Monatshefte für Chemie 126, 725–732 (1995)

Monatshefte für Chemie
Chemical Monthly

Synthesis of 2-(2-Methoxyethyl)- and 2-(2-Thiomethoxyethyl)-aniline and Related Compounds

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Summary. 2-(2-Nitrophenyl)-ethanol (**2**) was methylated with dimethyl sulfate to give 2-(2-methoxyethyl)-1-nitrobenzene (**3a**) which then was reduced with hydrazine hydrate in the presence of *Raney* nickel to 2-(2-methoxyethyl)-aniline (**1a**). Compound **1a** can be transformed into the N-monosilylated derivative **4** by lithiation with *n*-butyllithium and subsequent reaction with chlorotrimethylsilane. Reaction of **2** with *p*-toluenesulfonyl chloride yields 2-(2-nitrophenyl)-ethyl *p*-toluenesulfonate (**5**), which reacts with sodium thiomethoxide to give 2-(2-thiomethoxyethyl)-1-nitrobenzene (**3b**). **3b** was reduced with hydrazine hydrate in the presence of *Raney* nickel to yield 2-(2-thiomethoxyethyl)-aniline (**1b**). Ethyl (2-nitrophenyl)-acetate (**6**) could be dimethylated with methyl iodide in the presence of potassium *tert*-butoxide and 18-crown-6 to give ethyl 2-methyl-2-(2-nitrophenyl)-propionate (**7**). Reduction of **7** with lithium borohydride yields 2,3-dihydro-3,3-dimethyl-1*H*-indole (**9**) and 2-[(1-hydroxy-2-methyl)-2-propyl]-aniline (**10**).

Keywords. 2-Functionalized anilines; Hemilabile ligands; Imido ligands.

Synthese von 2-(2-Methoxyethyl)- und 2-(2-Thiomethoxyethyl)-anilin und verwandten Verbindungen

Zusammenfassung. 2-(2-Nitrophenyl)-ethanol (**2**) wurde mit Dimethylsulfat zu 2-(2-Methoxyethyl)-1-nitrobenzol (**3a**) methyliert, das sich mit Hydrazinhydrat in Gegenwart von *Raney*-Nickel zu 2-(2-Methoxyethyl)-anilin (**1a**) reduzieren läßt. Verbindung **1a** kann durch Metallierung mit *n*-Butyllithium und anschließende Reaktion mit Chlortrimethylsilan in das *N*-monosilylierte Derivat **4** umgewandelt werden. Reaktion von **2** mit *p*-Toluolsulfonylchlorid ergab 2-(2-Nitrophenyl)-ethyl-*p*-Toluolsulfonat (**5**), das mit Natriumthiomethanolat zu 1-Nitro-2-(2-thiomethoxyethyl)-benzol (**3b**) reagiert. **3b** wurde mit Hydrazinhydrat in Gegenwart von *Raney*-Nickel zu 2-(2-Thiomethoxyethyl)-anilin (**1b**) reduziert. Ethyl-2-(nitrophenyl)-acetat (**6**) kann mit Methyliodid in Gegenwart von Kalium-*tert*-butoxid und 18-Krone-6 zu Ethyl-2-methyl-2-(2-nitrophenyl)-propionat (**7**) dimethyliert werden. Reduktion von **7** mit Lithiumborhydrid lieferte 2,3-Dihydro-3,3-dimethyl-1*H*-indol (**9**) und 2-[(1-Hydroxy-2-methyl)-2-propyl]-anilin (**10**).

Introduction

Currently, hemilabile-functionalized spectator ligands are attracting a lot of attention [1]. In the course of our investigations concerning hemilabile aryl imido

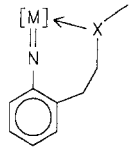
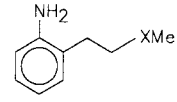


Fig. 1. Intramolecular stabilization of a reactive metal-ligand fragment in imido complexes based on anilines of type **1**

ligands [2] we have been interested in the synthesis of potential precursors derived from anilines bearing a pendant electron pair donor group *X* in the 2-position. In this respect, the anilines **1a, b**, which carry the chemically robust methoxy and thiomethoxy unit, respectively, are particularly attractive.



