Chemoselective Conversion of Conjugated Nitroalkenes into Ketones by Sodium Borohydride–Hydrogen Peroxide: A New Synthesis of 4-Oxoalkanoic Acids, Dihydrojasmone and (±)-*exo*-Brevicomin

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Received 25 January 1994; revised 7 March 1994

A new, simple, cheap, and practical procedure for the direct transformation of α,β-unsaturated nitroalkenes into ketones has been realized by the $NaBH_4/H_2O_2$ system. By this method, other functional groups such as C–C double bonds, ketals or aromatic nitro groups were preserved. Application of this methodology to the preparation of 4-oxoalkanoic acids, dihydrojasmone, and (±)-*exo*-brevicomin is also reported.

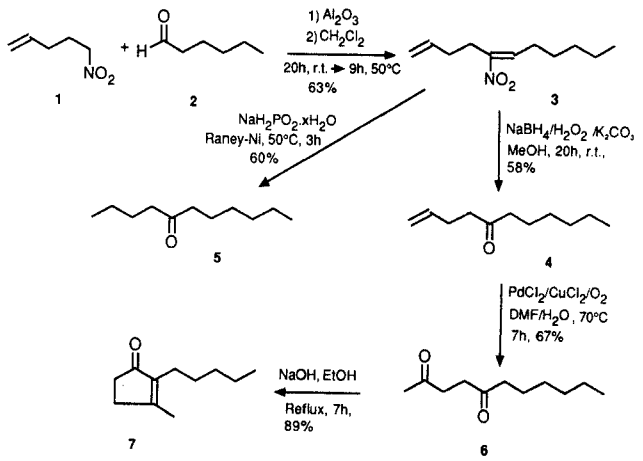
Nitroalkenes are powerful dienophiles in Diels–Alder reactions and readily undergo addition reactions with many different nucleophiles.[1] They are also used for conversion into a variety of functionalities, and the transformation of conjugated nitroalkenes into carbonyl compounds is particularly important. For this purpose various reagents have been used;[2-7] however, most of these procedures have some drawbacks, such as expensive reagents, harsh reaction conditions, or reagent instability.

During our studies on nitroalkenes, we found that sodium hypophosphite[3] was the most useful and practical reagent for the conversion of functionalized nitroalkenes into carbonyl derivatives.[8-9] Subsequently, in exploring the application of functionalized nitrolkenes in the synthesis of natural products,[10] we prepared nitroalkene **3** (Scheme 1) as the precursor for the synthesis of dihydrojasmone **7**, a naturally occurring substance widely used as a perfume ingredient,[11] and, during an attempt to obtain ketone **4** from nitroalkene **3**, using sodium hypophosphite,[3] instead of the expected enone **4**, we obtained the ketone **5**, in which the terminal C–C double bond had been reduced. To solve this problem we decided to develop a new method to convert nitroalkenes into their corresponding carbonyl derivatives, with the aim of preserving the C–C double bond in remote positions. We found that the $NaBH_4/H_2O_2$ combination gave good results for this purpose. Our method was effected by dissolving nitroalkene **3** (0.05 mol) in methanol, followed by the addition, at 0 °C, of 0.1 mol of sodium borohydride, and then, after 2 h, of 30% hydrogen peroxide and potassium carbonate (See experimental). After 20 h ketone **4** was obtained in 58% yield.

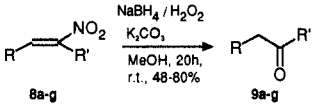
Subsequent oxidation of **4** by a Wacker process,[12] using O_2 and catalytic amounts of $PdCl_2$ and $CuCl_2$ gave the 2,5-undecadione **6** (67% yield); the cyclization of this latter compound yielded dihydrojasmone **7** (89%).

Using the same method we directly converted (Scheme 2) different nitroalkenes **8** into ketones **9** in satisfactory to good yields (48–80%). By this method C–C double bonds, ketal or aromatic nitro groups were preserved.

