Acta Chemica Scandinavica, 1995: 49: 423–427
Printed in Belgium – all rights reserved

ACTA
CHEMICA SCANDINAVICA
ISSN 0904–213X

A Concise Synthetic Route to Pure Isomers of the Antifungal Agents (*E*)- and (*Z*)-1,2-Diaryl-3-(1-imidazolyl)-1-propenes

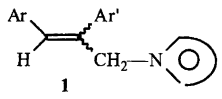
Jon K. F. Geirsson,[a,†] Birgir Ö. Gudmundsson,[a] Jonina F. Johannesdóttir,[a] Jon T. Njardarson[a] and Vilhjalmur G. Skulason[b]

[a]Science Institute, University of Iceland, Dunhaga 3, IS-107 Reykjavik, Iceland and [b]University of Iceland, Department of Pharmacy, Sudurgata, IS-107 Reykjavik, Iceland

Geirsson, J. K. F., Gudmundsson, B. Ö., Johannesdóttir, J. F., Njardarson, J. T. and Skulason, V. G., 1995. A Concise Synthetic Route to Pure Isomers of the Antifungal Agents (*E*)- and (*Z*)-1,2-diaryl-3-(1-imidazolyl)-1-propenes. – Acta Chem. Scand. 49: 423–427 © Acta Chemica Scandinavica 1995.

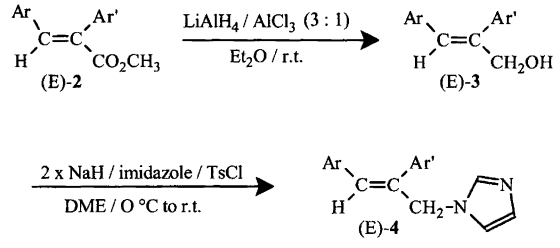
An efficient and relatively simple synthetic sequence is detailed for the preparation of 1,2-diaryl-3-(1-imidazolyl)-1-propenes **4**, a class of biologically active compounds. The method gives access to pure geometrical isomers. Spectral characteristics, which enable the distinction between *E* and *Z* isomers, are discussed.

We have been involved in an investigation aimed at the synthesis of compounds with the general structure **1**, and the examination of these for antibacterial activity. All these compounds contain 1,2-diarylpropenyl moieties bonded to the nitrogen atom of different heterocycles, preferably aromatic rings. Compounds **1** with imidazole as the aromatic heterocycle are known to exhibit biological activity towards fungi and bacteria, particularly derivatives with chloro-substituted aromatic rings. Such compounds, in concentrations between 10 and 100 ppm, completely inhibited the growth of *Staphylococcus aureus*, *Trichophyton mentagraphytes* and *Candida albicans*, and they also prevented lethal effects of *Candida albicans* infection in mice.[1] This paper describes the preparation of such compounds via a procedure which allows some structural variation with regard to the heterocycle. The synthetic route produces pure geometric isomers of the products, which is an advantage over a previously published procedure.[1]



1

The synthetic pathway is depicted in Scheme 1; it is valid for both *E* and *Z* isomers. Scheme 1 reveals that the employment of readily available pure geometric isomers of methyl α-arylcinnamates **2** as starting materials[2] affords pure isomers of (*E*)- and (*Z*)-1,2-diaryl-3-(1-imidazolyl)-1-propenes **4**.



2 - 4	Ar	Ar'
a	C_6H_5	C_6H_5
b	C_6H_5	4 - ClC_6H_4
c	2,4 - $Cl_2C_6H_3$	C_6H_5

Scheme 1.

Results and discussion

As outlined in Scheme 1, the synthesis of **4** was achieved by first reducing the α,β-unsaturated ester **2** to afford the allylic alcohol **3**. Subsequently the hydroxy functionality of **3** was converted into a better leaving group, which facilitated the introduction of the imidazolyl moiety.

(i) Reduction of **2**. The reduction of (*E*)-**2a** to yield (*E*)-**3a** without affecting the carbon–carbon double bond was readily achieved with lithium aluminium hydride. However, when the reactants were stirred for 3–4 h at ambient

† To whom correspondence should be addressed.

temperature, the crude product contained appreciable amounts of impurities as evidenced by the ^{1}H NMR spectrum. Analytical TLC showed at least four side-products, all of which could be suppressed either by shortening the reaction time (ambient temperature/15 min) or by lowering the reaction temperature (5 °C/25 min). Unfortunately, in the lithium aluminium hydride reduction of (*Z*)-**2a** the undesired side-products were much more pronounced and defied complete suppression.[3,4] It has been reported that the treatment of α,β-unsaturated esters with an ethereal suspension of lithium aluminium hydride–aluminium trichloride (3:1) reduces the ester group without affecting the carbon–carbon unsaturation.[5] Consistent with this, when an ethereal solution of (*Z*)-**2a** was stirred at ambient temperature in the presence of this reducing system, the ester underwent a facile reduction affording the allylic alcohol (*Z*)-**3a** in 74% yield after recrystallization.

