The Thermal Amidation of Carboxylic Acids Revisited

Lukas J. Gooßen,* Dominik M. Ohlmann, Paul P. Lange

Fachbereich Chemie, Technische Universität Kaiserslautern, Erwin-Schrödinger-Straße 54, 67663 Kaiserslautern, Germany
Fax +49(631)3921; E-mail: goossen@chemie.uni-kl.de

Received 12 November 2008

Abstract: Factors affecting the thermal condensation of carboxylic acids with amines have been investigated, and an effective protocol for this waste-minimized, environmentally benign transformation has been identified. Fourteen examples demonstrate the applicability of this procedure to aliphatic, aromatic and heteroaromatic carboxylic acids and primary and secondary aliphatic as well as aromatic amines. The approach leads to the corresponding amides in good yields.

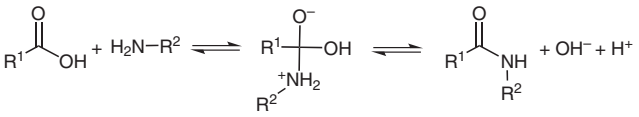
Key words: amides, amines, condensation, molecular sieves

The synthesis of amides from carboxylic acids is certainly one of the most commonly used transformations in organic chemistry.[1] A wealth of methods have been developed for this, which usually involve various coupling reagents.[2] These reagents activate the carboxylic acid for nucleophilic substitution by derivatization into acid chlorides, anhydrides, activated amides, or esters, thus replacing the free hydroxy group by a leaving group. Driven in particular by the requirements of automated peptide synthesis, these methods have become highly efficient. However, they suffer from the need for a separate activation step and from low atom economy. Alternatively, carboxylic acids can be condensed with amines in the presence of stoichiometric activators such as arylboronic acids,[3] bis[bis(trimethylsilyl)amino]tin(II),[4] titanium tetrachloride,[5] trimethylaluminium,[6] Lawesson's reagent,[7] dimethyl phosphoryl chloride,[8] tetrazoles,[9] benzoxazoles,[10] and oxalates.[11] Unfortunately, each of these reagents has their own disadvantage, being unstable, toxic, expensive or commercially unavailable, and requires the removal of by-products.

A direct conversion of carboxylic acids involving nucleophilic substitution of the hydroxy group by an amine is very desirable but intrinsically difficult: Under acidic conditions, the hydroxy group of a carboxylic acid is protonated and easily substituted via addition–elimination reactions allowing, for example, direct esterifications. In contrast, the acid is deprotonated by basic amines to form a resonance-stabilized carboxylate, which impedes nucleophilic attack at the carbonyl carbon.

A dehydration of the resulting ammonium carboxylates usually requires such drastic conditions that this approach can be applied only to particularly reactive and otherwise unfunctionalized derivatives. For example, the pyrolysis of primary ammonium salts of short-chain aliphatic carboxylic acids[12] or of β-phenethylamines with phenylacetic acid[13] requires a reaction temperature of 180 °C. Benzoic acid and aniline form benzanilide at 220 °C,[14] and oleic acid starts reacting with primary amines at temperatures in excess of 230 °C,[15] all in the absence of solvent.

The mechanism of this condensation is assumed to involve an equilibrium of free carboxylic acid, amine, and ammonium salt, from which the product amide forms via the reversal of an amide hydrolysis, driven by the removal of water from the mixture (Scheme 1).[16]



Scheme 1 Postulated mechanism for the direct amidation of carboxylic acids

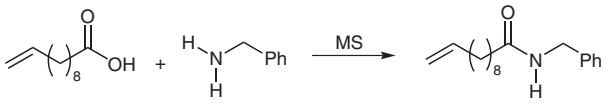
In an ongoing synthetic project, we required a simple, scalable, economically and ecologically viable process for the amidation of a fatty acid. After obtaining disappointing results with various processes, we finally came across a publication by Cossy et al., who used molecular sieves for the condensation of carboxylic acids with amines to give the amide products.[17] We considered this to be a very appealing approach although the reported examples included only a very limited range of mainly short-chain aliphatic carboxylic acids in combination with primary amines. Thus, we decided to take a new look at this process with the goal of making it generally applicable to a broad range of carboxylic acid and amine substrates.

