Synthesis of substituted isoxazolones and isoxazoles from cyanoenaminones

Kumar K. Mahalanabis[a]*, S.K. Dutta Chowdhury[b], Mili Sarkar[c] and Manisha Misra[a]

[a]*Department of Chemistry, Jadavpur University, Kolkata 700 032, India*

[b]*Jogesh Chandra Chaudhuri College, Kolkata 700 033, India*

[c]*Gurudas College, Kolkata 700 054, India*

α-Cyano-β-enaminones, obtained by regioselective acylation of β-aminocrotononitrile, are smoothly and regiospecifically converted into substituted 5-isoxazolones, which on alkaline hydrolysis afford 4-acyl-3-substituted-5-hydroxyisoxazoles in good to excellent yields.

Keywords: α-cyanoenaminones, β-aminocrotononitrile, 5-isoxazolones, 4-acyl-5-hydroxyisoxazoles

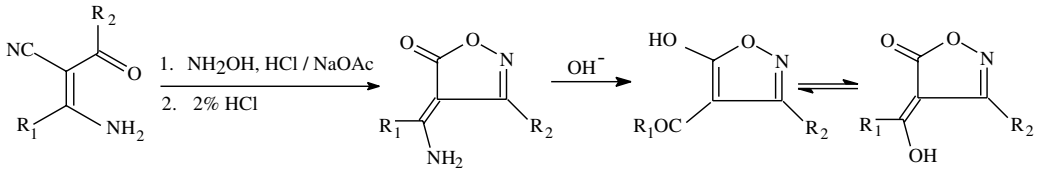
Isoxazoles and their derivatives have been recognised as highly useful in medicinal chemistry. Isoxazole-containing natural and non-natural compounds show interesting biological properties,[1] such as nicotinic acetylcholine receptor ligands,[2] glutamic receptors agonists,[3] fungicides and herbicides.[4] Continuing interest in the chemistry of isoxazoles stems from their usefulness as organic synthetic inetrmediates.[5]

Isoxazoles are most frequently prepared by reaction of 1,3-diketones with hydroxylamine.[6] The procedure works well for symmetrical 1,3-diketones. One serious limitation of this method is the formation of isomeric isoxazoles from an unsymmetrical 1,3-dicarbonyl compound.[7] Another important method for the synthesis of isoxazole involves cycloaddition of alkynes to nitrile oxides.[8]

The chemistry of 5-isoxazolones has been studied extensively[9] for their interesting properties and continues to attract the attention of chemists. General methods for the preparation of 5-isoxazolones include reactions of (i) β-ketoesters and hydroxylamine,[10] (ii) sulfur derivatives of β-ketoesters and hydroxylamine,[11] (iii) αβ-unsaturated esters and hydroxylamine[12], and (iv) β-ketonic and α-acetylenic nitriles with hydroxylamine.[13]

We have recently developed a very successful regioselective acylation of β-aminocrotononitrile to yield α-cyano-β-enaminones in good to excellent yields.[14] These compounds were successfully used as masked 1,3-diketones in the regiospecific synthesis of 5-substituted pyrazole-4-carbonitriles[15] and 5-substituted-4-cyanoisothiazoles.[16] Here, we report the preparation of substituted isoxazolones and isoxazoles via the corresponding α-cyanoenaminones. Thus, refluxing a mixture of α-cyanoenaminones (**1**), hydroxylamine hydrochloride and anhydrous sodium acetate in refluxing alcohol followed by stirring the reaction mixture with 2% hydrochloric acid at room temperature cleanly afforded 3-substituted-4-(1-aminoalkylidene)-isoxazol-5-ones (**2**) in high yields (Scheme 1and Table 1).

Compounds **2a–h** on treatment with alkali directly afforded **3a–h** via hydrolysis of the enamine moiety. However, in the case of compounds **2i** and **2j**, along with the enamine moiety, 3-chloromethyl and 3-dichloromethyl side chains were also hydrolysed to produce a 3-hydroxymethyl isoxazole (**3i**) and a 3-carboxy isoxazole (**3j**) respectively. The structures of **2** and **3** are supported by consistent spectral and elemental analyses (Tables 1–4). Except for compounds **2e** and **2g**, all other compounds in this series are assigned the *E*-configuration based on the chemical shift values of NH_2 protons which absorb in the region δ 5.13–6.77 ppm. However, for **2e**, and compound **2g** one of the amino protons shows absorption at δ 5.66 and δ 5.87 ppm respectively whereas the other amino protons of these compounds absorb at δ 6.81 and δ 6.92 ppm



1 **2** **3**

Scheme 1 Formation of 5–isoxazolones and isoxazoles **2** and **3**.

