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Friedel-Crafts Reactions. V.* *N*-Formyl-3-methoxymorpholine and *N*-Formyl-2,3-dihydro-4*H*-1,4-oxazine as Reactants in the Amidoalkylation of Aromatic Substrates

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N-Formyl-3-methoxymorpholine and *N*-formyl-2,3-dihydro-4*H*-1,4-oxazine react with aromatic compounds in the presence of $AlCl_3$ forming 3- or 2-aryl-*N*-formylmorpholines. The 2-aryl isomer can take part in a second alkylation reaction to yield an *N*-(2,2-diarylethyl)-*N*-(2-hydroxyethyl)-formamide. By proper adjustment of the reaction conditions, either product can be made the predominant one.

N-(2,2-Diarylethyl)-*N*-(2-hydroxyethyl)formamides are useful precursors to *N*-heterocyclic ring systems. Thus the hydroxyethyl group of the diphenyl derivative was converted to a vinyl group by reaction with thionyl chloride and subsequent treatment with a base. In the presence of $AlCl_3$ this compound was cyclized to give 3,4-dihydro-1-methyl-4-phenyl-2(1*H*)-isoquinolinecarboxaldehyde.

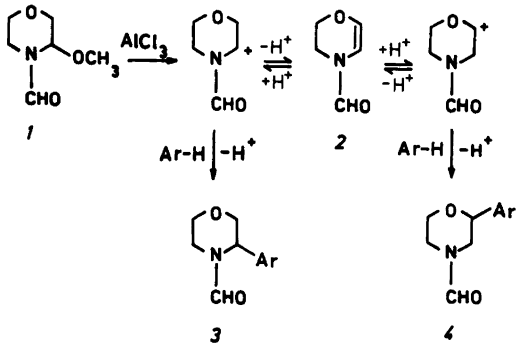
The amidoalkylation reaction is a well-known and thoroughly investigated method for the formation of a C–C bond.[1] In recent years, the scope of the reaction has been considerably extended[2–4] by the easy access to *cyclic* precursors to the key species in amidoalkylation, the *N*-acylimmonium ion. These compounds, cyclic *N*-formyl-2-methoxyamines, are prepared in large quantities and excellent yields by the anodic oxidation of *N*-formylamines in methanol.[5–8]

In most cases, amidoalkylations require a strong Lewis acid, *e.g.* aluminium chloride, in order to take place.[2–4] Under such conditions, it is frequently observed that a proton is abstracted from the *N*-acylimmonium ion with formation of an enamide. The enamides, easily prepared from the α-methoxyamides,[9] can be used equally well as amidoalkylating agents, sometimes even with some advantage over the methoxyamides.

In the case of the morpholine derivative *1*, the intervention of the enamide (*2*) opens a pathway for alkylation *via* the carbon α to oxygen (the 2-position) as well[2] (see Scheme 1). This paper outlines the synthetic possibilities available in using *2* for the alkylation of aromatic compounds, extended considerably by the further alkylation/ring opening reaction that the 2-aryl-*N*-formylmorpholine can undergo.

RESULTS

According to Scheme 1, the first intermediate from *1* is the *N*-formylimmonium ion which can



Scheme 1.

* Part IV, see Ref. 4.

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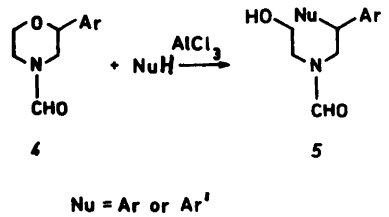
Table 1. Alkylation of aromatic compounds by *1*, *2*, and *4*.