We chose the amidation of undecenoic acid with benzylamine as our model reaction in order to optimize the mediator and the reaction conditions (Table 1).

The original procedure using activated 4 Å molecular sieves gave an encouraging 59% yield of *N*-benzyl-10-undecenamide after pretreating the molecular sieves by microwave heating (entry 1). Other dehydrating agents, Lewis or Brønsted acids led to decreased yields (entries 2–4). When attempting to further increase yields by varying the pore size (3 Å, entry 5), the pretreatment (drying, grinding) or the amount of the molecular sieves (entries 6–8), we were surprised to see only marginal differences. A control experiment performed in the absence of drying agents or mediators led to the surprising discovery that almost the same yields could be achieved when the reaction

SYNTHESIS 2009, No. 1, pp 0160–0164
Advanced online publication: 12.12.2008
DOI: 10.1055/s-0028-1083277; Art ID: C06008SS

Table 1 Optimization of Reaction Conditions[a]



1a **2a** **3aa**

Entry	MS size (Å)[b]	MS (mg)	Additive	Temp (°C)	Yield (%)[c]
1	4	100	–	120	59
2	4	100	$MgSO_4$	120	39
3	4	100	$Yb(OTf)_3$	120	14
4	4	100	H_2SO_4	120	45
5	3	100	–	120	48
6	4	200	–	120	58
7	4	400	–	120	52
8	4	60	–	120	49
9	–	–	–	120	49
10	4	100	–	160	89
11	–	–	–	160	95

[a] Reaction conditions: 10-undecenoic acid (**1a**; 2 mmol), benzylamine (**2a**; 2 mmol), additive (0.2 equiv), 2 h.
[b] Activated molecular sieves (MS).
[c] Determined by GC using *n*-tetradecane as internal standard.

water was simply allowed to evaporate (entry 9). After increasing the temperature to 160 °C, quantitative conversion was achieved after two hours both with and without molecular sieves present (entries 10 and 11).

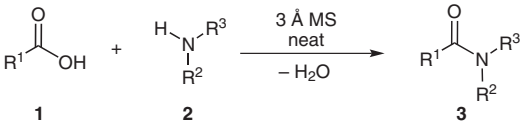
Intrigued by this finding, we searched for literature evidence that unmediated thermal amidations can be performed even at such relatively low temperatures. Indeed, Jursic et al. reported thermal amidations to proceed at 160–180 °C and successfully applied this method to the synthesis of a small selection of amides.[18] Jursic's findings confirm that the molecular sieves do not mediate the actual amidation step but merely trap the released reaction water. We still found the use of pelleted 3 Å molecular sieves to be beneficial from a practical point of view, as this way closed vessels can be used, and these prevent the evaporation of volatile amines. By trapping the reaction water, the molecular sieves help to reduce pressure build-up at temperatures in excess of 100 °C.

In order to investigate the scope of such thermal amidation reactions in more detail, we tested the scope of our optimized protocol by applying it to various combinations of amines and carboxylic acids (Table 2). We were pleased to find that the new protocol allows extending the method from aliphatic and olefinic carboxylic acids to the less reactive aromatic and even heteroaromatic carboxylic acids. Their reaction with benzylamine gave the corresponding amides in mostly high yields.

Aliphatic carboxylic acids reacted smoothly with primary and secondary aliphatic as well as aromatic amines. Even morpholine, which had previously been reported to be completely unreactive,[17] gave a good yield when converted just below its boiling point (120 °C). The protocol is particularly advantageous for the synthesis of fatty acid amides, which are of considerable commercial interest as antifriction lubricants and chemical additives, as well as nonstick and protective coatings.[19] The presence of alcoholic or phenolic OH groups is tolerated; in all cases, the amides rather than the esters were formed exclusively. The reaction of benzoic acid with a secondary amine represents the current performance limit of the transformation: in this case only traces of product were detected.

The workup of the reaction mixtures is simple, and consists of dilution with methanol and filtration. After removal of the volatile components, the products are in many cases obtained in high purity. Otherwise, they could easily be purified by recrystallization or column chromatography.