1 (a: X=O, b: X=S)

The two-carbon tether is expected to have the appropriate length for allowing the intramolecular stabilization of a reactive metal-ligand fragment attached to the imido unit by virtue of its electron pair donor group *X* (Fig. 1).

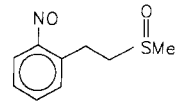
Furthermore, since it seems desirable to be able to influence the energetics of this interaction by using *Pearson*'s HSAB principle [3], the oxygen/sulfur variation should be of particular interest.

Results and Discussion

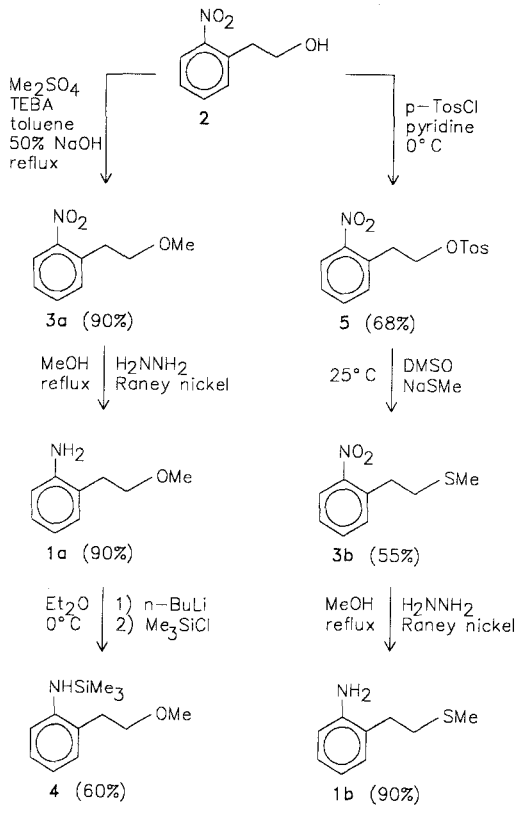
In this paper, we report on the synthesis of the hitherto unknown anilines **1a** and **1b** and of related compounds which were synthesized starting from commercially available 2-(2-nitrophenyl)-ethanol (**2**). The syntheses are outlined in Scheme 1.

The alcohol **2** can be cleanly converted to the corresponding methyl ether **3a** by a classical *Williamson* synthesis performed under PTC conditions [4]. It is essential to use dimethyl sulfate instead of methyl iodide as methylating agent; with the latter, benzylic C-alkylation becomes an important side reaction [5]. An alternative route using a large excess of diazomethane as methylating agent gave yields inferior to the *Williamson* reaction. The resulting 2-(2-methoxyethyl)-1-nitrobenzene (**3a**) is reduced to the desired aniline in excellent yield by using hydrazine hydrate and *Raney* nickel in refluxing methanol. The aniline **1a** can be monosilylated by deprotonation with one equivalent of *n*-butyllithium and subsequent reaction with one equivalent of chlorotrimethylsilane. In analogy to **1a** and **3a**, the monosilylated aniline **4** is obtained as a pale yellow liquid after vacuum distillation.

The synthesis of **1b** (the sulfur analogue of **1a**) involves transformation of the alcohol **2** into the corresponding tosylate **5**; this can be achieved by use of standard conditions. Compound **5** is isolated as white crystals [6]. Treatment of **5** with sodium thiomethoxide in *DMSO* gives 1-nitro-2-(2-thiomethoxyethyl)-benzene (**3b**) [7]. Not unexpectedly, no intramolecular oxygen transfer from nitrogen to sulfur takes place in this compound [8].



