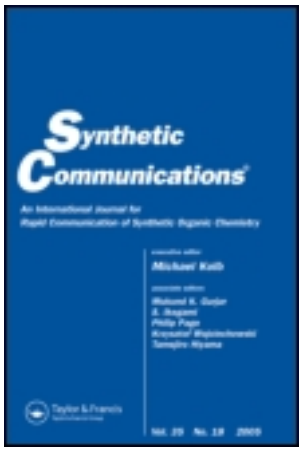


Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

INDIUM MEDIATED REDUCTIVE ACYLATIONS OF NITROARENES TOWARDS N,O-DIACYLATED N-ARYLHYDROXYLAMINES*

Byeong Hyo Kim [a], Jae Wook Cheong [a], Rongbi Han [a], Young Moo Jun [a], Woonphil Baik [b] & Byung Min Lee [c]

[a] Department of Chemistry, Kwangwoon University, Seoul, 139-701, South Korea

[b] Department of Chemistry, Myong Ji University, Kyung Ki Do, South Korea

[c] Korea Research Institute of Chemical Technology, Taejon, South Korea

Published online: 16 Aug 2006.

To cite this article: Byeong Hyo Kim , Jae Wook Cheong , Rongbi Han , Young Moo Jun , Woonphil Baik & Byung Min Lee (2001): INDIUM MEDIATED REDUCTIVE ACYLATIONS OF NITROARENES TOWARDS N,O-DIACYLATED N-ARYLHYDROXYLAMINES* , Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 31:23, 3577-3586

To link to this article: http://dx.doi.org/10.1081/SCC-100107004

SYNTHETIC COMMUNICATIONS, 31(23), 3577–3586 (2001)

INDIUM MEDIATED REDUCTIVE ACYLATIONS OF NITROARENES TOWARDS *N,O*-DIACYLATED *N*-ARYLHYDROXYLAMINES*

Byeong Hyo Kim,[1,†] Jae Wook Cheong,[1] Rongbi Han,[1] Young Moo Jun,[1] Woonphil Baik,[2] and Byung Min Lee[3]

[1]Department of Chemistry, Kwangwoon University, Seoul, 139-701, South Korea
[2]Department of Chemistry, Myong Ji University, Kyung Ki Do, South Korea
[3]Korea Research Institute of Chemical Technology, Taejon, South Korea

ABSTRACT

By applying indium, Ac_2O, MeOH, and catalytic amount of $InCl_3$ in $CHCl_3$ solution, nitroarenes were transformed into *N,O*-diacylated *N*-arylhydroxylamines in moderate to excellent yields.

*Dedicated to Professor Jack W. Timberlake on the occasion of his 60th Birthday.

†Corresponding author. E-mail: bhkim@daisy.gwu.ac.kr

Recently, indium-mediated reactions have been focused on synthetic applications because of environmental issues and the ease of reactions obviating the need for inflammable anhydrous organic solvents and inert atmosphere.[1]

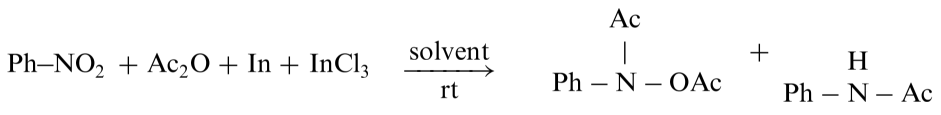
Indium was applied for the various reactions in aqueous conditions without inert atmosphere; alkylation of aldehydes and ketones,[2] reductive coupling of aldimines,[3] Reformatsky and aldol reactions,[4] allenylation of aldehydes,[5] ring expansion of carbocycles,[6] and reduction of some functional groups.[7]

Undoubtedly, indium metal can be a suitable candidate for an electron donor in single electron transfer (SET) processes and has some striking advantages over other metals, *i.e.*, indium metal is stable in air or oxygen at ordinary temperatures and is practically unaffected by water. Of the special interest to us was the possibility of utilizing indium promoted reaction for the preparation of *N*,*O*-diacylated *N*-arylhydroxylamines, since esters of *N*-hydroxy-*N*-arylacetamides, such as $ArN(OR)COCH_3$ ($R{=}COCH_3$, SO_3^-), are considered to be the reactive metabolites of mutagenic and carcinogenic aromatic amides.[8] In spite of their biological interest, useful preparative methods for esters of *N*-hydroxy-*N*-arylacetamides were not well established. While the direct chemical reductive diacylations of nitroarenes were not successful,[9,10] the electrochemical reduction of both aliphatic and aromatic nitro or nitroso compound in aprotic media with Ac_2O produced *N*,*O*-diacylated hydroxylamines in yields from 40 to 80%.[11] We previously reported Zn-mediated reductive diacylation of nitroarenes which revealed the improved result compared to other reactions.[12] Herein, we wish to report on the study of efficient chemical reductive one-pot *N*,*O*-diacylation reaction of nitroarenes using indium metal, $InCl_3$ and Ac_2O in aprotic solvent under mild condition.