Table 2 Substrate Scope of the Direct Amidation[a]



1 **2** **3**

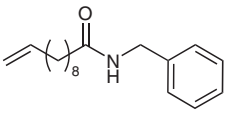
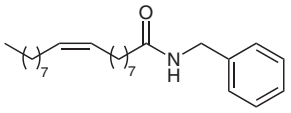
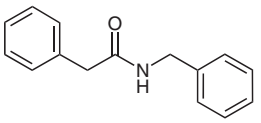
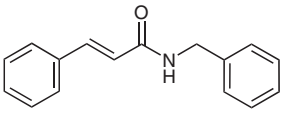
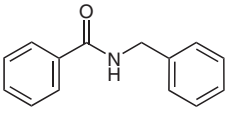
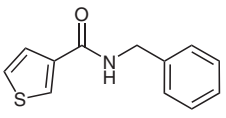
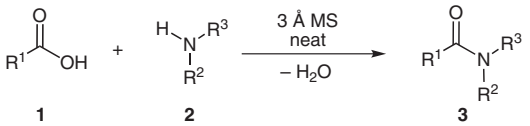
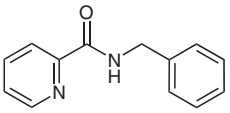
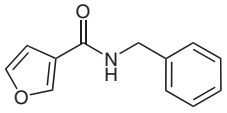
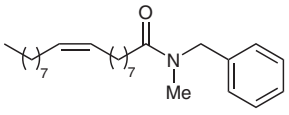
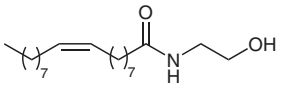
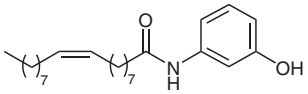
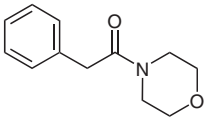
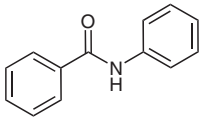
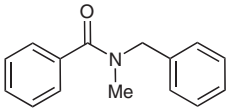
Product	Time (h)	Temp (°C)	Yield (%)[b]
 **3aa**	2	160	92
 **3ba**	2	160	99
 **3ca**	7	160	85
 **3da**	24	160	96
 **3ea**	24	160	75
 **3fa**	7	160	85

Table 2 Substrate Scope of the Direct Amidation[a] (continued)



1 2 3

Product	Time (h)	Temp (°C)	Yield (%)[b]
 **3ga**	24	160	75
 **3ha**	7	160	55
 **3bb**	12	160	96
 **3bc**	12	160	92
 **3bd**	24	160	75
 **3ce**	7	120	73
 **3ef**	24	160	49
 **3eb**	24	160	traces

[a] Reaction conditions: carboxylic acid **1** (2 mmol), amine **2** (2 mmol), activated MS (3 Å, 100 mg).
[b] Isolated yields.

Overall, the results show that the direct condensation of carboxylic acids and amines to give the corresponding amides is of higher synthetic applicability than commonly believed. This environmentally friendly method can be applied to the synthesis of a wide range of amides that are currently produced via thionyl chloride based protocols.[20]

All solvents and reagents were purified by standard techniques. Crude products were purified by column chromatography on silica gel or by recrystallization from EtOH or $CHCl_3$/hexane. IR spectra were recorded on a Perkin–Elmer FT IR spectrometer. ^1H and ^{13}C NMR spectra were recorded at r.t. in $CDCl_3$ or CD_3OD solvent on Bruker DPX 400 or Bruker Avance 600 NMR spectrometers. Chemical shifts (δ) are reported in ppm relative to solvent signals (δ = 7.25 and 77.0 ppm for $CDCl_3$ or 3.30/4.78 and 49.0 ppm for CD_3OD). Coupling constants (*J*) are quoted in Hz. Mass spectra were obtained on a Varian Saturn 2100 T mass spectrometer operating at 70 eV using EI ionization. Melting points were determined on a Büchi 520 melting point apparatus and are uncorrected.

3 Å molecular sieves (MS) were pre-treated in a household microwave for 5 min at 700 W in an open beaker. It is advisable to start with pre-dried sieves and to continually monitor the microwave process because tightly packed, wet sieves may dramatically overheat, leading to a meltdown of the material. In a flame-dried vessel, they were additionally heated in vacuo with a heat gun and cooled to r.t. before use.