We found that it was possible to obtain (Scheme 3), starting from aldehyde **10** and 4-nitro ester **11**, via the nitroalkenes **12**, the 4-oxoalkanoic acids **13**, an important class of organic compounds used to prepare lactones,[13] β-lactam antibiotics.,[14] isoquinolines,[15] and lactone sex pheromones.[16-18] Accordingly, nitroaldol condensation of **10** with **11**, using alumina at room temperature, followed by addition of dichloromethane and warming to 50 °C, gave[19] (*E*)-nitroalkenes **12**, which, using sodium borohydride/hydrogen peroxide, were directly converted into 4-oxoalkanoic acids **13** in 60–70% yields.

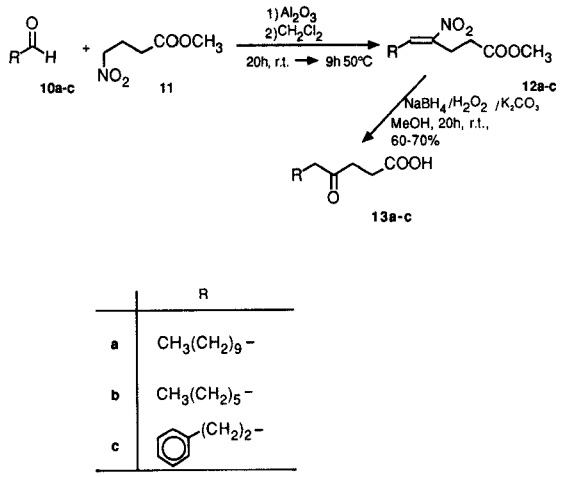


Scheme 1



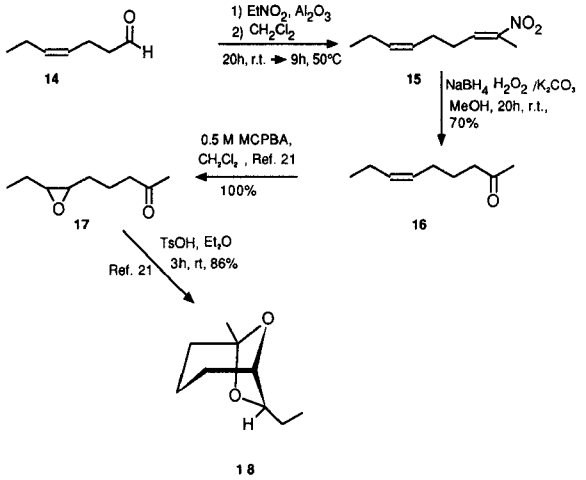
	R	R'
a	$-(CH_2)_4-$	
b	$-(CH_2)_{13}-$	
c	$-(CH_2)_2-$	$-(CH_2)_2$
d		$-(CH_2)_3CH_3$
e	NO_2	$-CH_3$
f	C_6H_5-	$-CH_3$
g	$CH_3(CH_2)_8-$	$-(CH_2)_3CH_3$

Scheme 2



Scheme 3

As a further application, our method was used to achieve a convenient, four-step synthesis of *exo*-brevicomin **(18)** (Scheme 4), the principal pheromone component of *Dentroctonus brevicomin*.[20] Thus, condensation of commercially available (*Z*)-4-heptenal **(14)** and nitroethane gave nitroalkene **15** (65% yield), which, on treatment with $NaBH_4/H_2O_2$, afforded enone **16** in 70% yield.



Scheme 4

Epoxidation of **16** with *m*-chloroperbenzoic acid afforded[21] the corresponding epoxy ketone **17** in quantitative yield, which with *p*-TsOH provided racemic *exo*-brevicomin **18** in 86% yield.

In summary, our method uses readily available reagents, and thus represents a practical alternative to existing procedures. This is especially important for the synthesis of natural products where preservation of the stereochemical purity of C–C double bonds is often crucial.

