L.; Janzen, E. G. *J. Chem. Soc., Chem. Commun.* **1993**, 1377.
(15) Suzuki, H. *Pharmacia* **1997**, *33*, 487.

adamantyl cation with NO was examined. Under a NO atmosphere, 1-bromoadamantane (**31**) was allowed to react with tributyltinhydride and AIBN in MeCN/AcOH, producing *N*-1-adamantylacetoamide (**32**) (56%) and adamantane (**33**) (44%) (eq 6). This indicates that the



resulting adamantyl radical **C** generated from **31** by the action of Bu_3SnH/AIBN was converted into an adamantyl cation **D** under the influence of NO, to give amide **32** through the reaction with MeCN, which is known as the Ritter reaction.[7]

In conclusion, we have developed a new type of oxygenation through a carbocation as a transient intermediate, using an NO/NHPI system. This method provides a facile synthetic route to aldehydes or acetals, which so far have been difficult to obtain. The most striking characteristic of the present reaction is that it did not produce a carboxylic acid since the reaction is based on the capture of water with carbocation.

Experimental Section

General Procedures. The starting materials such as **1**, **5**, **7**, **19**, **23**, **26**, and **31**, and catalysts used were commercially available and used without further purification. The compounds **8**, **9**, **11**, **13**, **15**, **17**, and **20** were synthesized by the reaction of the coresponding alcohols with sodium *tert*-butoxide in anhydride THF at room temperature and were isolated by column chromatography on silica gel (hexane/ethyl acetate = 10:1). GC analysis was performed with a flame ionization detector using a 0.2 mm × 25 m capillary column (OV-1). ^{1}H and ^{13}C NMR were measured at 270 and 67.5 MHz, respectively, in $CDCl_3$ with Me_4Si as the internal standard. The yields of all products except for **27** were estimated from the peak areas based on the internal standard technique. The yield of **27** is isolated yield.

A Typical Procedure for the Reaction of 1. To a solution of **1** (1 mmol) in acetonitrile (5 cm^3) in a three-necked flask was added NHPI (0.1 mmol). The flask was cooled to −78 °C to freeze the solvent and degassed in vacuo and filled with Ar gas. Then the frozen solvent was melted at room temperature and refrozen to reiterate the evacuation−Ar purge procedure. The series of operations was repeated three times, and then NO was added to the reaction vessel. The reaction mixture was allowed to react under an atmospheric pressure of NO at 60 °C for 5 h. After solvent was removed in vacuo, the products were isolated by column chromatography on silica gel (chloroform) and characterized by ^{1}H and ^{13}C NMR, respectively.

Products **2**, **6**, **10**, **12**, **14**, **16**, **21**, and **24** were identified by comparing of the isolated products with authentic samples.

1-Hydroxyphthalane (4): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 10.2 (s, 1 H), 7.86−7.23 (m, 8 H), 6.41 (s, 1 H), 5.28 (m, 2 H), 5.13−5.09 (m, 3 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 192.5, 140.4, 139.9, 133.7, 131.8, 129.2, 128.8, 127.9, 127.7, 123.1, 123.0, 121.0, 120.9, 106.6, 72.2, 63.4.

7-Methyl-2-naphthylaldehyde (18): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 9.99 (s, 1 H), 8.07−7.35 (m, 6 H), 2.42 (s, 3 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 192.2, 136.8, 134.6, 133.7, 132.7, 131.2, 128.6, 128.3, 127.7, 121.8, 21.5.

3-*tert*-Butoxymethylbenzaldehyde (22): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 10.6 (s, 1 H), 8.46−7.45 (m, 4 H), 4.51 (s, 2 H), 1.31 (s, 9 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 192.2, 136.3, 134.9, 133.2, 128.8, 128.4, 128.3, 73.6, 63.2, 27.5.

4-Methoxymethylbenzaldehyde (25): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 8.79 (s, 1 H), 6.66 (d, J = 7.8 Hz, 2 H), 6.29 (d, J = 7.8 Hz, 2 H), 3.33 (s, 2 H), 2.23 (s, 3 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 191.9, 145.3, 135.6, 129.8, 127.6, 73.8, 58.4.