N-Benzyloleamide (3ba);[21] Typical Amidation Procedure

A mixture of oleic acid (**1b**; 5.65 g, 20.0 mmol) and *N*-benzylamine (**2a**; 2.14 g, 20.0 mmol) was placed in a flame-dried 20 mL headspace vial, highly activated MS (3 Å, 1.80 g) were added in one portion and the mixture was heated at 160 °C. After completion of the reaction (2 h, monitored by TLC), the mixture was cooled, diluted with MeOH (150 mL), filtered through a thin pad of Celite, washed with MeOH (150 mL) and concentrated in vacuo to afford the amide **3ba**.

Yield: 7.41 g (99%); white solid; mp 58–59 °C; R_f = 0.8 (silica gel; EtOAc–hexane, 1:1).

IR (KBr): 3298, 1640, 1553 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.22–7.33 (m, 5 H), 6.32 (s, 1 H), 5.35 (s, 2 H), 4.38 (d, *J* = 5.7 Hz, 2 H), 2.18 (t, *J* = 7.6 Hz, 2 H), 2.02 (m, 4 H), 1.62 (m, 2 H), 1.29 (m, 20 H), 0.89 (t, *J* = 6.4 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.9, 138.5, 129.9, 129.6, 128.5, 127.6, 127.2, 43.4, 36.6, 31.8, 29.7, 29.6, 29.4, 29.2, 29.0, 27.1, 25.7, 22.5, 13.9.

MS (EI, 70 eV): *m*/*z* (%) = 371 (48) [M^+], 216 (50), 162 (75), 149 (100), 91 (86).

N-Benzyl-10-undecenamide (3aa)[22]

Synthesized from **1a** (369 mg, 2.00 mmol) and *N*-benzylamine (**2a**; 214 mg, 2.00 mmol) following the typical procedure (160 °C, 2 h) and purified by recrystallization (hexane).

Yield: 503 mg (92%); white solid.

^1H NMR (400 MHz, CD_3OD): δ = 7.21–7.32 (m, 5 H), 5.74–5.85 (m, 1 H), 4.90–5.01 (m, 2 H), 4.34 (s, 2 H), 2.22 (t, *J* = 7.5 Hz, 2 H), 2.03 (q, *J* = 6.9 Hz, 2 H), 1.57–1.66 (m, 2 H), 1.33–1.41 (m, 2 H), 1.30 (s, 8 H).

N-Benzyl-*N*-methyloleamide (3bb)[23]

Synthesized from **1b** (1.41 g, 5.00 mmol) and *N*-methylbenzylamine (**2b**; 606 mg, 5.00 mmol) following the typical procedure (160 °C, 12 h) and purified by column chromatography (Et_2O–hexane, 1:3). Spectroscopic data of this amide were obtained as a mixture of two rotational isomers consistent with literature.

Yield: 1.89 g (96%); light-yellow liquid.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.27–7.35 (m, 2 H), 7.21–7.25 (m, 2 H), 7.15 (d, *J* = 7.5 Hz, 1 H), 5.34 (d, *J* = 4.8 Hz, 2 H), 4.58 (s, 1 H), 4.52 (s, 1 H), 2.88–2.94 (m, 3 H), 2.36 (t, *J* = 7.5 Hz, 2 H),

2.01 (s, 4 H), 1.68 (d, *J* = 6.8 Hz, 2 H), 1.33 (s, 8 H), 1.27 (s, 13 H), 0.88 (t, *J* = 6.3 Hz, 3 H).

N-(2-Hydroxyethyl)oleamide (3bc)[24]
Synthesized from **1b** (1.41 g, 5.00 mmol) and 2-aminoethanol (**2c**; 305 mg, 5.00 mmol) following the typical procedure (160 °C, 12 h).

Yield: 1.57 g (92%); colorless solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.34 (s, 1 H), 5.31 (ddd, *J* = 5.5, 3.7, 3.4 Hz, 2 H), 3.56–3.68 (m, 3 H), 3.33–3.40 (m, 2 H), 2.11–2.20 (m, 2 H), 1.92–2.02 (m, 4 H), 1.53–1.63 (m, 2 H), 1.25 (m, 20 H), 0.81–0.88 (m, 3 H).