Alkylating agent	Aromatic compound (molar excess over substrate)	Molar excess of catalyst over substrate (temp/°C)	Products (yield/%)
1	PhH (10)	2.0 (80)	*3*, Ar=Ph (46) *4*, Ar=Ph (18)
1	PhH (~12)	4.0 (80)	*3*, R=Ph (57)[a]
2	PhH (10)	1.0+0.33[b] *(0)*	*4*, R=Ph (47) *5*, R=Nu=Ph (9)
2	PhH (10)	2.0 (80)	*5*, R=Nu=Ph (68)
1	Mesitylene (1.5)[c]	2.0 (25)	*3*, R=2,4,6-Me_3-phenyl (67)
1	1,3,5-Trimethoxybenzene[d]	1.7[e] (25)	*3*, R=2,4,6-$(MeO)_3$-phenyl (82)
2	Mesitylene (1.1)[c]	1.0 (25)	*4*, R=2,4,6-Me_3-phenyl (47)
2	*p*-Xylene (1.1)[c]	1 (0)	*4*, R=2,5-Me_2-phenyl (18)
2	*p*-Xylene (5)	2 (25)	*5*, R=Nu=2,5-Me_2-phenyl (56)
4, R=Ph	Mesitylene (10)	1.5 (55)	*5*, R=Ph, Nu=2,4,6-Me_3-phenyl (73)
4, R=Ph	*p*-Xylene (10)	1.5 (25)	*5*, R=Ph, Nu=2,5-Me_2-phenyl (48)

[a] Minor amounts of *5*, R=Nu=Ph were detectable. [b] Successive additions of catalyst. [c] Dichloromethane as solvent. [d] Ref. 2. [e] CF_3COOH was used as catalyst.

partly or completely equilibrate with the α-oxa cation *via* the enamide. Depending on the reactivity of the substrate it should then be possible to intercept the *N*-formylimmonium ion to give the 3-aryl compound (*3*) only or, with a sluggishly reacting compound, give an equilibrated mixture of *3* and *4*.

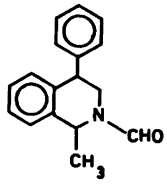
Benzene exemplifies the latter case and mesitylene and 1,3,5-trimethoxybenzene[2] the former one (Table 1) However, the reaction with benzene can be directed to give the 3-aryl isomer exclusively by taking advantage of the further ring opening/alkylation that the other isomer can undergo [eqn. (1)]. Under more forcing conditions *4* is then converted to *5*, and *3* is the only arylmorpholine obtained. This could be further substantiated by treating the *3*/*4* mixture by $AlCl_3$ in refluxing benzene; *3* remains unchanged whereas *4* is converted to *5*.



Starting from *2*, one would expect that a mixture of the two isomeric cations should be formed, in principle leading to mixtures of *3* and *4* upon alkylation of ArH. Table 1 shows however, that the 2-aryl isomer (*4*) is selectively formed, presumably due to the expected higher reactivity of the α-oxa cation. Under more forcing conditions, *2* can be converted directly to Ar_2-substituted *5* (exemplified by benzene and *p*-xylene) or can *4* first be isolated and then used to alkylate a second substrate to give Ar,Ar'-substituted *5*.

It was found important to carefully control the temperature (kept <5 °C; efficient stirring is required in the sometimes very viscous mixtures) when isolation of *4* is desired, especially during the addition of *1* or *2* to the solvent/substrate/catalyst mixture (exothermic reaction!). Details are given in the experimental section.

In principle, one might hope that *5* should be capable of undergoing elimination of water and intramolecular amidoalkylation to give *6* in the presence of $AlCl_3$. This did not take place, however, but the transformation could be achieved by converting the 2-hydroxyethyl group into a 2-chloroethyl group ($SOCl_2$), followed by elimination of HCl (*t*-BuOK). The *N*-vinyl derivative smoothly cyclized ($AlCl_3$ in dichloro-



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methane) to give a 4:1 mixture of the diastereoisomers of *6*.

Summarizing, the easily accessible morpholine enamide *2* has been found to be an interesting building block for the synthesis of 2- and 3-arylmorpholine derivatives and *N*-(2-hydroxyethyl)-*N*-(2,2-diarylethyl)amides.