3b'

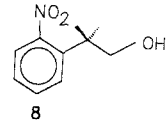


Scheme 1. Synthesis of anilines **1a** and **1b** and of the silyl derivative **4**

The identity of the nitro isomer **3b** (as opposed to the nitroso isomer **3b'**) was confirmed by ^{13}C NMR and IR spectroscopy. The *ipso* carbon atom of **3b** shows a ^{13}C NMR signal at 149.2 ppm, which is very close to the value observed in the case of nitrobenzene (148.4 ppm) [9]; the *ipso* carbon atom of nitrosobenzene resonates at 166.8 ppm [10]. The IR spectrum of **3b** does *not* exhibit an NO stretching mode at $1500 \pm 10\,cm^{-1}$ characteristic of aromatic nitroso compounds; instead, an intense band is observed at $1525\,cm^{-1}$, which is characteristic for an aromatic nitro compound [11].

3b is reduced to the desired aniline **1b** employing conditions essentially identical with those for the preparation of the oxygen analogue **1a**; in contrast to observations made by others [12], no desulfurisation occurs under the reaction conditions applied, and **1b** is obtained in excellent yield.

As a sideline of these investigations, we have also tried to block the benzylic position in such compounds by introducing two methyl groups, since it is anticipated that the relatively high acidity of the benzylic hydrogen atoms might cause some problems at a later stage of this work. A dimethylation can be achieved very conveniently by the procedure developed by *Prasad et al.* [13], starting from ethyl (2-nitrophenyl)-acetate (**6**) [14] and using potassium *tert*-butoxide in the presence of 18-crown-6 as deprotonating agent and methyl iodide as methylating agent; the desired dimethylated ester **7** is obtained in 88% yield after chromatographic work-up [15]. Unfortunately, we have not been able so far to obtain the alcohol **8** by selective reduction of **7**.



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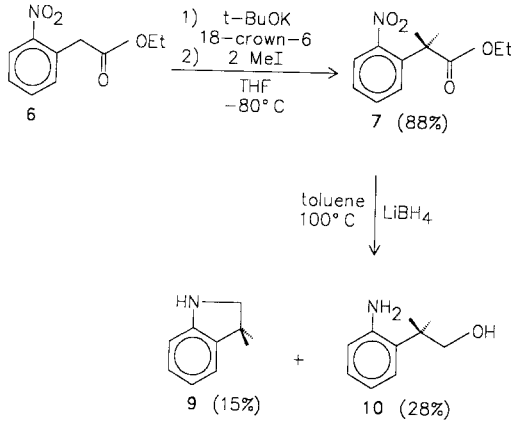
With sodium borohydride, either as such or in the presence of anhydrous calcium chloride [16] or aluminium chloride [17], no reaction is observed. With lithium borohydride in *THF*, a mixture of products is obtained; vacuum distillation leads to the isolation of 2,3-dihydro-3,3-dimethyl-1*H*-indole (**9**) and 2-[(1-hydroxy-2-methyl)-2-propyl]-aniline (**10**) in 15 and 28% yield, respectively (Scheme 2). Reduction with aluminium hydride in *THF* gives a similar mixture of products.



Scheme 2. Synthesis of compounds **9** and **10**

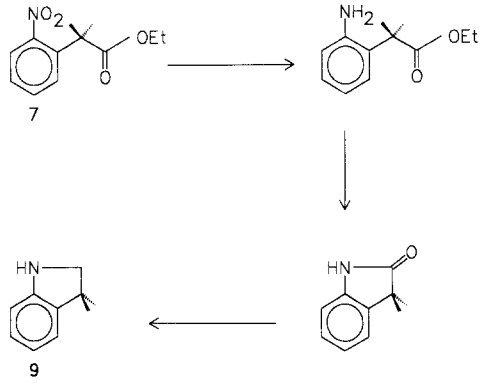


Fig. 2. Proposed mechanism for the formation of **9**

Whereas the formation of **10** can be explained quite easily (reduction of both functional groups), the formation of **9** is somewhat surprising. We propose the mechanism shown in Fig. 2.

The nitro group of **7** is reduced to the amino function, and an intramolecular amide formation takes place; the amide is then reduced to the corresponding amine **9**. Attempts to O-methylate the alcohol **10** to form the dimethylated analogue of **1a** are under way.