The remaining olefinic esters **2**, both *E* and *Z* isomers, were reduced by this method, producing the allylic alcohols **3** in yields ranging from 70 to 90% after recrystallization.

(ii) Conversion of **3** *into* **4**. A logical first step in a synthetic route from **3** to **4** would be to convert the hydroxy group of **3** into a better leaving group. However, it can be envisaged that replacing this hydroxy group by a good leaving group leaves us with compounds which are probably prone to undergo allylic rearrangement due to the relative ease with which they form carbocations. This notion found support in a preliminary effort to synthesize the corresponding allyl bromides[6] and allyl chlorides[7] from **3**. With this in mind, and in order to optimize the total yield, we developed an expedient procedure which entails the direct transformation of **3** into **4** without isolating an intermediately formed tosylate. It proved necessary to remove the hydroxy proton of **3** prior to it reacting with tosyl chloride (TsCl), and using a suspension of sodium hydride in 1,2-dimethoxyethane (DME) as the deprotonation agent, we established that the reaction solution had to contain 2 mol equiv. of sodium hydride in order for the conversion of **3** into **4** to occur. When a solution of **3** was added to a suspension of sodium hydride, no discernible evolution of hydrogen occurred[8] and, similarly, stirring a DME solution of **3**, TsCl, and sodium hydride, at both ambient and elevated temperatures, left the starting alcohol unchanged (TLC). Adding imidazole to this solution of **3**, TsCl, and sodium hydride engendered a vigorous evolution of hydrogen, but the formation of the desired product **4** was detected *only* if 2 mol equiv. of sodium hydride were present. The presence of only 1 mol equiv. of sodium hydride led to no formation of **4** (TLC), but upon addition of another mol equiv. of sodium hydride, a spot representing **4** emerged on TLC. The yield of **4** was seemingly insensitive to the order in which the reactants were added to the reaction flask. However, adding TsCl as the last component had the advantage of suppressing the otherwise occasionally observed formation of 1-(*p*-tolylsulfonyl)imidazole.[9,10] Following this procedure, the conversion of **3** into **4** was achieved in yields of 70–90%, after purification on a short silica-gel column and recrystallization.

(iii) Spectral characteristics of **3** *and* **4**. Structures of products **3** and **4** were established on the basis of their spectral properties (IR, ^{1}H NMR, ^{13}C NMR). Table 1 summarizes distinctive chemical shift values of compounds **2**, **3** and **4**. The configuration assignments originate from the ^{1}H NMR chemical shifts of the olefinic protons of the esters.[2]

The pertinent ^{1}H NMR values in Table 1 reveal that the most pronounced difference in olefinic chemical shifts for each pair of *E* and *Z* isomers was apparent for the esters **2**. In (*E*)-**2** the β-proton is subjected to the anisotropy of the carbonyl group, causing a downfield shift of its signal relative to that of the *Z* isomer. After reduction of the carbonyl functionality to a methylene group, the *E* isomer olefinic signals experienced an upfield shift larger than 1 ppm on going from (*E*)-**2** to (*E*)-**3**/**4**. Also noteworthy are two distinctive features in the NMR spectra of compounds **3** and **4**, both concerning the allylic position and seemingly characteristic for each isomer series. Firstly, in the ^{1}H NMR spectra of all **3** and **4** prepared, the allylic protons of the *E* isomers appeared as doublets with a small allylic coupling constant, $J_{allyl} = 1.1–1.6$ Hz (Table 1). Such an allylic coupling was not observed within the *Z* isomer series. Secondly, the ^{13}C NMR spectra revealed a significant difference between the allylic carbon chemical shifts of (*E*)-**3**/**4** and those of (*Z*)-**3**/**4**. These values have been incorporated into Table 1 and they exhibit noticeable consistency.[11] We thus conclude that the NMR spectra of all pairs of *E* and *Z* isomers of **2**, **3** and **4** differ sufficiently for an unambiguous configuration assignment to be made.