Conjugated nitroalkenes[19] **8**, 5-nitro-1-pentene[22] **(1)**, and methyl 4-nitrobutyrate[23] **(11)** were prepared as previously reported. (*Z*)-4-Heptanal **(14)** was commercially available (Alfa) or, alternatively, can be prepared, in good yield, according to the procedure of Stetter and Kuhlmann,[24] starting from commercial (*Z*)-3-hexen-1-ol. Hexanal **(2)** and aldehydes **10a–c** were commercially available (Aldrich).

The reactions were monitored by TLC and/or GC analyses, performed on a Carlo Erba Fractovap 4160 using a capillary column of duran glass (0.32 mm × 25 m), stationary phase OV1 (film thickness 0.4–0.45 nm). All ^{1}H NMR spectra were recorded in $CDCl_3$ at 200 MHz, on a Varian Gemini 200. Chemical shifts are expressed in ppm downfield from tetramethylsilane. IR spectra were recorded with a Perkin-Elmer 257 spectrophotometer. Mass spectra were determined on a Hewlett-Packard GC/MS 5988A. Elementary analyses were performed using a C, H, N Analyzer Model 185 from Hewlett-Packard.

(*Z*)-5-Nitro-1,5-undecanediene (3):
A solution of hexanal **(2)** (2.5 g, 25 mmol) and 5-nitro-1-pentene **(1)** (2.87 g, 25 mmol) was mechanically stirred for 10 min at 0°C, cooling with an ice-bath. After the addition of chromatographic basic alumina (activity I, 5 g) and stirring for 1 h at 0°C, the mixture was stored at r.t. for 20 h. CH_2Cl_2 (50 mL) was added, and the mixture was stirred and heated at 50°C for 9 h. The mixture was filtered and the alumina was washed with CH_2Cl_2 (3 × 20 mL). The organic layer was evaporated and purified by flash chromatography (EtOAc/cyclohexane, 1:9) to give 3.1 g (63%) of **3** as an oil.

IR (film): ν = 1505, 1635, 1660 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ = 0.9 (t, 3 H, J = 6.7 Hz, CH_3), 1.22–1.6 [m, 6 H, $(CH_2)_3$], 2.12–2.37 (m, 4 H), 2.65–2.75 [m, 2 H, $CH_2C(NO_2)$], 4.97–5.13 (m, 2 H, CH_2=CH), 5.7–5.91 (m, 1 H, CH=CH_2), 7.15 [t, 1 H, J = 7.9 Hz, CH=$C(NO_2)$]

5-Undecanone (5):
A suspension of Raney nickel (Fluka, 50% H_2O, 0.3 mL) was added in several portions to a solution of **3** (0.3 g, 1.52 mmol) and NaH_2PO_2 hydrate (1.5 g) in EtOH–aqueous acetate buffer, pH 5.0 (30 mL, 2:1). The reaction mixture was stirred at 50°C for 3 h,

Table. Ketones **9a–g** Prepared

Product[a]	Yield[b] (%)	bp/pressure (Torr) or mp (°C) (Lit. value)	IR (KBr) ν (cm^{-1})	^{1}H NMR ($CDCl_3$/TMS) δ, J (Hz)
9a	73	154/760 (155/760[34])		
9b	80	65–66 (64–66[34])		
9c	48	oil	1600, 1700	1.3 (s, 3H, CH_3), 1.86–2.03 [m, 4H, $(CH_2)_2$], 2.38–2.51 [m, 4H, $(CH_2)_2$], 2.63 (t, 2H, J= 7.5, CH_2CO), 3.83–3.89 [m, 4H, $O(CH_2)_2O$], 7.12–7.33 (m, 5H, ArH)
9d	69	oil	1705	0.88–0.98 (m, 3H, CH_3), 1.12–1.98 (m, 15H), 2.27 (d, 2H, J= 6.8, CH_2CO), 2.38 (t, 2H, J= 7.3, $COCH_2$)
9e	53	64–65 (62–63[35])	1525, 1600, 1710	2.23 (s, 3H, CH_3), 3.86 (s, 2H, CH_2), 7.8 (AA'BB', 4H, J= 8.8, ArH)
9f	50	oil	1600, 1710	2.21 (s, 3H, CH_3), 3.76 (s, 2H, CH_2), 7.29–7.62 (m, 9H, ArH)
9g	56	80/0.2	1710	0.91 (t, 6H, J= 7.1, 2 × CH_3), 1.2–1.6 (m, 20H), 2.39 (t, 4H, J= 7.4, CH_2COCH_2)