1,1′-Oxodiisochromane (27): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 7.18 (m, 8 H), 6.11 (s, 2 H), 4.34 (dt, J = 6.2 and 11.3 Hz, 2 H), 4.11−4.04 (m, 2 H), 3.11 (dt, J = 6.2 and 11.3 Hz, 2 H), 2.68−2.61 (m, 2 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 134.1, 133.8, 128.4, 128.0, 127.5, 126.3, 92.7, 58.1, 27.9. Anal. Calcd. for $C_{18}H_{18}O_3$: C, 76.48; H, 6.42. Found: C, 76.43, H, 6.38. The compound **27** ($C_{18}H_{18}O_3$; M_r = 282.34) crystallized in the orthorhombic space group *C*2/*c* with cell dimensions of a = 23.355(6) Å, b = 4.346(3) Å, and c = 15.926 (5) Å; β = 117.98-(2)°, V = 1427(1) Å^3 and an occupation of Z = 4 in cell unit. Data were collected at 24.0 ± 1 °C on a AFC7R Rigaku diffractometer (Mo Kα radiation), to a maximum $2q$ = 55.0°, giving 1649 unique reflections; the strucutre was solved by direct methods (SIR88) and refined within full matrix least squares, yielding R = 0.059, R_w = 0.113 (GOF = 1.76) for 1495 unique reflections with $I > 1.10\sigma(I)$.

Procedure for the Reaction of Isochroman (26) to 1-Ethoxyisochromane (29). To a solution of **26** (1 mmol) in acetonitrile (5 cm^3) in a three-necked flask was added NHPI (0.1 mmol). The flask was cooled to −78 °C to freeze the solvent and degassed in vacuo and filled with Ar gas. Then the frozen solvent was melted at room temperature and refrozen to reiterate the evacuation−Ar purge procedure. The series of operations was repeated three times, and then NO was added to the reaction vessel. After the reaction mixture was allowed to react under an atmospheric pressure of NO at 60 °C for 5 h, EtOH (5 cm^3) was added, then the reaction mixture was stirred at room temperature for 0.5 h. The products were purified and identified by the same method as previously described.

1-Ethoxyisochromane (29): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 7.20−7.07 (m, 4 H), 5.54 (s, 1H), 4.19−3.63 (m, 4 H), 3.05−2.92 (m, 1 H), 2.61−2.56 (m, 1 H), 1.28 (t, J = 7.0 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 134.3, 133.9, 128.3, 127.9, 127.3, 126.2, 96.4, 63.3, 57.7, 27.9, 15.2.

1-Ethoxyphthalane (30): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 7.29−7.10 (m, 4 H), 6.13 (s, 1H), 5.11−4.87 (m, 4 H), 3.70−3.49 (m, 2 H),1.14 (t, J = 7.0 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 139.7, 137.5, 128.8, 127.4, 122.7, 120.7, 106.5, 71.9, 62.7, 15.2.

Procedure for the Reaction of 1-Bromoadamantane (31) with NO in the Presence of Bu_3SnH and AIBN. To a solution of **31** (1 mmol) in a mixed solvent of acetonitrile and acetic acid (5:1 cm^3) in a three-necked flask was added Bu_3SnH (1.2 mmol) and AIBN (0.1 mmol). The flask was cooled to −78 °C to freeze the solvent, degassed in vacuo, and filled with Ar gas. Then, the frozen solvent was melted at room temperature and refrozen to reiterate the evacuation−Ar purge procedure. The series of operations was repeated three times, and then NO was added to the reaction vessel. The reaction mixture was allowed to react under an atmospheric pressure of NO at 75 °C for 20 h. The products were purified by the same method as previously described. Products **32** and **33** were identified by comparison of the isolated products with authentic samples.

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Supporting Information Available: Copies of ^{1}H and ^{13}C NMR spectra for the compounds **2**, **4**, **16**, **18**, **21**, **22**, **24**, **25**, **27**, **29**, **30**, and **32** and X-ray of $C_{18}H_{18}O_3$. This information is available free of charge via the Internet at http://pubs.acs.org.

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