Finally, we make a note on an alternative method for the preparation of (*E*)-**4**. The Perkin condensation of aryl-

Table 1. Selected NMR data of compounds **2**, **3** and **4**.

	^{1}H NMR (δ)					^{13}C NMR (δ)	
	H–C=C		C=C–C*H*$_2$			C=C–*C*H$_2$	
Product	*E*	*Z*	*E*	(J_{allyl}/Hz)[a]	*Z*	*E*	*Z*
2a	7.87	7.10					
2b	7.88	7.03					
2c	7.97	7.16					
3a	6.71	7.01	4.46	(1.43)	4.72	68.4	60.2
3b	6.70	6.97	4.41	(1.30)	4.66	68.2	60.2
3c	6.81	6.94	4.53	(1.58)	4.52	67.5	59.8
4a	6.48	7.14	4.82	(1.23)	5.08	55.2	45.9
4b	6.54	7.14	4.82	(1.18)	5.09	55.1	46.0
4c	6.60	7.03	4.92	(1.14)	4.97	54.1	45.9

[a] A small coupling constant was observed only for the *E* isomers.

acetic acids with benzaldehydes produces α-arylcinnamic acids in modest to good yields.[12,13] The thus-formed carboxylic acids can be converted into allylic alcohols **3** by reduction with lithium aluminium hydride.[14] This reaction sequence afforded compounds (*E*)-**4a–c** in acceptable total yields, the reduction to **3** being the most difficult step. One limitation of this procedure is that the Perkin reaction forms preferentially the *E*-configurated carboxylic acids, confining this approach to the preparation of the *E* series of isomers; only the procedure described here gives access to the *Z* series of isomers.

In order to corroborate further the possibility of distinguishing between the geometrical isomers at the stage of the final products, we decided to employ the Perkin reaction sequence to prepare two additional compounds **4**, for which we then had only one isomer available. The first compound was 1-phenyl-2-(4-nitrophenyl)-3-(1-imidazolyl)-1-propene (**4**: Ar = phenyl and Ar′ = 4-nitrophenyl) and its ^{13}C NMR spectrum exhibited a signal for the allylic carbon at δ = 54.4. The allylic protons appeared in the ^{1}H NMR spectrum as a doublet at δ = 4.89 with J_{allyl} = 1.04 Hz. The latter compound was 1-phenyl-2-(4-methoxyphenyl)-3-(1-imidazolyl)-1-propene (**4**: Ar = phenyl and Ar′ = 4-methoxyphenyl). The ^{13}C NMR spectrum for this latter compound showed a signal for the allylic carbon at δ = 55.1, and the allylic protons appeared in the ^{1}H NMR spectrum as a doublet at δ = 4.78 with J_{allyl} = 1.06 Hz. As expected, both compounds displayed signals characteristic of the *E* isomers.

In summary, we have disclosed our approach to the preparation of the biologically active compounds 1,2-diaryl-3-(1-imidazolyl)-1-propenes **4**. The simplicity of the procedures involved and the generally high yields support the reaction sequence as a solution to the general preparation of isomerically pure **1**.

Experimental

^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC 250P spectrometer. Chemical shifts are reported in δ values relative to $CHCl_3$. Multiplicities in the ^{13}C NMR spectra were determined by means of DEPT experiments. IR spectra were obtained on a Perkin-Elmer Model 283 spectrophotometer. Elemental analyses were carried out at the Schwarzkopf Microanalytical Laboratory, New York, USA, and on a Model 1106 Carlo Erba microanalyzer at the University of Iceland. Melting points (m.p.) were obtained on a Büchi 520 melting point apparatus and are uncorrected. Analytical TLC was performed by using 0.25 mm coated silica gel plates (Merck) with F-254 indicator. Visualization was accomplished with UV light.

General procedure for the preparation of 2,3-diaryl-2-propen-1-ols **3a–c**. The glassware was thoroughly dried prior to use, and the reaction was run under nitrogen. $AlCl_3$ (0.98 g, 7.3 mmol) was carefully added in small portions to an ice-cooled, stirred suspension of $LiAlH_4$ (0.83 g, 22 mmol) in 30 ml of dry ether. This mixture was stirred for 15 min in an ice–water bath and then for further 15 min at ambient temperature. An ethereal solution of the ester **2** (22 mmol) was then added dropwise over a period of 15 min and the resulting mixture was stirred at ambient temperature until TLC (CH_2Cl_2) showed the absence of **2**. Water was carefully added to the cooled solution until gas evolution had ceased, the ether was decanted off and the residue washed once with ether (2 ml). The combined organic phases were washed with water, dried over $MgSO_4$, and evaporated to obtain the crude products as colorless oils which upon crystallization from hexane–Et_2O afforded the pure products as white crystals.