Table 1 Preparation, and physical and analytical data of compounds **2a–2j**

Compd	R_1	R_2	Yield/%	M.p./°C	Mol. formula	Analyses: % Found (Required)
2a	Me	Me	70	147–148	$C_6H_8N_2O_2$	C, 51.13 (51.41); H, 5.78 (5.76); N, 20.02 (19.99)
2b	Me	CH_2Me	70	112	$C_7H_{10}N_2O_2$	C, 54.37 (54.52); H,6.58 (6.55); N, 18.20 (18.17)
2c	Me	CH_2CH_2Me	89	96	$C_8H_{12}N_2O_2$	C, 56.98 (57.11); H, 7.23 (7.20); N, 16.64 (16.66)
2d	Me	CH_2CH_2Ph	66	128	$C_{13}H_{14}N_2O_2$	C, 67.68 (67.79); H, 6.16 (6.14); N, 12.15 (12.16)
2e	Me	$CH_2OC_6H_3Cl_2(2,4)$	82	162–163	$C_{12}H_{10}Cl_2N_2O_3$	C, 47.78 (47.85); H, 3.37 (3.35); N, 9.32 (9.30)
2f	*p*–$MeOC_6H_4$	Me	75	173–174	$C_{12}H_{12}N_2O_3$	C, 61.99 (62.05); H, 5.22 (5.21); N, 12.05 (12.06)
2g	Me	2–furyl	72	149–150	$C_9H_8N_2O_3$	C, 56.32 (56.45); H, 4.22 (4.19); N, 14.53 (14.57)
2h	Me	2–thienyl	66	182–183	$C_9H_8N_2O_2S$	C, 52.14 (51.92); H, 3.92 (3.87); N, 13.28 (13.46)
2i	Me	CH_2Cl	67	132	$C_6H_7ClN_2O_2$	C, 41.50 (41.28); H, 4.07 (4.04); N, 16.02 (16.04)
2j	Me	$CHCl_2$	65	138–139	$C_6H_6Cl_2N_2O_2$	C, 34.52 (34.47); H, 2.90 (2.89); N, 13.37 (13.40)

* Correspondent. E-mail: kkmahalanabis@yahoo.co.in

Table 2 Preparation, and physical and analytical data of compounds **3a–3j**

Compd	R_1	R_2	Yield/%	M.p./°C	Mol. formula	Analyses: % Found (Required)
3a	Me	Me	82	136–137	$C_6H_7NO_3$	C, 50.95 (51.06); H, 5.03 (5.00); N, 9.95 (9.92)
3b	Me	CH_2Me	77	129–130	$C_7H_9NO_3$	C, 53.98 (54.18); H, 5.86 (5.84); N, 9.06 (9.03)
3c	Me	CH_2CH_2Me	75	114	$C_8H_{11}NO_3$	C, 56.62 (56.79); H, 6.53 (6.55); N, 8.26 (8.28)
3d	Me	CH_2CH_2Ph	72	134	$C_{13}H_{13}NO_3$	C, 67.63 (67.52); H, 5.69 (5.66); N, 5.97 (6.06)
3e	Me	$CH_2OC_6H_3Cl_2$(2,4)	74	202–204	$C_{12}H_9Cl_2NO_4$	C, 48.01 (47.70); H, 2.97 (3.00); N, 4.59 (4.64)
3f	*p*–$MeOC_6H_4$	Me	80	165–166	$C_{12}H_{11}NO_4$	C, 50.95 (51.06); H, 5.03 (5.00); N, 9.95 (9.92)
3g	Me	2–furyl	74	210	$C_9H_7NO_4$	C, 53.98 (54.18); H, 5.86 (5.84); N, 9.06 (9.03)
3h	Me	2–thienyl	72	178	$C_9H_7NO_3S$	C, 56.62 (56.79); H, 6.53 (6.55); N, 8.26 (8.28)
3i	Me	CH_2OH	68	186–187	$C_6H_7NO_4$	C, 67.63 (67.52); H, 5.69 (5.66); N, 5.97 (6.06)
3j	Me	COOH	71	168	$C_6H_5NO_5$	C, 48.01 (47.70); H, 2.97 (3.00); N, 4.59 (4.64)