EXPERIMENTAL

General procedure for amidoalkylation. The alkylating agent was dissolved in benzene, *p*-xylene or mesitylene or in a mixture of dichloromethane and the aromatic compound. The resulting solution was added dropwise to a vigorously stirred mixture of the aromatic compound, or dichloromethane and aluminium chloride. In some cases the reaction mixture was chilled with ice during the addition. After complete reaction, as monitored by GLC analysis, the reaction mixture was hydrolyzed with an equal volume of water. To avoid troublesome precipitates and vigorous reaction, the hydrolysis was best carried out by pouring the reaction mixture into a stirred mixture of ice and water. The layers were separated and the aqueous one was extracted twice with dichloromethane. The organic layers were combined and washed with water, saturated sodium hydrogen carbonate solution and finally with water. After drying over magnesium sulfate, the solvent/excess nucleophiles was removed by evaporation *in vacuo*. The isolated product was obtained by recrystallization or, when necessary, distillation of the crude oil. In one case polymeric material was removed by column chromatography on silica gel. The course of the reactions and the purity of the isolated products were checked on a Hewlett-Packard HP-5830 instrument equipped with a Dexsil 300 column (0.5 m × 0.3 cm) or, in order to separate 2- and 3-phenyl-4-morpholinecarboxaldehyde, a 10 % Carbowax column (3 m × 0.3 cm). ^{1}H NMR spectra were recorded on a Jeol 100 MHz instrument or on a Nicolet 360 WB spectrometer using $CDCl_3$ as solvent. MS analyses were carried out on a Finnigan 4021 spectrometer at 70 eV. In some cases the product was distilled using a Kugelrohr apparatus, commercially available from Aldrich Co.

The morpholine derivatives *1* and *2* were prepared according to published methods.[6,9] Compound *1* is also commercially available from SynthElec AB, P.O. Box 1128, S-221 04 Lund, Sweden (see also *J. Org. Chem. 48* (1983) No. 2, p. 7A).

Phenyl-4-morpholinecarboxaldehyde (*3* and *4*, R=Ph). Compound *1* (0.5 mol) dissolved in benzene (100 ml) was added dropwise to a stirred and boiling mixture of $AlCl_3$ (1.0 mol) in benzene (400 ml). The reaction mixture was refluxed for 30 min and worked up as described above. The product was distilled at reduced pressure using a 25×1.5 cm Vigreux column. The product was a 72:28 mixture of 3- and 2-phenyl isomers, yield 61.6 g (64 %), b.p. 137–147 °C/0.7–0.8 mmHg. NMR and MS data for both isomers are given below.

3-Phenyl-4-morpholinecarboxaldehyde (*3*, R=Ph). Compound *1* (0.1 mol) in benzene (20 ml) was added dropwise to a stirred and hot mixture of $AlCl_3$ (0.4 mol) in benzene (100 ml). The reaction mixture was refluxed for 24 h and worked up as usual. The product was distilled at reduced pressure from a Claisen flask, yield 10.9 g (57 %), b.p. 130–134 °C/0.4 mmHg. MS *m/e* (% rel. int.): 192 (5, M+1) 191 (37, M), 190 (18), 162 (22, M–CHO), 160 (21), 148 (10), 132 (27), 118 (11), 105 (35), 104 (100, $C_6H_5CHCH_2$), 91 (33) 78 (36), 77 (39). ^{1}H NMR: δ 3.21–4.04 (5H, m), 4.20–4.59 (1H, m), 4.47–4.56 and 5.37–5.46 (1H, 2d, *J* 3.5 Hz), 7.12–7.57 (5H, m), 8.09 and 8.15 (1H, 2s).