*(E)-2,3-Diphenyl-2-propen-1-ol [(E)-**3a**].* Yield 3.9 g (85%), m.p. 70–72°C. IR (KBr): 3340, 1600 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 2.04 (1 H, br s, OH), 4.46 (2 H, d, *J* 1.43 Hz, CH_2), 6.71 (1 H, t, *J* 1.43 Hz, H–C = C), 7.00–7.36 (10 H, m, Ar–H). ^{13}C NMR ($CDCl_3$): δ 68.4 (*C*H_2), 126.3, 126.7, 127.5, 127.9, 128.69, 128.73, 129.2, 136.4, 138.5, 141.4.

*(Z)-2,3-Diphenyl-2-propen-1-ol [(Z)-**3a**].* Yield 3.4 g (74%), m.p. 75–76°C. IR (KBr): 3260, 1600 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.80 (1 H, br s, OH), 4.72 (2 H, s, CH_2), 7.01 (1 H, s, H–C = C), 7.26–7.64 (10 H, m, Ar–H). ^{13}C NMR ($CDCl_3$): δ 60.2 (*C*H_2), 126.5, 127.3, 127.6, 128.3, 128.6, 128.9, 131.2, 136.8, 140.0, 140.6.

*(E)-2-(4-Chlorophenyl)-3-phenyl-2-propen-1-ol [(E)-**3b**].* Yield 4.3 g (80%), m.p. 79–80°C. IR (KBr): 3240, 1595 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 2.05 (1 H, br s, OH), 4.41 (2 H, d, *J* 1.31 Hz, CH_2), 6.70 (1 H, t, *J* 1.31 Hz, H–C = C), 6.97–7.31 (9 H, m, Ar–H). ^{13}C NMR ($CDCl_3$): δ 68.2 (*C*H_2), 127.0, 127.3, 128.0, 128.9, 129.1, 130.1, 133.3, 136.0, 136.9, 140.1.

*(Z)-2-(4-Chlorophenyl)-3-phenyl-2-propen-1-ol [(Z)-**3b**].* Yield 4.9 g (90%), m.p. 77–78°C. IR (KBr): 3360, 1495 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.84 (1 H, br s, OH), 4.66 (2 H, s, CH_2), 6.97 (1 H, s, H–C = C), 7.32–7.54 (9 H, m, Ar–H). ^{13}C NMR ($CDCl_3$): δ 60.2 (*C*H_2), 127.6, 127.9, 128.5, 128.8, 129.0, 131.7, 133.5, 136.6, 139.0, 139.2.

*(E)-2-Phenyl-3-(2,4-dichlorophenyl)-2-propen-1-ol [(E)-**3c**].* Yield 4.5 g (73%), m.p. 79–81°C. IR (KBr): 3330, 1580 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.57 (1 H, br s, OH), 4.53 (2 H, d, *J* 1.58 Hz, CH_2), 6.81 (1 H, t, *J* 1.58 Hz, H–C = C), 6.68–7.35 (8 H, m, Ar–H). A spectrum run in Me_2CO-d_6 enabled a better analysis of the vinyl proton. ^{13}C NMR ($CDCl_3$): δ 67.5 (*C*H_2), 122.2, 126.4, 127.8, 128.63, 128.69, 129.0, 131.8, 132.8, 133.8, 134.6, 137.3, 144.2.

*(Z)-2-Phenyl-3-(2,4-dichlorophenyl)-2-propen-1-ol [(Z)-**3c**].* Yield 4.1 g (67%), m.p. 76–77°C. IR (KBr): 3350, 1585 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 2.38 (1 H, br s, OH),

4.52 (2 H, s, CH_2), 6.94 (1 H, s, H–C = C), 7.23–7.62 (8 H, m, Ar–H). ^{13}C NMR ($CDCl_3$): δ 59.8 (*C*H_2), 126.6, 126.72, 126.78, 128.0, 128.6, 129.1, 131.2, 133.64, 133.66, 134.6, 139.6, 141.7.