[a] Compounds **3**, **9a–g**, **12a–c** and **15** gave C, H (and N where appropriate) ±0.26%; except **9g**, H −3.46%.
[b] Yield of pure, isolated product.

the catalyst was filtered off, water added (40 mL), and the solution was extracted with Et_2O (3 × 10 mL). Evaporation of the solvent and purification of the crude product by flash chromatography afforded 0.153 g (60%) of **5**. Bp 100°C/0.4 Torr (Kugelrohr) (Lit.,[25] 80°C/2 Torr).

IR (film): $\nu = 1700\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.87$ (t, 3H, $J = 6.9$ Hz, CH_3), 0.9 (t, 3H, $J = 7.2$ Hz, CH_3), 1.2–1.65 (m, 12H, $6 \times CH_2$), 2.4 (t, 4H, $J = 7.4$ Hz, CH_2COCH_2).

MS: *m/z* (%) = 170 (M^+, 7), 141 (9), 128 (11), 114 (11), 113 (98), 85 (99), 71 (33), 58 (91), 57 (100), 54 (70).

1-Undecen-5-one (4):
Compound **3** (2.36 g, 12 mmol) was dissolved in MeOH (70 mL), then, after cooling with an ice-bath, $NaBH_4$ (0.924 g, 24 mmol) was added with stirring. After 2 h at r.t., the solution was cooled at 0°C and 32 mL of aq 30% H_2O_2 and 13.2 g of K_2CO_3 were added and stirring was continued for 18 h at r.t. The solution was then acidified with 2N HCl and extracted with CH_2Cl_2 (3 × 35 mL). The organic layer was washed with water (40 mL), dried (Na_2SO_4), evaporated, and purified by flash chromatography (EtOAc/cyclohexane, 2:8) to give 1.17 g (58%) of pure **4**. Bp 95°C/8 Torr (Lit.,[26] 79–81°C/0.2 Torr).

IR (film): $\nu = 1640$, $1715\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.88$ (t, 3H, $J = 6.5$ Hz, CH_3), 1.2–1.4 [m, 6H, $(CH_2)_3$], 1.53–1.68 (m, 2H), 2.25–2.52 [m, 6H, $(CH_2)_2COCH_2$], 4.91–5.11 (m, 2H, CH_2=CH), 5.68–5.93 (ddt, 1H, $J = 16.8$ and 6.3 Hz, CH=CH_2).

Undecane-2,5-dione (6):
A solution of $PdCl_2$ (15 mg) and $CuCl_2$ (130 mg) in DMF (40 mL) and water (40 mL) was prepared. To this solution, a solution of **4** (1 g, 5.95 mmol) in DMF (20 mL) and water (20 mL) was added slowly over 1.5 h at 70°C with passage of molecular oxygen. After the addition, the mixture was stirred for 5.5 h at the same temperature. The solution was extracted with $CHCl_3$ (3 × 40 mL), the organic layer dried (Na_2SO_4), evaporated, and the crude product was purified by flash chromatography (EtOAc/cyclohexane, 1:9) giving 0.73 g (67%) of pure **6**. Mp 33–34°C (Lit.,[27] bp 141°C/14 Torr).

IR (film): $\nu = 1705\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.85$ (t, 3H, $J = 6$ Hz, CH_3), 1.15–1.75 [m, 8, $(CH_2)_4$], 2.18 (s, 3H, CH_3), 2.45 (t, 2H, $J = 7.5$ Hz, $COCH_2$), 2.68 [m, 4H, $CO(CH_2)_2CO$].