The same product was also isolated from a 2.6:1 mixture of 3-phenyl and 2-phenyl-4-morpholinecarboxaldehyde (see preparation of phenyl-4-morpholinecarboxaldehyde): The mixture (0.2 mol) was dissolved in benzene (35 ml) and added to a stirred mixture of $AlCl_3$ (0.2 mol) in benzene (140 ml). The resulting solution was refluxed and its composition was periodically checked by GLC analysis. After 12 h the isomer ratio had stabilized itself at 5:1 in favour of the 3-phenyl isomer and additional $AlCl_3$ (0.05 mol) was added. After another 48 h at reflux temperature the 2-phenyl isomer had been consumed and the reaction mixture was worked up as usual. The 3-isomer was isolated by distillation at reduced pressure, yield 21.9 g (57 %), b.p. 124–128 °C/0.25 mmHg.

2-Phenyl-4-morpholinecarboxaldehyde (*4*, R=Ph). Compound *2* (0.15 mol), dissolved in benzene (25 ml), was added slowly to a stirred and ice-cooled mixture of $AlCl_3$ (0.15 mol) in benzene (110 ml). The rate of addition was

adjusted to keep the reaction temperature below 8 °C (this reaction was sometimes, especially on the 0.1 molar scale, accompanied by the formation of troublesome precipitates, causing decreased yields; the problem was minimized when the addition was performed dropwise with efficient stirring. After 6 h at 0 °C the reaction mixture was stirred for 24 h at room temperature. GLC analysis showed substantial amounts of unreacted enamide and therefore another portion of $AlCl_3$ (0.05 mol) was added (at 0 °C). The starting material was completely consumed after another 12 h at room temperature. After the usual work-up the crude oil was finally distilled at reduced pressure. From the distillation residue, *N,N*-(2,2-diphenylethyl)(2-hydroxyethyl)formamide (9 % yield) was isolated by recrystallization from benzene. The yield of *4*, R=Ph was 13.4 g (47 %), b.p. 157–172 °C/1.5–2.5 mmHg.

MS *m/e* (% rel. int.): 191 (20, M), 190 (6, M–H), 162 (4, M–CHO), 132 (8), 105 (16, C_7H_5O), 104 (14), 91 (6), 85 (100, C_4H_7NO), 77 (18), 57 (42), 56 (28).

^1H NMR: δ 2.59–3.78, 3.95–4.25 and 4.27–4.54 (7H, m), 7.24–7.40 (5H, apparently a s), 8.09 (1H, s).

2-(2,4,6-Trimethylphenyl)-4-morpholinecarboxaldehyde (*4*, R=2,4,6-Me_3-phenyl). Compound *2* (0.1 mol) was dissolved in mesitylene (0.11 mol) and dichloromethane (20 ml) and was added dropwise to an ice-cooled and stirred mixture of $AlCl_3$ (0.1 mol) in dichloromethane (80 ml). After the addition the mixture was stirred for 6 h at room temperature. The work-up was performed as usual. The crude oil was distilled in a Kugelrohr apparatus. The main fraction was dissolved in ether (15 ml) and chilled (4 °C) and the solid product was filtered off and washed with ether. Additional product was recovered from the evaporated mother liquor when dissolved in a 1:1 mixture of ether/ligroin and chilled (–25 °C). Yield, 16.0 g after distillation (slightly impure) and 10.9 g (47 %) after recrystallization. The main fraction was collected at 130–160 °C/0.1–0.2 mmHg, m.p. 83–87 °C.

MS *m/e* (% rel. int.): 233 (7, M), 147 (7, $C_{10}H_{11}O$), 119 (5, C_9H_{11}), 91 (6), 86 (15), 85 (100, C_4H_7NO), 57 (55), 56 (37). ^1H NMR: δ 2.22 (3H, s), 2.40 (6H, s), 2.79–3.72 (4H, m), 3.95–4.38 (2H, m), 4.59–4.81 (1H, m), 6.77 (2H, s), 8.03 and 8.06 (1H, 2s).