Table 3 ^{1}H NMR spectra of compounds **2a–2j** and **3a–3j** (in $CDCl_3$, δ; *J*/Hz)

Compd	^{1}H NMR
2a	2.44 (s, 3H, =CCH_3), 2.60 (s, 3H, 3–CH_3), 5.55 (bs, 2H, NH_2)
2b	1.33 (t, *J* = 7.2, 3H, CH_2CH_3), 2.44 (s, 3H, =CCH_3), 3.04 (q, 2H, *J* = 7.2, CH_2CH_3), 5.75 (bs, 2H, NH_2)
2c	0.98 (t, *J* = 7, 3H, $CH_2CH_2CH_3$), 1.78 (sextet, 2H, *J* = 7, $CH_2CH_2CH_3$), 2.44 (s, 3H, =CCH_3), 3.00 (t, $CH_2CH_2CH_3$), 5.91 (bs, 2H, NH_2)
2d	2.40 (s, 3H, CH_3), 3.15 and 3.20 (t, *J* = 7.2, each 2H, $(CH_2)_2$), 5.13 (bs, 2H, NH_2), 7.17 (m, 5H, Ar–*H*)
2e	2.50 (s, 3H, CH_3), 5.38 (s, 2H, CH_2), 5.66 (bs, 1H, N*H*), 6.81 (bs, 1H, N*H*), 7.03–7.47 (m, 3H, Ar–*H*)
2f	2.66 (s, 3H, CH_3), 3.79 (s, 3H, OCH_3), 5.56 (bs, 2H, NH_2), 7.01 and 7.54 (d, each 2H, *J* = 8.4, Ar–*H*)
2g	2.53 (s, 3H, CH_3), 5.87 (bs, 1H, N*H*), 6.65 (1H, d, *J* = 0.9, furan β′H, 6.92 (bs, 1H, N*H*), 7.21 (1H, d, *J* = 3.6, furan βH), 7.66 (1H, d, *J* = 1.0, furan α′H)
2h	2.49 (s, 3 H, CH_3), 5.78 (bs, 2H, NH_2), 7.18 (1H, dd, *J* = 5.1, 3.6, thiophene β′H), 7.61 (1H, d, *J* = 5.1, thiophene βH), 7.82 (1H, d, *J* = 3.6, thiophene α′H)
2i	2.39 (s, 3H, CH_3), 4.79 (s, 2H, CH_2Cl), 6.77 (bs, 2H, NH_2)
2j	2.50 (s, 3H, CH_3), 6.39 (bs, 2H, NH_2), 7.42 (s, 1H, C*H*Cl_2)
3a	2.47 (s, 3H, COCH_3), 2.70 (s, 3H, 3–CH_3), 10.87 (s, 1H, O*H*)
3b	1.35 (t, *J* = 7, 3H, CH_2CH_3), 2.47 (s, 3H, COCH_3), 3.13 (q, 2H, *J* = 7, CH_2CH_3), 9.65 (s, 1H, O*H*)
3c	1.00 (t, *J* = 7.5, 3H, $CH_2CH_2CH_3$), 1.79 (sextet, 2H, *J* = 7.5, $CH_2CH_2CH_3$), 2.47 (s, 3H, COCH_3), 3.08 (t, 2H, *J* = 7.5, $CH_2CH_2CH_3$), 6.16 (s, 1H, O*H*)
3d	2.46 (s, 3H, CH_3), 3.16 and 3.29 (t, *J* = 7.2, each 2H, $(CH_2)_2$), 4.85 (bs, 1H, O*H*), 7.24 (m, 5H, Ar–*H*)
3e	2.51 (s, 3H, CH_3), 3.95 (bs, 1H, O*H*), 5.52 (s, 2H, CH_2), 7.05–7.41 (m, 3H, Ar–*H*)
3f	2.75 (s, 3H, CH_3), 3.86 (s, 3H, OCH_3), 6.19 (bs, 1H, O*H*), 6.96 and 7.62 (d, each 2H, *J* = 9, Ar–*H*)
3g	2.55 (s, 3H, CH_3), 6.65 (1H, dd, *J* = 1.5 & 3.3 furan β′H), 7.70 (1H, d, *J* = 1.0, furan α′H), 7.78 (1H, d, *J* = 3.6, furan βH)
3h	2.54 (s, 3H, CH_3), 7.22 (1H, dd, *J* = 4.8 & 3.6, thiophene β′H), 7.66 (1H, d, *J* = 4.8, thiophene β–H), 7.84 (1H, bs, O*H*), 8.22 (1H, d, *J* = 3.6, thiophene α′H)
3i	2.61 (s, 3H, CH_3), 4.90 (s, 2H, CH_2–OH), 6.78 (bs, 1H, CH_2–O*H*), 12.18 (1H, bs, –O*H*)
3j	2.43 (s, 3H, CH_3), 6.66 (bs, 1H, O*H*), 12.18 (1H, bs, COO*H*)