Dihydrojasmone (7):
Following a previously reported procedure,[28] intramolecular aldol condensation of **6** (0.7 g, 3.8 mmol) afforded 0.56 g (89%) of the pure **7**. Bp 85°C/2 Torr. Analytical data were in agreement with those previously reported.[29]

Conversion of Nitroalkene 8 into Ketones 9; General Procedure:
Following the reported experimental conditions for the conversion of **3** into **4**, nitroalkenes **8** were transformed into ketones **9**.

α,β-Unsaturated Nitroalkenes 12; General Procedure:
Starting from 0.02 mol of aldehydes **10** and 0.02 mol of the nitro ester **11**, and following the same procedure as for **3**, the nitroalkenes **12** were prepared.

(E)-Methyl 4-Nitro-4-pentadecenoate **(12a)**:
Yield 3.58 g (60%); oil.

IR (film): $\nu = 1510$, $1730\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.88$ (t, 3H, $J = 7.2$ Hz, CH_3), 1.18–1.65 [m, 16H, $(CH_2)_8$], 2.2–2.35 (m, 2H, CH_2CH=C), 2.58 (t, 2H, $J = 7.5$ Hz, CH_2CO), 2.9 [t, 2H, $J = 7.5$ Hz, $C(NO_2)CH_2$], 3.68 (s, 3H, OCH_3), 7.18 [t, 1H, $J = 7.9$ Hz, CH=$C(NO_2)$].

(E)-Methyl 4-Nitro-4-undecenoate **(12b)**:
Yield 3.45 g (71%); oil.

IR (film): $\nu = 1510$, $1728\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.88$ (t, 3H, $J = 7.3$ Hz, CH_3), 1.18–1.66 [m, 8H, $(CH_2)_4$], 2.2–2.34 (m, 2H, CH_2CH=C), 2.58 (t, 2H, $J = 7.5$ Hz, CH_2CO), 2.9 [t, 2H, $J = 7.5$ Hz, $C(NO_2)CH_2$], 3.68 [s, 3H, OCH_3), 7.17 [t, 1H, $J = 8$ Hz, CH=$C(NO_2)$].

(E)-Methyl 4-Nitro-7-phenyl-4-heptenoate **(12c)**:
Yield 3.15 g (60%); oil.

IR (film): $\nu = 1505$, $1720\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 2.38$ (t, 2H, $J = 7.4$ Hz, $ArCH_2$), 2.63 (m, 2H, $ArCH_2CH_2$), 2.8 [m, 4H, $(CH_2)_2CO$], 3.68 (s, 3H, OCH_3), 7.18–7.38 [m, 6H, ArH and CH=$C(NO_2)$].

4-Oxoalkanoic Acids 13; General Procedure:
4-Oxoalkanoic acids **13** were prepared, from 0.01 mol of **12**, following the procedure reported for the conversion of the nitroalkene **3** into **4**, except that the purification of the product was effected by extraction of **13** from the CH_2Cl_2 solution with sat. aq $NaHCO_3$ (3 × 40 mL) acidification (2N HCl) extraction with Et_2O (3 × 50 mL). Evaporation of the solvent gave pure **13**.

4-Oxopentadecanoic Acid **(13a)**:
Yield 1.79 g (70%); mp 90–92°C (Lit.[30] 92.6°C).

IR (KBr): $\nu = 1700$, $3400\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.9$ (t, 3H, $J = 7.3$ Hz, CH_3), 1.18–1.72 [m, 18H, $(CH_2)_9$)], 2.35 (t, 2H, $J = 7.4$ Hz, CH_2CO), 2.47 (t, 2H, $J = 7.4$ Hz, CH_2CO_2H), 2.69 (t, 2H, $J = 6.9$ Hz, $COCH_2$).

4-Oxoundecanoic Acid **(13b)**:
Yield 1.36 g (68%); mp 76–78°C (Lit.,[31] 77–79°C).

IR (KBr): $\nu = 1710$, $3400\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.88$ (t, 3H, $J = 7.2$ Hz, CH_3), 1.2–1.7 [m, 12H, $CH_2)_6$], 2.45 (t, 2H, $J = 7.4$ Hz, CH_2CO), 2.58–2.8 [m, 4H, $CO(CH_2)_2CO$].