General procedure for the preparation of 1,2-diaryl-3-(1-imidazolyl)-1-propenes **4a–c**. The glassware was thoroughly dried prior to use, and the reaction was run under nitrogen. Imidazole (0.095 g, 1.39 mmol), dissolved in 5 ml of DME, was carefully added to a stirred suspension of NaH (0.07 g, 2.9 mmol) in DME (10 ml) at room temperature. After the evolution of hydrogen had ceased, the allylic alcohol **3** (1.33 mmol), dissolved in 5 ml of DME, was slowly added and the resulting mixture was cooled in an ice–water bath. A solution of TsCl (0.27 g, 1.39 mmol) in 5 ml of DME was now added at such a rate as to maintain a slow and steady gas evolution. After stirring the resulting mixture for 30 min in an ice–water bath, stirring was continued at ambient temperature. The reaction was conveniently monitored by TLC (silica gel; $CHCl_3$–MeOH 99:1). Unchanged NaH was carefully destroyed with water, most of the DME was evaporated off and the residue was partitioned between water and $CHCl_3$. The organic phase was separated, dried over $MgSO_4$, filtered and evaporated to leave a pale yellow oil that was purified by chromatography (silica gel, elution with $CHCl_3$–MeOH 9:1). Recrystallization from hexane–Et_2O or hexane–DME afforded the pure products.

*(E)-1,2-Diphenyl-3-(1-imidazolyl)-1-propene [(E)-***4a***]*. Yield 0.31 g (90%), m.p. 117–118°C (hexane–Et_2O). Anal. $C_{18}H_{16}N_2$: C, H, N. IR (KBr): 1600 cm^{-1}. ^1H NMR ($CDCl_3$): δ 4.82 (2 H, d, *J* 1.23 Hz, CH_2), 6.48 (1 H, t, *J* 1.23 Hz, H–C = C), 6.90–7.26 (12 H, m), 7.40 (1 H, br s). ^{13}C NMR ($CDCl_3$): δ 55.2 (*C*H_2), 119.1, 127.3, 127.8, 127.9, 128.2, 128.9, 129.1, 129.4, 129.6, 135.4, 137.3, 137.4, 137.6.

*(Z)-1,2-Diphenyl-3-(1-imidazolyl)-1-propene [(Z)-***4a***]*. Yield 0.26 g (75%), m.p. 104–105°C (hexane–Et_2O). Anal. $C_{18}H_{16}N_2$: C, H, N. IR (KBr): 1600 cm^{-1}. ^1H NMR ($CDCl_3$): δ 5.08 (2 H, s, CH_2), 6.83 (1 H, br s), 6.96 (1 H, br s), 7.14 (1 H, s, H–C = C), 7.24–7.42 (11 H, m). ^{13}C NMR ($CDCl_3$): δ 45.9 (*C*H_2), 118.6, 126.2, 127.8, 128.0, 128.4, 128.6, 129.2, 133.6, 135.3, 136.1, 136.8, 139.6.

*(E)-1-Phenyl-2-(4-chlorophenyl)-3-(1-imidazolyl)-1-propene [(E)-***4b***]*. Yield 0.33 g (85%), m.p. 72–73°C (hexane–Et_2O). Anal. $C_{18}H_{15}ClN_2$: C, H, N. IR (KBr): 1595 cm^{-1}. ^1H NMR ($CDCl_3$): δ 4.82 (2 H, d, *J* 1.18 Hz, CH_2), 6.54 (1 H, t, *J* 1.18 Hz, H–C = C), 6.89–7.25 (11 H, m), 7.42 (1 H, br s). ^{13}C NMR ($CDCl_3$): δ 55.1 (*C*H_2), 119.0, 127.7, 128.2, 129.18, 129.24, 129.4, 129.7, 130.6, 133.9, 135.1, 135.9, 136.0, 137.3.

*(Z)-1-Phenyl-2-(4-chlorophenyl)-3-(1-imidazolyl)-1-propene [(Z)-***4b***]*. Yield 0.29 g (73%), m.p. 124–125°C (hexane–Et_2O). Anal. $C_{18}H_{15}ClN_2$: C, H, N. IR (KBr): 1490 cm^{-1}. ^1H NMR ($CDCl_3$): δ 5.09 (2 H, s, CH_2), 6.83 (1 H, br s), 6.99 (1 H, br s), 7.14 (1 H, s, H–C = C), 7.26–7.43 (10 H, m). ^{13}C NMR ($CDCl_3$): δ 46.0 (*C*H_2), 118.6, 127.6, 128.1, 128.5, 128.8, 129.0, 129.6, 134.1, 134.2, 134.3, 135.9, 136.9, 138.1.