Experimental

Air- and/or moisture-sensitive compounds were handled under an atmosphere of purified argon by using standard *Schlenk* techniques. NMR: Bruker AM 300 (300.133 MHz, ^{1}H, ext. *TMS*; 75.453 MHz, ^{13}C, ext. *TMS*; 59.595 MHz, ^{29}Si, ext. *TMS*); MS: VG Autospec (70 eV); only characteristic fragments are listed; IR: Mattson Polaris FTIR; Elemental analyses: Microanalytical laboratory, Universität Bielefeld.

2-(2-Methoxyethyl)-1-nitrobenzene (**3a**)

15.3 g (91.2 mmol) of 2-(2-nitrophenyl)-ethanol (**2**) is dissolved in 320 ml of toluene. 248 mg (0.91 mmol) of benzyltriethyl ammonium bromide (*TEBA*), 23.0 g (182 mmol) of dimethyl sulfate, and 200 ml of an aqueous solution of sodium hydroxide (50%) are added to the solution, and the mixture is stirred under reflux for 6 h. After cooling to room temperature, the organic layer is separated and the aqueous layer is extracted with toluene (2 × 100 ml). The combined organic layers are dried with sodium sulfate. Volatiles are removed *in vacuo*, and the remaining brownish oil is distilled to give 14.8 g (90%) of a pale yellow liquid, b.p. 69 °C (0.05 mbar). ^{1}H NMR ($CDCl_3$): $\delta = 3.16$ (t, $^3J = 6.5$ Hz, 2H, $ArCH_2$), 3.31 (s, 3H, OCH_3), 3.64 (t, $^3J = 6.5$ Hz, 2H, CH_2O), 7.32–7.40 (m, 2H, ArH), 7.51 (m, 1H, ArH), 7.89 (m, 1H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 33.0$ ($ArCH_2$), 58.8 (OCH_3), 72.0 (CH_2O), 124.5, 127.3, 132.5, 132.6 (arom. CH), 133.9 (arom. CCH_2) 149.6 (arom. CNO_2); IR (neat): $\nu = 1520$ vs, 1340 s, 1120 s cm^{-1}; MS (CI, isobutane carrier gas): m/z (%) = 182 [$M^+ + H$] (10), 150 [M^+-OCH_3] (100); $C_9H_{11}NO_3$ (181.2); calcd.: C 59.66, H 6.11, N 7.73; found: C 59.65, H 5.85, N 7.74.

2-(2-Methoxyethyl)-aniline (**1a**)

Raney nickel is prepared by adding 11.9 g (298 mmol) of sodium hydroxide pellets to a suspension of 4.07 g of Ni/Al alloy in 120 ml of water; the mixture is stirred at room temperature and then at 80 °C for 90 min each, decanted, washed with water (3 × 40 ml) and finally with methanol (2 × 40 ml). A solution of 19.0 g (105 mmol) of **3a** in 75 ml of methanol is added to the catalyst and the mixture is heated to reflux. Then 54 ml of hydrazine hydrate is added dropwise, and the mixture is stirred under reflux for 1 h. After cooling to 0 °C, the catalyst is filtered off and washed with methanol (2 × 50 ml). The solvent is removed *in vacuo*, the resultant oil is dissolved in 50 ml of dichloromethane, the solution is washed with water (2 × 10 ml) and dried with sodium sulfate. Volatiles are removed *in vacuo*, and the crude product is distilled to yield 14.2 g (90%) of a pale yellow liquid, b.p. 60 °C (0.2 mbar). ^{1}H NMR ($CDCl_3$): $\delta = 2.78$ (t, $^3J = 6.3$ Hz, 2H, $ArCH_2$), 3.33 (s, 3H, OCH_3), 3.61 (t, $^3J = 6.3$ Hz, CH_2O), 3.95 (br. s, 2H, NH_2), 6.66–6.74 (m, 2H, ArH), 7.01–7.06 (m, 2H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 32.3$ ($ArCH_2$), 58.7 (OCH_3), 73.4 (CH_2O), 115.8, 118.5, 127.3, 130.2 (arom. CH), 124.7 (arom. CCH_2), 145.4 (arom. CNH_2); IR (neat): $\nu = 3435$ vs, 3350 vs, 3020 s, 2930 s, 2880 s, 1625 vs, 1580 s, 1500 s, 1100 s cm^{-1}; MS (CI, NH_3 carrier gas): m/z (%) = 257 [$M_2^+ + H$] (23), 152 [$M^+ + H$] (100), 151 [M^+] (57), 120 [M^+-OCH_3] (16), 106 [M^+-CH_2OCH_3] (47); $C_9H_{13}NO$ (151.2); calcd.: C 71.42, H 8.60, N 9.26; found: C 71.31, H 8.55, N 9.12.