Various control experiments with nitrobenzene were exerted to explore the optimum condition for the effective reductive diacylation on nitro functional group towards Ph-N(Ac)OAc. Unfortunately, the reaction did not proceed in the presence of Ac_2O/indium only (Table 1, entries 1, 2). To make the reaction progress, some additives seem to be needed to activate a substrate and/or an electrophile. Thus, reactions with nitrobenzene/Ac_2O (4 equiv)/In (5 equiv)/$InCl_3$ (0.2 equiv) were tried, and surprisingly the reaction was started to take place through the influence of a catalytic amount of $InCl_3$ (entries 3–8). The reaction in aprotic solvents such as THF (entry 3), CH_2Cl_2 (entry 4), and $CHCl_3$ (entry 8) proceeded quite slowly while reactions in protic solvent system revealed fast reactions (entries 5–7). Protic solvent system definitely promotes the reaction.

However, because of the increased formation of undesirable acetanilide rather than desired *N*,*O*-diacylated product, it was required to

Table 1. Control Experiments for the Reductive Acylation of Nitrobenzene in the Presence of Indium and Additives at Room Temperature



1 **2** **3** **4** **5a** **6a**

Entry	Molar Ratio **1:2:3:4:**MeOH	Solvent	Time (h)	Yield (%)[a] **5a**	**6a**
1	1:4:5:- :-	MeOH	48	tr	tr
2	1:4:5:- :-	$CHCl_3$	48	tr	tr
3	1:4:5:0.2:-	THF	48	52	10
4	1:4:5:0.2:-	CH_2Cl_2	48	27	23
5	1:4:5:0.2:-	MeOH	5	10	65
6	1:4:5:0.2:-	THF/H_2O[b]	4	40	12
7	1:4:5:0.2:-	$CHCl_3$/MeOH[b]	2.5	25	50
8	1:4:5:0.2:-	$CHCl_3$	42	57	3
9	1:4:5:- :2	$CHCl_3$	48	69	13
10	1:4:5:0.2:2	$CHCl_3$	20	81	13

[a]GC yield with an internal standard.
[b]v/v = 4/1.

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

apply just minimum amount of proton source. Nitrobenzene when was treated with Ac_2O (4 equiv)/In (5 equiv)/$InCl_3$ (0.2 equiv)/MeOH (2 equiv) in $CHCl_3$ at room temperature for 20 h, gave *N,O*-diacylated phenylhydroxylamine, PhN(OAc)Ac in 81% along with 13% of acetanilide (entry 10) which was the best result for the desired *N,O*-diacylated phenylhydroxylamine.

We also tried several Lewis acids instead of $InCl_3$ to see whether or not other Lewis acids would exhibit a better result compared to $InCl_3$. Table 2 shows the results of the reactions of nitrobenzene with several selected Lewis acids.

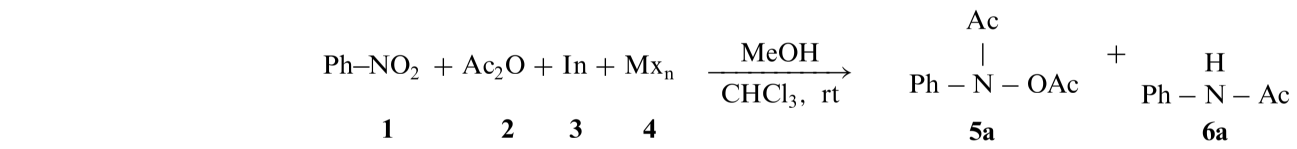
$InCl_3$ was found to be the best choice for our reductive diacylation. Reaction with $AlCl_3$ or BF_3 resulted in relatively lower yield compared to the reaction with $InCl_3$ (Table 2, entries 2, 3) while reaction with $ZnCl_2$ or $TiCl_4$ exhibited an increase of a by-product, acetanilide (entries 4, 5).