*(E)-1-(2,4-Dichlorophenyl)-2-phenyl-3-(1-imidazolyl)-1-propene [(E)-***4c***]*. Yield 0.35 g (80%), m.p. 90–91°C (hexane–DME). Anal. $C_{18}H_{14}Cl_2N_2$: C, H, N. IR (KBr): 1585 cm^{-1}. ^1H NMR ($CDCl_3$): δ 4.92 (2 H, d, *J* 1.14 Hz, CH_2), 6.60 (1 H, t, *J* 1.14 Hz, H–C = C), 6.64 (1 H, d, *J* 8.44 Hz), 6.82 (1 H, dd, *J* 8.44 and 2.10 Hz), 6.92–6.99 (3 H, m), 7.04 (1 H, br s), 7.15–7.26 (3 H, m), 7.32 (1 H, d, *J* 2.10 Hz), 7.48 (1 H, br s). ^{13}C NMR ($CDCl_3$): δ 54.1 (*C*H_2), 119.1, 125.8, 126.4, 128.16, 128.19, 128.8, 129.0, 129.3, 131.5, 132.8, 133.4, 134.5, 136.3, 137.4, 139.9.

*(Z)-1-(2,4-Dichlorophenyl)-2-phenyl-3-(1-imidazolyl)-1-propene [(Z)-***4c***]*. Yield 0.31 g (70%), pale yellow oil. An analytically pure sample was not obtained. IR (CCl_4): 1580 cm^{-1}. ^1H NMR ($CDCl_3$): δ 4.97 (2 H, s, CH_2), 6.78 (1 H, br s), 6.95 (1 H, br s), 7.03 (1 H, s, H–C = C), 7.25–7.49 (9 H, m). ^{13}C NMR ($CDCl_3$): δ 45.9 (*C*H_2), 118.6, 126.4, 127.2, 128.6, 128.8, 129.4, 129.5, 129.7, 130.6, 133.2, 134.5, 134.8, 136.9, 137.7, 138.6.

Acknowledgements. Support of this work from the University of Iceland Research Fund is gratefully acknowledged. J. T. N. thanks the University of Iceland Student Innovation Fund for financial support. We are also indebted to Dr. S. Jónsdóttir for recording the NMR spectra and to Ms. S. H. Stefánsdóttir, Fil. lic., for performing the elemental analyses. J. K. F. G. thanks the Department of Chemistry of the University of North Carolina at Greensboro for use of facilities during a stay in North Carolina.

References

1. Gutsche, K., Kohlmann, F. W. and Scharwächter, P. (Nordmark Werke GmbH) *Ger. Offen.*, 18.5. 1977, 25 49 798; *Chem. Abstr. 87* (1977) 102327a.
2. Geirsson, J. K. F., Gudmundsson, B. Ö. and Sigurdardóttir, R. *Acta Chem. Scand. 47* (1993) 1112.
3. Carruthers, W. *Some Modern Methods of Organic Synthesis*, 3rd ed., Cambridge University Press, Cambridge, UK 1986, p. 460.
4. Gammill, R. B., Gold, P. M. and Mizsak, S. A. *J. Am. Chem. Soc. 102* (1980) 3095.
5. Dauben, W. G., Williams, R. G. and McKelvey, R. D. *J. Am. Chem. Soc. 95* (1973) 3932.
6. Schaefer, J. P., Higgins, J. G. and Shenoy, P. K. *Org. Synth. Coll. Vol. V* (1973) 249.
7. Collington, E. W. and Meyers, A. I. *J. Org. Chem. 36* (1971) 3044.
8. House, H. O. *Modern Synthetic Reactions*, 2nd ed., Benjamin, Menlo Park, California 1972, pp. 546–552.
9. Staab, H. A. and Wendel, K. *Chem. Ber. 93* (1960) 2902.
10. Abe, Y., Horii, T., Oka, K., Kawamura, S. and Nakabayashi, T. *Bull. Chem. Soc. Jpn. 53* (1980) 3678.

11. Breitmaier, E. and Voelter, W. *Carbon-13 NMR Spectroscopy*, 3rd ed., VCH, Weinheim 1987, pp. 107–119.
12. Ketcham, R. and Jambotkar, D. *J. Org. Chem. 28* (1963) 1034.
13. Schauble, J. H., Walter, G. J. and Morin, J. G. *J. Org. Chem. 39* (1974) 755.
14. Axenrod, T., Bierig, E. and Schwartz, L. H. *Tetrahedron Lett.* (1965) 2181.

Received October 13, 1994.