2-(2-Methoxyethyl)-N-trimethylsilylaniline (**4**)

46.0 ml of a 1.59 M solution of *n*-butyllithium in hexane (73.1 mmol) is added dropwise with stirring to a solution of 11.0 g (72.7 mmol) of **1a** in 80 ml of diethyl ether at 0 °C. The mixture is stirred at room temperature for 1 h and then again cooled to 0 °C. 7.90 g (72.7 mmol) of trimethylsilyl chloride are added and the mixture is stirred at room temperature overnight. The precipitate (LiCl) is filtered off and washed with diethyl ether (2 × 5 ml). Volatiles are removed *in vacuo*, and the remaining brownish oil is distilled to give 9.62 g (60%) of a pale yellow liquid, b.p. 51–52 °C (0.01 mbar). ^{1}H NMR ($CDCl_3$): $\delta = 0.35$ (s, 9H, $SiMe_3$), 2.84 (t, $^3J = 6.5$ Hz, 2H, $ArCH_2$), 3.42 (s, 3H, OCH_3), 3.66 (t, $^3J = 6.5$ Hz, 2H, CH_2O), 4.16 (br. s, 1H, NH), 6.75 (m, 1H, ArH), 6.84 (m, 1H, ArH), 7.08–7.16 (m, 2H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 0.2$ ($SiMe_3$), 32.8 ($ArCH_2$), 58.6 (OCH_3), 73.0 (CH_2O), 115.8, 117.6, 127.3, 130.3 (arom. CH), 125.9 (arom. CCH_2), 146.0 (arom. $CNHSiMe_3$); ^{29}Si NMR ($CDCl_3$): $\delta = 2.6$; IR (neat): $\nu = 3300$ s, 2850 vs, 1610 s, 1590 s, 1120 s cm^{-1}; MS (CI, isobutane carrier gas): m/z (%) = 223 [M^+] (16), 208 [M^+-CH_3] (7), 192 [M^+-OCH_3] (7), 178 [M^+-CH_2OCH_3], 73 [$SiMe_3^+$] (14), 59 [$CH_2OCH_3^+$] (100); $C_{12}H_{21}NOSi$ (223.4); calcd.: C 64.52, H 9.47, N 6.27; found: C 64.30, H 9.19, N 6.30.

2-(2-Nitrophenyl)-ethyl-p-toluenesulfonate (**5**)

A solution of 4.64 g (24.3 mmol) of *p*-toluenesulfonyl chloride in 20 ml of pyridine is added dropwise with stirring to a solution of 2.94 g (19.4 mmol) of 2-(2-nitrophenyl)-ethanol in 25 ml of pyridine cooled to 0 °C. The mixture is stirred at 0 °C for 2 h and subsequently hydrolyzed by adding 200 ml of ice-cold water. The precipitate is filtered off and dried *in vacuo* to give 4.00 g (68%) of a white, microcrystalline powder, m.p. 48 °C. ^{1}H NMR ($CDCl_3$): $\delta = 2.39$ (s, 3H, CH_3), 3.22 (t, $^3J = 6.3$ Hz, 2H, CH_2O), 4.31 (t, $^3J = 6.3$ Hz, 2H, $ArCH_2$), 7.25 (m, 2H, ArH), 7.33–7.41 (m, 2H, ArH), 7.52 (m, 1H, ArH), 7.63 ("d", 2H, ArH), 7.90 (m, 1H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 21.6$ (CH_3), 33.1 ($ArCH_2$), 69.3 (CH_2O), 125.1, 127.8, 128.2, 129.8, 133.3, 133.4 (arom. CH), 131.7, 132.9 (quart. arom. C), 144.8 (arom. CSO_3), 149.1 (arom. CNO_2); IR (KBr): $\nu = 1610$ m, 1600 m, 1530 s, 1350 s, 1340 s, 1170 s cm^{-1}; MS (EI): m/z (%) = 275 [M^+-NO_2] (2), 155 [M^+-$SO_2C_7H_7$] (66), 136 [$H_2CC_6H_4NO_2^+$] (67), 91 [$C_7H_7^+$] (100); $C_{15}H_{15}NO_5S$ (321.4); calcd.: C 56.06, H 4.70, N 4.35; found: C 55.93, H 4.80, N 4.58.