N-(3-Hydroxyphenyl)oleamide (3bd)[25]
Synthesized from **1b** (1.41 g, 5.00 mmol) and 3-aminophenol (**2d**; 546 mg, 5.00 mmol) following the typical procedure (160 °C, 24 h) and purified by column chromatography (Et_2O–hexane, 1:2).

Yield: 1.41 g (75%); colorless solid; mp 93–94 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95 (t, *J* = 2.0 Hz, 1 H), 7.25 (s, 1 H), 7.14 (t, *J* = 8.5 Hz, 1 H), 6.63–6.66 (m, 1 H), 6.49–6.52 (m, 1 H), 5.29–5.38 (m, 2 H), 2.37 (t, *J* = 7.5 Hz, 2 H), 1.96–2.05 (m, 5 H), 1.69–1.77 (m, 2 H), 1.24–1.35 (m, 20 H), 0.87 (t, *J* = 6.5 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.6, 157.7, 138.6, 130.0, 129.7, 129.6, 111.9, 107.5, 38.1, 31.9, 29.8, 29.3, 29.2, 29.1, 27.2, 25.8, 22.7, 14.2.

***N*-Benzyl-2-phenylacetamide (3ca)[26]**
Synthesized from phenylacetic acid (**1c**; 408 mg, 3.00 mmol) and *N*-benzylamine (**2a**; 321 mg, 3.00 mmol) following the typical procedure (160 °C, 7 h) and purified by column chromatography (Et_2O–hexane, 9:1).

Yield: 574 mg (85%); white solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.31–7.34 (m, 2 H), 7.21–7.29 (m, 6 H), 7.14–7.17 (m, 2 H), 5.80 (s, 1 H), 4.38 (d, *J* = 5.9 Hz, 2 H), 3.59 (s, 2 H).

(*E*)-*N*-Benzyl Cinnamic Amide (3da)[27]
Synthesized from cinnamic acid (**1d**; 444 mg, 3.00 mmol) and *N*-benzylamine (**2a**; 321 mg, 3.00 mmol) following the typical procedure (160 °C, 24 h) and recrystallized ($CHCl_3$–hexane).

Yield: 685 mg (96%); light-yellow solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.64 (t, *J* = 5.8 Hz, 1 H), 7.55–7.61 (m, 2 H), 7.47 (d, *J* = 15.9 Hz, 1 H), 7.35–7.42 (m, 3 H), 7.28–7.34 (m, 3 H), 7.24 (t, *J* = 7.2 Hz, 1 H), 6.70 (d, *J* = 15.9 Hz, 1 H), 4.40 (d, *J* = 5.9 Hz, 2 H), 2.49 (d, *J* = 3.3 Hz, 1 H).

***N*-Benzylbenzamide (3ea)[28]**
Synthesized from benzoic acid (**1e**; 374 mg, 3.00 mmol) and *N*-benzylamine (**2a**; 321 mg, 3.00 mmol) following the typical procedure (160 °C, 24 h) and purified by column chromatography (EtOAc–hexane, 2:3).

Yield: 478 mg (75%); white solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.79 (d, *J* = 7.4 Hz, 2 H), 7.44–7.50 (m, 1 H), 7.39–7.42 (m, 2 H), 7.32–7.35 (m, 4 H), 7.27–7.30 (m, 1 H), 6.60 (s, 1 H), 4.62 (d, *J* = 5.6 Hz, 2 H).

***N*-Benzylthiophene-3-carboxamide (3fa)**
Synthesized from thiophene-3-carboxylic acid (**1f**; 384 mg, 3.00 mmol) and *N*-benzylamine (**2a**; 321 mg, 3.00 mmol) following the typical procedure (160 °C, 7 h) and purified by column chromatography (EtOAc–hexane, 2:3).

Yield: 553 mg (85%); white solid; mp 92–93 °C; R_f = 0.1 (Et_2O–hexane, 1:1).

^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.86–7.88 (m, 1 H), 7.37–7.40 (m, 1 H), 7.27–7.34 (m, 6 H), 6.45 (s, 1 H), 4.58 (d, *J* = 5.9 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 162.9, 138.1, 137.2, 128.7, 128.4, 127.9, 127.5, 126.5, 126.0, 43.8.