On the basis of these results, we have extended the diacylation reaction to a variety of nitroarenes and results are summarized in Table 3. In most cases, diacylations of nitroarenes were successful with fair to excellent yields. We tried the same reaction with nitrosobenzene in place of nitrobenzene and diacylated product **5a** was obtained in 45% along with 40% of acetanilide (Table 3, entry 1) within an hour that implies fast formation of nitrosobenzene radical anion. It is clear that the acylation of nitroarenes seems to proceed through a nitrosobenzene intermediate. Not only nitroarenes but also nitropyridine derivative, diacylated product was obtained in a reasonable yield (entry 10).

Similar to our previous study using Zn,[12] nitroarenes could be initialized by accepting an electron from indium metal since nitroarenes have been shown the ability to form radical anions spontaneously in the presence of electron donors.[13] In fact, the LUMO energy level of nitroarenes lies in a relatively low, thus the formation of $ArNO_2^-$ can be explained by a SET process. In addition, nitrosoarenes are also capable of accepting an electron. Russell reported that the SET process of nitrosobenzene in the presence of hydroxide ion occurs in > 0.5 sec to give nitrosobenzene radical anion.[14] In the case of nitrobenzene, reductive wave was observed at -0.92 V that indicated nitrobenzene could be a good electron acceptor while acetic anhydride at the same cyclic voltametric condition didn't exhibit any observable reduction wave.[12] Even in the presence of $InCl_3$, reductive wave of nitrobenzene was not much changed (intensity was diminished) which indicated that $InCl_3$ did not influence the electron transferring ability from In to nitrobenzene. We believe the role of $InCl_3$ is to increase the electrophilicity of acetic anhydride by forming complexes with carbonyl group. For mechanistic purposes, some inhibition experiments were carried out. In the presence of 20 mol% of 1,3-dinitrobenzene,[15a] the reductive

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

Table 2. Reactions of Nitrobenzene with Various Lewis Acids (0.2 Equiv) and Ac_2O (4 Equiv)/Indium (5 Equiv)/MeOH (2 Equiv) in $CHCl_3$ at Room Temperature



1 **2** **3** **4** **5a** **6a**

Entry	MX_n	Time (h)	Yield (%)[a] **5a**	**6a**
1	$InCl_3$	20	81	13
2	$AlCl_3$	36	60	16
3[b]	$BF_3 \cdot Et_2O$	20	30	18
4	$ZnCl_2$	36	42	27
5	$TiCl_4$	7	30	37

[a]GC yield with an internal standard.
[b]Nitrobenzene remained.

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

Table 3. The Reactions of Substituted Nitroarenes in the Presence of Ac_2O (4 Equiv)/Indium (5 Equiv)/$InCl_3$ (0.2 Equiv)/MeOH (2 Equiv) in $CHCl_3$ at Room Temperature

$$Ar-NO_2 + Ac_2O + In + InCl_3 \xrightarrow[rt]{MeOH/CHCl_3} \underset{\mathbf{5}}{Ar-\overset{\overset{Ac}{|}}{N}-OAc}$$

Entry	Substrate	Time (h)	Yield (**5**, %)[a]
1	nitrosobenzene	1	45 (**5a**)[b]
2	nitrobenzene	20	81 (**5a**)
3	3-nitrotoluene	31	39 (**5b**)
4	4-nitrotoluene	48	74 (**5c**)
5	1-chloro-2-nitrobenzene	44	95 (**5d**)
6	1-chloro-3-nitrobenzene	19	93 (**5e**)
7	1-bromo-4-nitrobenzene	26	99 (**5f**)
8	1-cyano-3-nitrobenzene	12	83 (**5g**)
9	1-cyano-4-nitrobenzene	8	71 (**5h**)
10[c]	2-chloro-5-nitropyridine	6	64 (**5i**)

[a]GC yield with an internal standard, acetanilide was obtained as a minor product mostly.
[b]40% of acetanilide was obtained.
[c]At 50°C.

acylation was retarded and the yield of *N*,*O*-diacylated product decreased to less than 35% (20 h). Clearly, electron transfer processes through the radical anion species are involved during the reductive acylation reaction. The possibility of radical intermediate could be excluded as the reaction of nitrobenzene/Ac_2O/In/$InCl_3$ at room temperature in the presence of 20 mol% of di-*tert*-butyl nitroxide[15b–d] resulted in ineffective inhibition. Thus radical anion of nitrobenzene generated from the electron transfer from In to nitrobenzene may react rapidly as a nucleophile towards the acetic anhydride electrophile activated by $InCl_3$ to form a reactive intermediate followed by an electron transfer and the loss of acetate anion to produce the nitroso intermediate. With similar continuous electron transfer and acylation, the nitroso intermediate could be transformed to *N*,*O*-diacylated *N*-arylhydroxylamine.