4-Oxo-7-phenylheptanoic Acid **(13c)**:
Yield 1.3 g (60%); mp 91–92°C (Lit.,[32] mp 93–94°C).

IR (KBr): $\nu = 1695$, $3400\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 1.82$–2.05 (m, 2H, $ArCH_2$), 2.35–2.5 (m, 2H, CH_2CO), 2.58–2.78 [m, 4H, $CO(CH_2)_2CO$], 7.1–7.35 (m, 5H, ArH).

(2*E*,6*Z*)-2-Nitro-2,6-nonadiene (15):
Compound **15** (1.65 g, 65%) was prepared starting from 0.015 mol of (*Z*)-4-heptenal **(14)** and 0.015 mol of nitroethane, following the procedure used for the synthesis of nitroalkene **3**. Bp 136°C/0.55 Torr (Kugelrohr).

IR (film): $\nu = 1520$, $1670\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.88$ (t, 3H, $J = 7.6$ Hz, CH_3), 1.9–2.1 (m, 2H, CH_3CH_2), 2.18 (s, 3H, CH_3), 2.15–2.3 [m, 4H, $(CH_2)_2$], 5.22–5.32 (m, 1H, CH=CH), 5.4–5.5 (m, 1H, CH=CH), 7.12 [t, 1H, $J = 7.4$ Hz, CH=$C(NO_2)$].

(*Z*)-Non-6-en-2-one (16):
Starting from 1.5 g (8.87 mmol) of **15** and following the same procedure used in the conversion of nitroalkene **3** into ketone **4**, 0.87 g (70%) of the pure **16** were obtained. Bp 150°C/45 Torr (Kugelrohr) (Lit.,[33] 94–96°C/20 Torr).

IR (film): $\nu = 1708\ cm^{-1}$.

^1H NMR ($CDCl_3$): $\delta = 0.95$ (t, 3H, $J = 7.4$ Hz, CH_3), 1.55–1.7 (m, 2H, CH_3CH_2), 1.92–2.08 [m, 4H, $(CH_2)_2$], 2.12 (s, 3H, CH_3CO), 2.37–2.45 (m, 2H, CH_2CO), 5.2–5.5 (m, 2H, CH=CH).

MS: *m/z* (%) = 140 (M^+, 31), 125 (37), 111 (57), 97 (43), 82 (93), 71 (50), 67 (100).

(±)-*exo*-Brevicomin (18):
(±)-*exo*-Brevicomin **(18)** (0.77 g, 86%) was obtained from the ketone **16** (0.8 g, 5.7 mmol) via epoxidation with *m*-chloroperbenzoic acid in CH_2Cl_2, by a known method,[21] and treatment of the obtained epoxide **17** with *p*-TsOH in Et_2O; bp 105°C/17 Torr (Kugelrohr) (Lit.,[33] 86–88°C/50 Torr).

The financial assistance of Progetto Chimica Fine II (C.N.R.-Italy) is acknowledged.