1-Nitro-2-(2-thiomethoxyethyl)-benzene (**3b**)

A solution of 3.72 g (51.6 mmol) of sodium thiomethoxide in 70 ml of *DMSO* is added dropwise with stirring to a solution of 15.8 g (51.6 mmol) **5** in 30 ml of *DMSO*. The mixture is stirred at room temperature for 1 h, hydrolyzed with 400 ml of ice-cold water, and the aqueous layer extracted with diethyl ether (4 × 30 ml). The combined organic layers are dried with sodium sulfate and volatiles are removed *in vacuo*. The crude product is distilled to yield 6.11 g (60%) of **3b** as an orange oil, b.p. 87–89 °C (0.01 mbar). ^{1}H NMR ($CDCl_3$): $\delta = 2.12$ (s, 3H, SCH_3), 2.76 (m, 2H, CH_2S), 3.14 (m, 2H, $ArCH_2$), 7.34 (m, 2H, ArH), 7.52 (m, 1H, ArH), 7.90 (m, 1H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 15.5$ (SCH_3), 33.3, 34.6 (CH_2CH_2), 124.8, 127.5, 132.4, 133.0 (arom. CH), 135.3 (arom. CCH_2), 149.2 (arom. CNO_2); IR (neat): $\nu = 1610$ w, 1580 w, 1525 s, 1350 s cm^{-1}; MS (CI, isobutane carrier gas): m/z (%) = 198 [$M^+ + H$] (47), 150 [M^+-SCH_3] (60), 134 [M^+-$OSCH_3$] (100); $C_9H_{11}NO_2S$ (197.3); calcd.: C 54.80, H 5.62, N 7.10; found: C 54.90, H 5.60, N 7.07.

2-(2-Thiomethoxyethyl)-aniline (**1b**)

By a procedure identical to that described for the preparation of **1a**, 3.49 g (90%) of **1b** is obtained from 4.58 g (23.2 mmol) of **3b**; the crude product is already analytically pure. ^{1}H NMR ($CDCl_3$): $\delta = 2.13$ (s, 3H, SCH_3), 2.73–2.81 (m, 4H, CH_2CH_2), 3.67 (br.s, 2H, NH_2), 6.66–6.76 (m, 2H, ArH), 7.02–7.06 (m, 2 H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 15.7$ (SCH_3), 31.5, 33.4 (CH_2CH_2), 115.7, 118.7, 127.4, 129.6 (arom. CH), 124.8 (arom. CCH_2), 144.1 (arom. CNH_2); IR (neat): $\nu = 3450$ s, 3360 s, 3020 m, 2920 s, 2850 m, 1630 s, 1580 s, 1490 s, 1450 s, 1320 s, 1280 s, 1260 s, 1160 s, 1040 m cm^{-1}; MS (FAB+): m/z (%) = 168 [$M^+ + H$] (100), 120 [M^+-SCH_3] (78), 106 [M^+-CH_2SCH_3]; $C_9H_{13}NS$ (167.3); calcd.: C 64.62 H 7.83, N 8.37; found: C 64.20, H 7.86, N 8.32.