In conclusion, we have now established a mild and novel reaction route for *N*,*O*-diacylated *N*-arylhydroxylamines by using Ac_2O/In/$InCl_3$/MeOH in $CHCl_3$ which would be a useful chemical method for the reductive diacylation of nitroarenes.

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

EXPERIMENTAL

A Typical Procedure for the *N,O*-Diacetylation of Nitroarenes

Indium powder 574 mg (5 mmol), indium trichloride 44 mg (0.2 mmol) and $CHCl_3$ (4 mL) were placed in 20 mL vial equipped with a rubber septum. Acetic anhydride 0.38 mL (4 mmol) and MeOH 0.05 mL (2 mmol) were added to the reaction mixture followed by nitroarene (1 mmol). The mixture was stirred under nitrogen at room temperature. After the reaction was completed, the mixture was quenched with 10% NH_4Cl and was extracted with CH_2Cl_2 (50 mL × 3). The combined CH_2Cl_2 extract was dried over $MgSO_4$ and the solvent was evaporated. The GC yield was determined with an internal standard and if necessary, the products were isolated by flash column chromatography with ethyl acetate–hexane co-solvent.

N,O-Diacetylphenylhydroxylamine (5a)[12]

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.37; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46–7.39 (m, 5H), 2.17 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.8, 139.3, 129.4, 128.3 (bd), 21.5, 18.8; IR (nujol) 3059, 2993, 1803, 1694, 1600, 1496 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 193 (M^+, 3), 151 (94), 109 (100); HRMS (EI) calcd for $C_{10}H_{11}NO_3$ 193.0739, found 193.0754.

N,O-Diacetyl-3-methylphenylhydroxylamine (5b)[12]

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.39; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34–7.21 (m, 4H), 2.38 (s, 3H), 2.19 (s, 3H), 2.06 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.7, 166.6, 139.4, 139.1, 130.6, 130.4, 129.1, 125.1, 21.3, 21.0, 18.2; IR (nujol) 3057, 2992, 1799, 1688, 1378 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 207 (M^+, 1), 165 (66), 123 (100), 105 (40); HRMS (EI) calcd for $C_{11}H_{13}NO_3$ 207.0895, found 207.0899.

N,O-Diacetyl-4-methylphenylhydroxylamine (5c)[12]

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.39; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36–7.34 (d, 2H, $J = 6.5$ Hz), 7.24–7.22 (d, 2H, $J = 8.6$ Hz), 2.38 (s, 3H), 2.18 (s, 3H), 2.03 (s, 3H); ^{13}C (125 MHz, $CDCl_3$) δ 167.8, 136.8,

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

132.2, 130.1, 128.2, 21.4, 21.1, 18.2, IR (nujol) 3063, 2992, 1799, 1688, 1427, 1284 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 207 (M^+, 3), 165 (82), 123 (100), 105 (100); HRMS (EI) calcd for $C_{11}H_{13}NO_3$ 207.0895, found 207.0888.

N,*O*-Diacetyl-2-chlorophenylhydroxylamine (5d)[11c]

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.30; ^1H NMR (300 MHz, $CDCl_3$) δ 7.65–7.00 (m, 4H), 2.14 (s, 3H), 2.00 (s, 3H); ^{13}C (125 MHz, $CDCl_3$) δ 168.6, 167.6, 136.6, 132.0, 130.6, 129.0, 127.4, 124.8, 20.8, 18.1; IR (nujol) 3073, 2938, 1797, 1702, 1476 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 229 (1), 227 (M^+, 3), 187 (23), 185 (69), 145 (35), 143 (100) 125 (27), 101 (4), 99 (14); HRMS (EI) calcd for $C_{10}H_{10}ClNO_3$ 227.0349, found 227.0344.