MS (EI, 70 eV): *m*/*z* (%) = 217 (99) [M^+], 184 (100), 156 (10), 111 (98), 83 (20), 77 (27), 51 (16).

Anal. Calcd for $C_{12}H_{11}NOS$: C, 66.33; H, 5.10; N, 6.45. Found: C, 66.31; H, 5.05; N, 6.66.

***N*-Benzylpicolinamide (3ga)[29]**
Synthesized from picolinic acid (**1g**; 369 mg, 3.00 mmol) and *N*-benzylamine (**2a**; 321 mg, 3.00 mmol) following the typical procedure (160 °C, 24 h) and purified by column chromatography (EtOAc–hexane, 1:1).

Yield: 477 mg (75%); white solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.51 (d, *J* = 4.1 Hz, 1 H), 8.38 (s, 1 H), 8.23 (d, *J* = 7.9 Hz, 1 H), 7.83 (td, *J* = 7.7, 1.7 Hz, 1 H), 7.40 (ddd, *J* = 7.6, 4.7, 1.3 Hz, 1 H), 7.31–7.37 (m, 4 H), 7.27 (t, *J* = 7.0 Hz, 1 H), 4.66 (d, *J* = 5.9 Hz, 2 H).

***N*-Benzylfuran-3-carboxamide (3ha)[30]**
Synthesized from furan-3-carboxylic acid (**1h**; 336 mg, 3.00 mmol) and *N*-benzylamine (**2a**; 321 mg, 3.00 mmol) following the typical procedure (160 °C, 24 h) and purified by column chromatography (EtOAc–hexane, 3:1).

Yield: 331 mg (55%); light-yellow solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.94 (s, 1 H), 7.41 (dt, *J* = 16.1, 1.7 Hz, 1 H), 7.27–7.33 (m, 5 H), 6.62 (d, *J* = 1.0 Hz, 1 H), 6.38 (s, 1 H), 4.55 (d, *J* = 5.9 Hz, 2 H).

4-(Phenylacetyl)morpholine (3ce)[31]
Synthesized from phenylacetic acid (**1c**; 408 mg, 3.00 mmol) and morpholine (**2e**; 261 mg, 3.00 mmol) following the typical procedure (120 °C, 7 h) and purified by column chromatography (EtOAc–hexane, 3:1).

Yield: 452 mg (73%); light-yellow solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.31 (t, *J* = 7.5 Hz, 2 H), 7.21–7.25 (m, 3 H), 3.72 (s, 2 H), 3.63 (s, 4 H), 3.40–3.47 (m, 4 H).

***N*-Phenylbenzamide (3ef)[32]**
Synthesized from benzoic acid (**1e**; 366 mg, 3.00 mmol) and aniline (**2f**; 279 mg, 3.00 mmol) following the typical procedure (160 °C, 24 h) and purified by column chromatography (EtOAc–hexane, 1:2) and recrystallization ($CHCl_3$–hexane).

Yield: 287 mg (49%); colorless solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.83–7.88 (m, 3 H), 7.64 (d, *J* = 7.7 Hz, 2 H), 7.54 (t, *J* = 7.4 Hz, 1 H), 7.48 (t, *J* = 7.6 Hz, 2 H), 7.36 (t, *J* = 8.1 Hz, 2 H), 7.15 (t, *J* = 7.4 Hz, 1 H).

Acknowledgment

We gratefully acknowledge the financial support by Cognis GmbH and NanoKat and thank Felix Rudolphi for helpful discussions and technical assistance.

References

(1) Smith, M. B.; March, J. *Advanced Organic Chemistry*, 5th ed.; Wiley: New York, **2001**, 509; and references cited.