Table 4 UV and IR spectra of compounds **2a–2j** and **3a–3j**

Compd.	UV[a]	IR[b]
2a	212 (7.17); 273 (0.44)	3360, 3204, 1650, 1468, 1434, 1396, 1154, 1132, 722
2b	218 (8.43)	3380, 3200, 1646, 1626, 1590, 1468, 1430
2c	206 (13.88)	3380, 3192, 2968, 1646, 1590, 1502, 1464, 1428, 1154, 1126
2d	232 (7.69); 279 (11.58)	3372, 3192, 1648, 1620, 1462, 1432, 698
2e	229 (0.99); 283 (1.33)	3440, 3172, 2920, 1698, 1632, 1486, 1462, 1388, 1282, 1020
2f	254 (12.47)	3368, 3188, 1660, 1644, 1454, 1440, 1256, 1172, 834
2g	259 (12.12); 288 (4.71)	3464, 2944, 1624, 1577, 1558, 1128
2h	264 (13.56); 299 (4.99)	3374, 3152, 1609, 1597, 1435, 1384, 1128
2i	278 (2.07)	3336, 2960, 1668, 1618, 1426, 1390, 1260, 1128, 1024, 798
2j	220 (8.70)	3284, 3148, 3020, 1682, 1608, 1422, 1396, 1292, 1130, 788
3a	214 (5.89)	3000–2500(b), 1720, 1600, 1474, 1426, 1376, 1276, 1240, 1126
3b	213 (7.15)	3000–2524(b), 1708, 1606, 1460, 1408, 1272, 1122, 1034, 1026
3c	216 (5.75)	2973–2602(b), 1724, 1683, 1600, 1474, 1312, 1139, 745
3d	224 (6.09)	3660, 3452, 2920, 1714, 1606, 1452, 1260, 1108, 740
3e	229 (13.10); 284 (2.03)	2900(b), 1702, 1600, 1478, 1440, 1392, 1264, 1124, 1102, 788
3f	252 (14.98)	3660, 3640, 2920, 1692, 1612, 1588, 1468, 1256, 1158, 1032
3g	254 (11.68)	2878, 1678, 1593, 1488, 1400, 1113
3h	261 (13.47)	2944, 1667, 1571, 1484, 1404, 1259
3i	265 (12.10); 204 (13.67)	3302, 2588, 1678, 1635, 1590, 1480, 1285, 1119
3j	259 (10.96)	3452, 3266, 1672, 1588, 1438, 1385, 1288, 1129

[a]In EtOH, $\lambda_{max,}$ nm ($\varepsilon \times 10^{-3}$); [b]$\nu_{max}$ cm^{-1}, KBr.

respectively. The low absorption of one of the protons is indicative of the presence of hydrogen bonding with carbonyl oxygen, suggesting the *Z*-configuration for these compounds.

Compounds **3** can exist as tautomeric mixtures. ^{1}H NMR spectral analyses of these compounds did not provide any clear evidence of the presence of any tautomers. However, careful examination of the ^{13}C NMR spectra in some cases supported the presence of tautomeric alternatives. Thus, compound **3b** in its ^{13}C NMR spectrum showed absorptions at δ 11.33, 11.73, 21.13, 107.16, 160.27, 168.13 and 181.30 ppm supportive of the simple 4-acylisoxazole structure, while **3c** showed absorptions at δ 11.66, 11.74, 13.45, 13.70, 20.68, 29.12, 107.66, 108.17, 160. 20, 168.10, 176.80 and 180.34 ppm

which, suggest **3c** to be a mixture of keto and enol tautomers.