N,*O*-Diacetyl-3-chlorophenylhydroxylamine (5e)

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.23; ^1H NMR (300 MHz, $CDCl_3$) δ 7.38 (s, 1H), 7.27–7.19 (m, 3H), 2.10 (s, 3H); 1.98 (s, 3H); ^{13}C (125 MHz, $CDCl_3$) δ 167.7, 140.1, 134.5, 130.1, 127.9, 123.5 (bd), 21.3, 18.1; IR (nujol) 3057, 2987, 1804, 1695, 1429 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 229 (1), 227 (M^+, 3), 187 (25), 185 (74), 145 (35), 143 (100), 125 (28), 101 (3), 99 (9); HRMS (EI) calcd for $C_{10}H_{10}ClNO_3$ 227.0349, found 227.0349.

N,*O*-Diacetyl-4-bromophenylhydroxylamine (5f)[11c]

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.21; ^1H NMR (300 MHz, $CDCl_3$) δ 7.56–7.53 (d, 2H, $J = 8.4$ Hz), 7.37–7.34 (d, 2H, $J = 8.2$ Hz), 2.20 (s, 3H), 2.05 (s, 3H); ^{13}C (125 MHz, $CDCl_3$) δ 167.8, 138.3, 132.5, 131.0, 130.7, 21.4, 18.3; IR (nujol) 3093, 2933, 1799, 1696, 1486 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 231 (48), 229 (50), 189 (65), 187 (68), 172 (20), 171 (100), 170 (22), 169 (94) 90 (43); HRMS (EI) calcd for $C_{10}H_{10}BrNO_3$ 270.9844, found 270.9852.

N,*O*-Diacetyl-3-cynophenylhydroxylamine (5g)

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.20; ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.71–7.67 (m, 1H), 7.49–7.44 (m, 2H), 2.23 (s, 3H), 2.12 (s, 3H); ^{13}C (125 MHz, $CDCl_3$) δ 168.6, 167.5, 139.6, 129.8, 126.8, 123.3, 122.2, 117.5, 112.9, 21.3, 18.2; IR (nujol) 3053, 2991, 2233, 1808, 1699,

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

1421, 1282 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 218 (M^+, 2), 176 (100), 134 (58), 116 (18); HRMS (EI) calcd for $C_{11}H_{10}N_2O_3$ 218.0691, found 218.0687.

N,*O*-Diacetyl-4-cyanophenylhydroxylamine (5h)

White solid, mp = 80.1–80.3°C; TLC (30% ethyl acetate/hexane) R_f 0.26; ^1H NMR (300 MHz, $CDCl_3$) δ 7.45 (s, 4H), 2.16 (s, 3H), 2.04 (s, 3H); ^{13}C (125 MHz, $CDCl_3$) δ 168.5, 167.9, 142.7, 133.1, 121.7, 118.2, 109.5, 21.7, 18.2; IR (nujol) 3053, 2987, 2237, 1811, 1699, 1510, 1270 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 218 (M^+, 3), 176 (100), 134 (85), 116 (29); HRMS (EI) calcd for $C_{11}H_{10}N_2O_3$ 218.0691, found 218.0681.

2-Chloro-5-*N*,*O*-diacetylpyridine (5i)[12]

Liquid, TLC (30% ethyl acetate/hexane) R_f 0.32; ^1H NMR (300 MHz, $CDCl_3$) δ 8.49–8.48 (d, 1H, *J* = 2.6 Hz), 7.87–7.83 (dd, 1H, *J* = 2.6, 8.5 Hz), 7.40–7.37 (d, 1H, *J* = 8.5 Hz), 2.26 (s, 3H), 2.16 (s, 3H); ^{13}C (125 MHz, $CDCl_3$) δ 171.0, 167.8, 135.1, 133.4, 129.2, 128.7, 124.4, 21.1, 18.2; IR (nujol) 3063, 2983, 1810, 1703, 1612, 1425, 1266 cm^{-1}; GC-MS *m*/*z* (rel. intensity) 230 (1), 228 (M^+, 3), 188 (24), 186 (70), 146 (35), 144 (100), 126 (22); HRMS (EI) calcd for $C_9H_9ClN_2O_3$ 228.0302, found 228.0307.

ACKNOWLEDGMENTS

This work was supported by the Brain Korea 21 project and partly by Kwangwoon University in the year 2000.