3-(2,4,6-Trimethylphenyl)-4-morpholinecarboxaldehyde (*3* R=2,4,6-Me_3-phenyl). Compound *1* (0.1 mol) and mesitylene (0.15 mol) were dissolved in dichloromethane (20 ml) and added to a stirred mixture of $AlCl_3$ (0.2 mol) in dichloromethane (80 ml). The solution was allowed to stand for 12 h at room temperature before being worked up as usual. The crude product was dissolved in ligroin/ether (10+10 ml) and the product precipitated when cooled (4 °C). The crystals were filtered off and washed with ether, yield 15.6 g (67 %), m.p. 84–87 °C.

MS *m/e* (% rel. int.): 233 (10,M), 218 (9, M–CH_3), 188 (13), 160 (15), 146 (40), 131 (16), 114 (100, M–mesityl), 105 (21), 91 (23), 77 (17), 72 (75). ^1H NMR: δ 2.26 (3H, s), 2.37 (6H, s), 2.83–3.20 (1H, t with additional splitting, *J* 12 and 4 Hz resp.), 3.90–413 (3H, m), 4.20–4.41 (1H, br d, *J* 13.3 Hz), 4.82–5.01 (1H, dd, *J* 10.3 and 4.4 Hz), 6.88 (2H, s), 7.86 (1H, s).

2-(2,5-Dimethylphenyl)-4-morpholinecarboxaldehyde (*4*, R=2,5-Me_2-phenyl). *p*-Xylene (0.11 mol) and *2* (0.1 mol) were dissolved in dichloromethane (20 ml) and slowly added to an ice-cooled and stirred mixture of $AlCl_3$ (0.1 mol) in dichloromethane (80 ml). The reaction mixture was kept at 0 °C for 3 h and then at room temperature for 18 h.

The product was isolated by distillation in a Kugelrohr apparatus at reduced pressure, yield 3.97 g (18 %), b.p. 130–180 °C at 0.2 mmHg; GLC analysis showed a 100 % pure product.

MS *m/e* (% rel. int.): 219 (8, M), 133 (7, C_9H_9O), 105 (7, C_8H_9), 91 (9), 85 (100, C_4H_7NO), 57 (72), 56 (45). ^1H NMR: δ 2.29 (6H, s), 2.54–4.59 (7H, several coincident m), 7.01 (2H, narrow m), 7.26 (1H, narrow m), 8.08 and 8.10 (1H, 2s).

N,N-*(2,2-Diphenylethyl)(2-hydroxyethyl)formamide* (*5*, R=Nu=Ph). Compound *2* (0.05 mol) dissolved in benzene (10 ml) was added dropwise to a boiling mixture of $AlCl_3$ (0.1 mol) in benzene (40 ml). After 40 min at reflux temperature the mixture was allowed to stand at room temperature for 3 h before work-up.

The crystalline residue, after evaporation of the solvents, was recrystallized from benzene (25 ml), yield 9.1 g (68 %), m.p. 109–111 °C.

MS *m/e* (% rel. int.): 269 (6, M), 238 (3), 181 (16), 180 (74, M–$C_3H_7NO_2$), 168 (16), 167 (98, $C_{13}H_{11}$), 166 (16), 165 (38), 152 (16), 102 (26, M–$C_{13}H_{11}$), 91 (10), 77 (10), 74 (100), 56 (32). ^1H NMR (360 MHz): δ 2.54 and 3.07 (1H, 2t, *J* 6.2 Hz), 3.01 and 3.43 (2H, 2t, *J* 5.1 Hz), 3.53 and 3.73 (2H, apparently 2 q, *J* 5.1 and 6.2 Hz), 3.93 and 3.97 (2H, 2d, *J* 8.6 Hz), 4.21 and 4.45 (1H, 2t, *J* 8.6 Hz), 7.15–7.42 (10H, m), 7.71 and 7.96 (1H, 2s).