(1) (a) Barrett, A.G.M.; Graboski, G.G. *Chem. Rev.* **1986**, *86*, 751.
(b) Kabalka, G.W.; Varma, R.S. *Org. Prep. Proc. Int.* **1987**, *19*, 283.
(c) Barrett, A.G.M. *Chem. Soc. Rev.* **1991**, *20*, 95.
(2) Varma, R.S.; Varma, M.; Kabalka, G.W. *Tetrahedron Lett.* **1985**, *26*, 3777.
(3) Monti, D.; Grammatica, P.; Speranza, G.; Manitto, P. *Tetrahedron Lett.* **1983**, *24*, 417.
(4) Torii, S.; Tanaka, H.; Katoch, T. *Chem. Lett.* **1983**, 607.
(5) Mourad, M.S.; Varma, R.S.; Kabalka, G.W. *Synthesis* **1985**, 654.
(6) Palomo, C.; Aizpurua, J.M.; Cossio, F.P.; Garcia, J.M.; Lopez, M.C.; Oiarbide, M. *J. Org. Chem.* **1990**, *55*, 2070.
(7) Hwu, J.R.; Gilbert, B.A. *J. Am. Chem. Soc.* **1991**, *113*, 5917.
(8) Ballini, R.; Bartoli, G. *Synthesis* **1993**, 965.
(9) Ballini, R.; Bosica, G. *J. Chem. Research (S)* **1993**, 435.
(10) Ballini, R.; Petrini, M. *J. Chem. Soc., Perkin Trans 1* **1992**, 3159.
(11) Saul, E.L. *Amer. Perfume Essent Oil Record* **1943**, *45*, 27.
(12) For a review see: Huttel, R. *Synthesis* **1970**, 225.
(13) Edwards, M.L.; Bambury, R.E.; Ritter, H.W. *J. Med. Chem.* **1976**, *19*, 330.
(14) Kigasawa, K.; Hiiragi, M.; Ishimaru, H.; Haga, S. Japan Patent 75 111098 (1975), Grelan Pharmaceutical Co.; *Chem. Abstr.* **1976**, *84*, 135496.
(15) Naoshima, Y.; Ozawa, H.; Xondo, H.; Hayashi, S. *Agric. Biol. Chem.* **1983**, *47*, 1431.
(16) Robach, M.C. US Patent 4356204 (1982), Monsanto Co.; *Chem. Abstr.* **1983**, *98*, 15734.
(17) Mayr, H.H.; Prosoly, E.; Rittmeyer, G.Z. *Pflanzenernahr Bodenkd* **1977**, *140*, 463; *Chem. Abstr.* **1977**, *87*, 113000.
(18) Reynolds, R.S. Neth. Appl. 6516702 (1966), Tobacco Co.; *Chem. Abstr.* **1967**, *66*, 53041.
(19) Ballini, R.; Castagnani, R.; Petrini, M. *J. Org. Chem.* **1992**, *57*, 2160.
(20) Silverstein, R.M.; Brownlee, R.G.; Belas, T.E.; Wood, D.L.; Browne, L.E. *Science* **1968**, *159*, 889.
(21) Yoshi, N.N.; Mandapur, V.R.; Chadha, M. *J. Chem. Soc., Perkin Trans 1* **1983**, 2963.
(22) Ballini, R. *Synthesis* **1993**, 687.
(23) Chasar, D.W. *Synthesis* **1982**, 841.
(24) Stetter, H.; Kuhlmann, H. *Synthesis* **1975**, 379.
(25) Yamamoto, Y.; Yamamoto, S.; Yatagai, H.; Ishihara, Y.; Maruyama, K. *J. Org. Chem.* **1982**, *47*, 119.
(26) Wijkens, P.; Vermeer, P. *J. Organomet. Chem.* **1986**, *301*, 247.
(27) Hunsdiecker, H. *Chem. Ber.* **1942**, *75*, 447.
(28) Buchi, G.; Wuest, H. *J. Org. Chem.* **1966**, *31*, 977.
(29) Pecunioso, A.; Menicagli, R. *J. Org. Chem.* **1989**, *54*, 2391.
(30) Clutterbuck, P.W.; Raistrick, H.; Rintul, L. *Trans. Roy. Soc. (London)* **1931**, *B220*, 301; *Chem. Abstr.* **1932**, *26*, 2487.
(31) Jelinkova, E.; Jelinkova, H. *Collection Czechoslov. Chem. Commun.* **1959**, *24*, 3341; *Chem. Abstr.* **1960**, *50*, 4376c.
(32) Hayashi, Y.; Sakan, T. *Nippon Kagaku Zasshi* **1960**, *81*, 1894; *Chem. Abstr.* **1962**, *56*, 2396e.
(33) Joshi, N.N.; Mandapur, V.R.; Chadha, M.S. *J. Chem. Soc., Perkin Trans 1* **1983**, 2963.
(34) *Handbook of Chemistry and Physics, 57th ed*; (1976/77), CRC Press: Cleveland, Ohio.
(35) Overberger, C.G.; Bileth, H. *J. Am. Chem. Soc.* **1951**, *73*, 4880.