(2) (a) Larock, R. C. *Comprehensive Organic Transformations*; VCH: New York, **1989**, 972. (b) Klausner, Y. S.; Bodansky, M. *Synthesis* **1972**, 453.
(3) Ishihara, K.; Ohara, S.; Yamamoto, H. *J. Org. Chem.* **1996**, *61*, 4196.
(4) Burnell-Curty, C.; Roskamp, E. J. *Tetrahedron Lett.* **1993**, *34*, 5193.
(5) Wilson, J. D.; Weingarten, H. *Can. J. Chem.* **1970**, *48*, 983.
(6) Levin, J. I.; Turos, E.; Weinreb, S. M. *Synth. Commun.* **1982**, *12*, 989.
(7) Thorsen, M.; Andersen, T. P.; Pedersen, U.; Yde, B.; Lawesson, S. *Tetrahedron* **1985**, *41*, 5633.
(8) Jászay, Z. M.; Petnecházy, I.; Töke, L. *Synth. Commun.* **1998**, *28*, 2761.
(9) Schmidt, U.; Dietsche, M. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 143; *Angew. Chem.* **1982**, *94*, 145.
(10) Ueda, M.; Oikawa, H.; Kawaharasaki, N.; Imai, Y. *Bull. Chem. Soc. Jpn.* **1983**, *56*, 2485.
(11) Takeda, K.; Sawada, I.; Suzuki, A.; Ogura, H. *Tetrahedron Lett.* **1983**, *24*, 4451.
(12) Mitchell, J. A.; Reid, E. E. *J. Am. Chem. Soc.* **1931**, *53*, 1879.
(13) Shepard, E. E.; Porter, H. D.; Noth, J. F.; Simmans, C. K. *J. Org. Chem.* **1952**, *17*, 568.
(14) Webb, C. N. *Org. Synth., Coll. Vol. I*; John Wiley & Sons: London, **1941**, 82.
(15) Roe, E. T.; Scanlan, J. T.; Swern, D. *J. Am. Chem. Soc.* **1949**, *71*, 2215.
(16) Beckwith, A. L. J. In *The Chemistry of Amides*; Zabicky, J., Ed.; Wiley: New York, **1970**, 105.
(17) Cossy, J.; Pale-Grosdemange, C. *Tetrahedron Lett.* **1989**, *30*, 2771.
(18) Jursic, B. S.; Zdravkovski, Z. *Synth. Commun.* **1993**, *23*, 2761.
(19) Kaneshiro, T.; Vesonder, R. F.; Peterson, R. E.; Weisleder, D.; Bagby, M. O. *J. Am. Oil Chem. Soc.* **1994**, *71*, 491.
(20) (a) Fong, C.; Wells, D.; Krodkiewska, I.; Hartley, P. G.; Drummond, C. J. *Chem. Mater.* **2006**, *18*, 594. (b) Rosini, G.; Andreotti, D. G.; D'Ambrosio, P.; Marotta, E.; Tinavelli, A.; Righi, P. *Synthesis* **2007**, 3051.
(21) Mod, R. R.; Magne, F. C.; Skau, E. L. *J. Chem. Eng. Data* **1962**, 31.
(22) Bartra, M.; Vilarrasa, J. *J. Org. Chem.* **1991**, *56*, 5132.
(23) Hanada, S.; Motoyama, Y.; Nagashima, H. *Tetrahedron Lett.* **2006**, *47*, 6173.
(24) Lin, S.; Khanolkar, A. D.; Fan, P.; Goutopoulos, A.; Qin, C.; Papahadjis, D.; Makriyannis, A. *J. Med. Chem.* **1998**, *41*, 5353.
(25) Flerz-David, H. E.; Meister, H. *Helv. Chim. Acta* **1939**, *22*, 579.
(26) Katritzky, A. R.; Cai, C.; Singh, S. K. *J. Org. Chem.* **2006**, *71*, 3375.
(27) Maki, T.; Ishihara, K.; Yamamoto, H. *Org. Lett.* **2005**, *7*, 5043.
(28) Davidsen, S. K.; May, P. D.; Summers, J. B. *J. Org. Chem.* **1991**, *56*, 5482.
(29) Paruszewski, R.; Strupinska, M.; Stables, J. P.; Swiader, M.; Czuczwar, S.; Kleinrok, Z.; Turski, W. *Chem. Pharm. Bull.* **2001**, *49*, 629.
(30) Zanatta, N.; Faoro, D.; Silva, S. C.; Bonacorso, H. G.; Martins, M. A. P. *Tetrahedron Lett.* **2004**, *45*, 5689.
(31) Heyde, C.; Zug, I.; Hartmann, H. *Eur. J. Org. Chem.* **2000**, *19*, 3273.
(32) Chandrasekhar, S.; Sultana, S. S.; Yaragorla, S. R.; Reddy, N. R. *Synthesis* **2006**, 839.