The key to the success of these isoxazolone- and isoxazole-forming reactions is the presence of a cyano group in the enaminone moiety. Neither compound **2** nor compound **3** shows any absorption for a nitrile group in the IR spectrum, which may suggest that the reaction involves initial attack of hydroxylamine on the carbonyl group followed by ring closure through nitrile participation. Such exclusive preference for initial 1,2-addition rather than conjugate addition of hydroxylamine has also been observed during formation of isoxazoles or isothiazoles by reacting hydroxylamine with α-oxoketene dithioacetals in a process which does not involve initial conjugate addition of hydroxylamine.[17]

The methodology described here for the sequential preparation of substituted isoxazolones and isoxazoles offers several advantages over literature documented procedures. Thus, a substituent at C-3 can be an alkyl (**3a–e**), heteroaryl (**3g** and **3h**) as well as hydroxymethyl (**3i**) or carboxyl group (**3j**). Variation in the acyl functionality can also be achieved by the use of a suitable enaminonitrile (**3f**). Direct regioselective acylations of the isoxazole nucleus are limited in number. The presence of the enamine functionality in **2** offers the unique advantage of incorporating an acyl group at C-4 with concomitant generation of a hydroxyl group at C-5. 4-Acyl-3-alkyl-5-hydroxyisoxazoles, described in patented material, are found to be useful as pesticide intermediates[18] and are usually quite difficult to prepare. 4-Acylisoxazoles are also used as herbicides.[19] The preparation of 4-acylisoxazoles by the reaction of 1,3-dials with hydroxylamine, or from the reaction of triformylmethane with hydroxylamine, leading to the formation of 4-isoxazolecarbaldehyde oxime, have been reported.[5] However, these methods are associated with some serious disadvantages in terms of starting materials and reaction conditions.

In conclusion, the ready availability of C-acylated enaminonitriles allows a facile and high yielding methodology for the preparation of adjacently-substituted isoxazoles and isoxazolones. This communication and our earlier works[15,16] demonstrate the usefulness of α-cyanoenaminones as unique synthons for synthesis of all three classes of 1,2-azoles by selectively activating the different reacting sites.

Experimental

Melting points were determined in open capillaries. UV spectra were taken on a Hitachi U-2000 spectrometer, IR spectra on a Hitachi 270-30 spectrometer, and ^{1}H NMR spectra in $CDCl_3$ on Hitachi R-600, Bruker-AC 200, Bruker–AC 300, and AV 500 spectrometers. Elemental analyses were performed using a Perkin-Elmer 240C elemental analyser.

β-Aminocrotononitrile[20] and acid chlorides[21] were prepared according to literature procedures. α-Cyanoenaminones **1** were prepared by the reaction of β-aminocrotononitrile with suitable acid chlorides in dry benzene in the presence of pyridine.[14,15]

General procedure for the preparation of **2**

A mixture of α-cyanoenaminone (**1**) (5 mmol), hydroxylamine hydrochloride (15 mmol) and anhydrous sodium acetate (15 mmol) was refluxed on steam bath for 1 to 3h in aqueous alcohol (10 ml water and 5 ml ethanol). Alcohol was removed by evaporation. The reaction mixture was cooled, saturated with NaCl and extracted with ethyl acetate (3 × 20 ml). The organic layer was further washed with brine and dried over anhydrous Na_2SO_4. Removal of the solvent afforded a gummy material consisting of a mixture of 5-imino and 5-oxo compounds which was stirred with 2% HCl at room temperature for 24h. The hydrolysed product was then extracted with ethyl acetate or diethyl ether (3 × 20ml), washed with brine and dried over anhydrous Na_2SO_4. The crude material obtained after removal of the solvent was purified by crystallisation from a suitable solvent to afford **2**.

General procedure for the preparation of **3**

A mixture of compound **2** (5 mmol) and sodium hydroxide solution (1N, 10 ml) was heated on steam bath for a period of 1–4h. The reaction mixture was cooled and acidified with HCl (2N) solution. Usual work up and removal of the solvent produced a solid material, which on crystallisation from a suitable solvent afforded **3** in good to excellent yields.