N,N-*[2,2-Di(2,5-dimethylphenyl)ethyl](2-hydroxyethyl)formamide* (*5*, R=Nu=2,5-Me_2-phenyl). Compound *2* (0.05 mol) was dissolved in *p*-xylene (12 ml) and added to a stirred mixture of $AlCl_3$ (0.1 mol) in *p*-xylene (50 ml). The reaction mixture was stirred for 24 h at room temperature and worked up as usual. The crude product was

recrystallized from ether (10 ml), yield 9.2 g (56 %), m.p. 116–118 °C.

MS *m/e* (% rel. int.): 325 (2,M), 236 (25, M–$C_3H_7NO_2$), 223 (100, $C_{17}H_{19}$), 208 (12, 193 (20), 119 (13), 102 (17, M–$C_{17}H_{19}$), 91 (12), 74 (43, $C_3H_8NO)_1$ 56 (70).

^{1}H NMR (360 MHz): δ 2.22, 2.25, 2.28 and 2.30 (6H, 4s), 2.62 and 2.88 (1H, indistinct t), 3.04 and 3.41 (2H, 2t, *J* 5 Hz), 3.57 and 3.68 (2H, 2m), 3.83 and 3.87 (2H, 2d, *J* 7.3 Hz), 4.45 and 4.73 (1H, 2t, *J* 7.3 Hz), 6.90–705 (6H, m), 7.72 and 8.01 (1H, 2s).

N,N-*(2-Hydroxyethyl)[2-phenyl-2-(2,4,6-trimethylphenyl)ethyl]formamide* (*5*, R=Ph, Nu= 2,4,6-Me_3-phenyl). 2-Phenyl-4-morpholinecarboxaldehyde (0.025 mol) was dissolved in mesitylene (7 ml) and added to a stirred mixture of $AlCl_3$ (0.0375 mol) in mesitylene (28 ml). The dark red two-phase system was heated to 55 °C for 3 h. The product was recrystallized from benzene (7 ml), yield 5.66 g (73 %), m.p. 132–134 °C.

MS *m/e* (% rel. int., direct inlet): 311 (3, M), 222 (20, M–$C_3H_7NO_2$), 209 ($C_{16}H_{17}$), 194 (10), 179 (18), 102 (13, M–$C_{16}H_{17}$), 91 (15), 74 (100, C_3H_8NO), 56 (50).

^{1}H NMR (360 MHz): δ 2.14 (6H, br s), 2.25 and 2.26 (3H, 2s), 2.63–5.06 (8H, several coincident m), 6.79 (2H, br s), 7.00–7.35 (5H, m), 7.79 and 8.00 (1H, 2s). Due to complex and coincident signals the spectra could not be completely resolved.

N,N-*[2,5-Dimethylphenyl)-2-phenylethyl](2-hydroxyethyl)formamide* (*5*, R=Ph, Nu=2,5-Me_2-phenyl). 2-Phenyl-4-morpholinecarboxaldehyde (0.025 mol) dissolved in *p*-xylene (6 ml) was added to a stirred mixture of $AlCl_3$ (0.0375 mol) in *p*-xylene (25 ml). After 1 h at room temperature the reaction mixture was worked up as usual. The crude product was dissolved in a minimum amount of ligroin/ether (v/v=1/1) at reflux temperature and chilled (–25 °C). The solid product was filtered off and washed with ligroin/ether, yield 3.56 g (48 %), m.p. 68–73 °C. MS *m/e* (% rel. int., direct inlet): 297 (2, M), 208 (17, M–$C_3H_7NO_2$), 195 (42, $C_{15}H_{15}$), 180 (10), 165 (13), 102 (10, M–$C_{15}H_{15}$), 91 (10), 74 (100), 56 (47).