REFERENCES

1. (a) Li, C.J.; Chan, T.H. *Organic Reactions in Aqueous Media*, Wiley-Interscience, New York, 1997; (b) Li, C.J. Tetrahedron **1996**, *52*, 5643.
2. (a) Chan, T.H.; Li, C.J. Tetrahedron Lett. **1991**, *32*, 7017; (b) Paquette, L.A.; Bennett, G.D.; Chhatriwalla. A.; Issac, M.B. J. Org. Chem. **1997**, *62*, 3370; (c) Bose, A.K.; Jayaraman, M.; Manhas, M.S. Tetrahedron Lett. **1997**, *38*, 709.
3. Kalyanam, N.; Rao, G.V. Tetrahedron Lett. **1993**, *34*, 1647.
4. Chan, T.H.; Li, C.J.; Lee, M.C.; Wei, Z.Y. Can. J. Chem. **1994**, *72*, 1181.
5. Chan, T.H.; Issac, M.B. J. Chem. Soc. Chem. Commun. **1995**, 1003.

MARCEL DEKKER, INC.
270 Madison Avenue, New York, New York 10016

6. Li, C.J.; Haberman, J.X. Tetrahedron Lett. **1997**, *38*, 4735.
7. (a) Moody, C.J.; Pitts, M.R. Synlett. **1998**, 1028; (b) Moody, C.J.; Pitts, M. Synlett, **1998**, 1029; (c) Moody, C.J.; Pitts, M.R. Synlett. **1999**, 1575; (d) Ranu, B.C.; Guchhait, S.K.; Sarkar, A. J. Chem. Soc. Chem. Commun. **1998**, 113; (e) Ranu, B.C.; Dutta, P.; Sarkar, A. J. Chem. Soc. Perkin Trans. 1, **1999**, 1139; (f) Reddy, G.V.; Rao, G.V.; Iyengar, D.S. Tetrahedron Lett. **1999**, *40*, 3937.
8. Several recent reviews include: a) Miller, J.A. Cancer Res. **1970**, *30*, 559; b) Miller, J.A.; Miller, E.C. EHP, Environ. Health Perspect. **1983**, *49*, 3; c) Kriek, E.; Westra, J.G. in *Chemical Carcinogens and DNA*, Grover, P.L. (Ed.), CRC Press, Boca Raton, FL, 1979; vol. *2*, pp. 1–28; d) Dalejka-Giganti, D. in *Chemistry and Biology of Hydroxamic Acids*, Kehl, H. (Ed.), Karger, Basel, 1982; pp. 150–159; e) Hathway, D.E.; Kolar, G.F. Chem. Soc. Rev. **1980**, *9*, 241.
9. Koul, A.K.; Bachhawat, J.M.; Ramegowda, N.S.; Prashad, B.; Mathur, N.K. Indian J. Chem. Educ. **1972**, *3*, 22.
10. Miller, J.A.; Wyatt, C.S.; Miller, E.C.; Hartmann, H.A. Cancer Res. **1961**, *21*, 1465.
11. a) Klemm, L.H.; Iversion, P.E.; Lund, H. Acta Chem. Scand. **1974**, *B28*, 593; b) Christensen, L.; Iversion, P.E. Acta Chem. Scand. **1979**, *B33*, 352; c) Wagenknecht, J.H.; Johnson, G.V. J. Electrochem. Soc. **1987**, *134*, 2754.
12. Kim, B.H.; Jun, Y.M.; Suh, S.W.; Baik, W.; Lee, B.M. J. Chem. Res. (S) **1998**, 46.
13. a) Russell, G.A.; Janzen, E.G. J. Am. Chem. Soc. **1962**, *84*, 4153; b) Russell, G.A.; Danen, W.C. J. Am. Chem. Soc. **1966**, *88*, 5663; c) Kornblum, N.; Michel, R.E.; Kerber, R.C. J. Am. Chem. Soc. **1966**, *88*, 5662.
14. Russell, A.; Geels, E.J. J. Am. Chem. Soc. **1965**, *87*, 122.
15. *p*-Dinitrobenzene and di-*tert*-butyl nitroxide are good radical anion and radical scavengers, respectively, see: (a) Russell, G.A.; Jawdosiuk, M.A.; Makosza, M. J. Am. Chem. Soc. **1979**, *101*, 2355; (b) Rosse, R.A. Acc. Chem. Res. **1982**, 164; (c) Hoffmann, A.K.; Feldman, M.; Gelblum, E.; Hodgson, G. J Am. Chem. Soc. **1964**, *86*, 639; (d) Bunnett, J.F. Acc. Chem. Res. **1978**, 413.

Received in Japan December 25, 2000