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References

1 (a) P. Grünanger and P. Vita-Finzi in *The Chemistry of Heterocyclic Compounds, Isoxazoles*, Part 1, A. Weissberger amd E.C. Taylor, eds; Wiley-Interscience: New York, 1990; (b) T.N. Johansen, B. Ebert, H. Brauner-Osborne, M. Didriksen, I.T. Christensen, K.K. Søby, U. Madsen, P. Krogsgaard-Larsen and L. Brehm, *J. Med. Chem*., 1998, **41**, 930; (c) Y.Y. Ku, T. Grieme, P. Sharma, Y.M. Pu, P. Raje, H. Morton and S. King, *Org. Lett.,* 2001, **3**, 4185.
2 N.M. Silva, J.L.M. Tributino, A.L.P. Mirando, E.J. Barreiro and C.A.M. Fraga, *Eur. J. Med. Chem.,* 2002, **37**, 163.
3 E.J. Bjerrum, A.S. Kristensen, D.S. Pickering, J.R. Greenwood, B. Neilsen, T. Liljefors, A. Schousboe, H. Brauner-Osborne and U. Madsen, *J. Med. Chem*., 2003, **46**, 2246.
4 M.A.P. Martins, A.F.C. Flores, G.P. Bastos, A. Sinhorin, H.G. Bonacorso and N. Zanatta, *Tetrahedron Lett*., 2000, **41**, 293 and references cited therein.
5 R.O. Angus, M. R. Bryce, M. Keshavarz-K and F. Wudl, *Synthesis*, 1988, 746.
6 (a) L.V. Claisen and O. Lowman, *Ber*., 1888, **21**, 1149; (b) C. Kashima, *Heterocycles*, 1979, **12**, 1343.
7 S.A. Lang and Y. Lin in *Comprehensive Heterocyclic Chemistry*, K.T. Potts, ed., Pergamon Press, 1984, vol. **6**, 61.
8 (a) A. Dondoni and G. Barbaro, *J. Chem. Soc., Perkin Trans. 2*, 1974, 1591; (b) H.P. Albrecht, D.B. Repke and J.G. Moffatt, *J. Org. Chem*., 1975, **40**, 2143; (c) T.M. Bathazor and R.A. Flores, *J. Org. Chem*., 1980, **45**, 529.
9 S. Batra, M.S. Akhtar, M. Seth and A.P. Bhaduri, *J. Heterocyclic Chem*., 1990, **27**, 337.
10 (a) V.H. Wallingfort, A.H. Homeyer and D.H. Jones, *J. Am. Chem. Soc*., 1941, **63**, 2252; (b) A.R. Katritzky, P. Barczynski, D.L. Ostercamp and I.T. Yousaf, *J. Org. Chem*., 1986, **51**, 4037.
11 D.E. Worral, *J. Am. Chem. Soc*., 1924, **46**, 2832.
12 S. Posner and J. Schreiber, *Ber*., 1924, **57**, 1127.
13 (a) P.S. Burns, *J. Prakt. Chem*., 1893, **47**, 121; (b) E. Meyer, *J. Prakt. Chem*., 1895, **52**, 110.
14 K.K. Mahalanabis, M. Sarkar, S.K. Dutta Chowdhury and C.R. Ghosal, *Ind. J. Chem*., 2002, **41B**, 1902.
15 S.K. Dutta Chowdhury, M. Sarkar and K.K. Mahalanabis, *J. Chem. Res. (S)*, 2003, 746.
16 M. Mishra, S.K. Dutta Chowdhury and K.K. Mahalanabis, *Synth. Commun*., 2004, **34**, 2681.
17 R.K. Dieter and H.J. Chang, *J. Org. Chem*., 1989, **54**, 1088.
18 M. Muneharu, S. Kazuo and U. Takayuki, *Jpn. Kokai Tokkyo Koho* JP 62, 153, 278 [87, 153, 278] (Cl. C07, D261/12), (08 Jul 1987, Appl. 85/297, 662, 26 Dec. 1986, 9pp); *CA*. 1987, **107**, 236 689k.
19 W. Freund, T. Kuekenhoehner, G. Hamprecht, B. Wuerzer, K.O. Westpahlen, N. Meyer and H. Theobald (BASF A.-G) *Eur. Pat. Appl.* EP 337 263 (Cl C07 D261/18), 18 Oct. 1989, DE Appl. 3 812 225 (13 Apr. 1988, 56 pp); *CA*. 1990, **112**, 235 289w.
20 H. Adkins and G.M. Whitman, *J. Am. Chem. Soc*., 1942, **64**, 150.
21 (a) H.C. Brown, *J. Am. Chem. Soc*., 1938, **60**, 1325; (b) Org. Synth. Coll. Vol. 3, E.C. Hornung (ed.), John Wiley and Sons Inc., New York; Chapman and Hall, London, Ltd. 1955, p 29, 714; (c) A.I. Vogel, in *A Text Book of Practical Organic Chemistry*, 3rd edn, ELBS and Longman, 1973, p 121, p 791.