^{1}H NMR (360 MHz): δ 2.18, 2.20, 2.32 and 2.35 (6H, 4s), 2.86–2.95, 2.95–3.13 and 3.45–3.60 (3H, m), 3.43 and 3.72 (2H, 2t, *J* 4.2 Hz), 3.82–4.03 (2H, m), 4.31 and 4.62 (1H, 2t, *J* 7.4 Hz), 6.86–7.37 (8H, m), 7.61 and 7.95 (1H, 2s).

N,N-*(2-Chloroethyl)(2,2-diphenylethyl)formamide.* Thionyl chloride (0.28 mol) was added to a solution of *N,N*-(2,2-diphenylethyl)(2-hydroxyethyl)formamide (0.171 mol) in dichloromethane (250 ml). The mixture was refluxed for 2 h and the solvent and excess thionyl chloride was removed by evaporation *in vacuo*. The solid residue was recrystallized from ethanol (30 ml), yield 40.5 g (82 %), m.p. 83–86 °C.

MS *m/e* (% rel. int., direct inlet): 290 and 288 (0.5 and 1.5, m+1), 289 and 287 (0.7 and 2, M), 180 (45, M–C_3H_6ClNO), 167 (100, $C_{13}H_{11}$), 165 (30), 152 (17), 122 and 120 (6 and 18, M–$C_{13}H_{11}$), 94 (18, C_3H_7ClN), 92 (55, C_3H_7ClN) 74 (18) 56 (30). The poduct was only identified by MS analysis.

N,N-*(2,2-Diphenylethyl)(ethenyl)formamide. N,N*-(2-Chloroethyl)(2,2-diphenylethyl)formamide (0.025 mol) was dissolved in DMSO (25 ml, freshly distilled and dried over molecular sieves, 3 Å) and potassium *t*-butoxide (0.0275 mol, sublimed *in vacuo* at 1 mmHg) was added to the stirred solution. After 1 h at room temperature, the starting material had been consumed and water (50 ml) was added. The resulting mixture was extracted twice with dichloromethane (2×50 ml) and the combined organic layers were washed with water (3×50 ml). The dichloromethane solution was dried over magnesium sulfate and the solvent was removed by evaporation *in vacuo*. Hexane (150 ml) was added to the crude product and after reflux the solution was decanted and cooled off (–25 °C). The solid product was filtered off and washed with hexane, yield 3.3 g (53 %).

MS *m/e* (% rel. int.): 251 (4, M), 206 (3), 180 (48, M–C_3H_5NO), 167 (100, $C_{13}H_{11}$), 165 (38), 152 (19), 84 (23, M–$C_{13}H_{11}$), 77 (12), 56 (52). The product was only identified by MS analysis.

3,4-Dihydro-1-methyl-4-phenyl-2(1H)-isoquinolinecarboxaldehyde (*6*). *N,N*-(2,2-Diphenylethyl(ethenyl)formamide (0.0252 mol) dissolved in dichloromethane (10 ml) was added to a stirred mixture of $AlCl_3$ (0.05 mol) in dichloromethane (15 ml). After 2 h at room temperature water (25 ml) was added and the work-up was performed as usual. The filtrate, after drying, was contaminated by a finely divided precipitate and dark-coloured polymeric material. These were removed from the product by column chromatography on neutral alumina with dichloromethane as eluent. The product, a mixture of the two diastereoisomers in a 4:1 ratio, solidified when the solvent was removed by evaporation *in vacuo*, yield 3.4 g (53 %) m.p. 70–80 °C. MS *m/e* (% rel. int.): 252 (13, M+1) 251 (72, M), 236 (100, M–CH_3), 208 (30, M–CHNO), 179 (87, $C_{14}H_{11}$), 165 (18), 130 (33), 117 (40), 91 (62, C_7H_7), 77 (37, C_6H_5).

^{1}H NMR: δ 1.43–1.72 (3H, 4d, *J* 7 Hz), 3.04–4.24 (3H, m), 4.40–4.82 and 5.37–5.69 (1H, m), 6.65–7.48 (9H, m), 8.09 and 8.23 (1H, s).

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