Ethyl 2-Methyl-2-(2-nitrophenyl)-propionate (**7**)

A solution of 2.06 g (9.85 mmol) of **6** [14], 3.08 g (21.7 mmol) of methyl iodide, and 0.70 g (2.46 mmol) of 18-crown-6 in 70 ml of *THF* is cooled to −80 °C. Then, 2.65 g (21.7 mmol) of potassium *tert*-butoxide is added to the solution and the mixture is allowed to warm to room temperature over a period of 4 h. After cooling to −80 °C, 30 ml of a saturated aqueous solution of ammonium chloride is added. The mixture is allowed to warm to room temperature and is extracted with dichloromethane (2 × 100 ml). The combined organic layers are washed with water (100 ml) and brine (100 ml) and dried with magnesium sulfate. Volatiles are removed *in vacuo*, and the crude product is purified by chromatography (silica gel, eluent: *n*-hexane/ethyl acetate); yield 2.05 g (88%) of a yellow oil. ^{1}H NMR ($CDCl_3$): $\delta = 1.17$ (t, $^3J = 7.1$ Hz, 3H, CH_2CH_3), 1.65 (s, 6H, $C(CH_3)_2$), 4.11 (q, $^3J = 7.1$ Hz, 2H, CH_2), 7.36–7.42 (m, 1H, ArH), 7.57–7.59 (m, 1H, ArH), 7.88–7.91 (m, 1H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 13.8$ (CH_2CH_3), 27.4

($C(CH_3)_2$), 46.3 ($C(CH_3)_2$), 61.0 (OCH_2), 125.5, 127.6, 128.9, 133.1 (arom. CH), 139.4 (arom. CCH_2), 148.7 (CNO_2), 175.1 (COOEt); IR (neat): $\nu = 2985$ m, 2940 m, 2875 m, 1735 s, 1610 m, 1580 w, 1530 s, 1360 s, 1145 s cm^{-1}; $C_{12}H_{15}NO_4$ (237.3); calcd.: C 60.63, H 6.20, N 6.05; found: C 60.74, H 6.37, N 5.90.

2,3-Dihydro-3,3-dimethyl-1H-indole (**9**) *and 2-[(1-Hydroxy-2-methyl)-2-propyl]-aniline* (**10**)

480 mg (21.4 mmol) of lithium borohydride are dissolved in 30 ml of *THF*. Then, 2.00 g (8.40 mmol) of **7** and 10 ml of toluene are added to the solution, and the *THF* is distilled off at 100 °C. The mixture is stirred at this temperature for 4 h and at room temperature overnight. Volatiles are removed *in vacuo*. Then, 20 ml of water and 50 ml of diethyl ether are added. After equilibration, the organic layer is separated; the aqueous layer is neutralized with ammonium chloride and extracted with diethyl ether (2 × 20 ml). The combined organic layers are dried with sodium sulfate, volatiles are removed *in vacuo*, and the remaining oil is distilled to yield 190 mg (15%) of **9** (b.p. 37 °C (0.001 mbar)), m.p. 34 °C (Ref. [18]: 34 °C)) and 460 mg (28%) of **10** (b.p. 92 °C (0.001 mbar)) as pale yellow oils. The spectroscopic properties of **9** are identical with those reported in the literature (Ref. [19]). Data for **10**: ^{1}H NMR ($CDCl_3$): $\delta = 1.40$ (s, 6H, CH_3): 3.70 (s, 2H, CH_2), 3.75 (br. s, 3H, OH and NH_2), 6.60–6.64 (m, 1H, ArH), 6.77–6.82 (m, 1H, ArH), 7.02–7.08 (m, 1H, ArH), 7.23–7.26 (m, 1H, ArH); ^{13}C NMR ($CDCl_3$): $\delta = 25.3$ (CH_3), 40.1 (CMe_2), 71.3 (CH_2), 118.6, 119.1, 127.3, 128.1 (arom. CH), 131.1 (arom. $CCMe_2$), 144.9 (arom. CNH_2); IR (neat): $\nu = 3360$ s, 2965 s, 2875 m, 1622 s, 1500 s, 1445 s, 1300 m, 1040 s, 750 s cm^{-1}; MS (EI): m/z (%) = 165 [M^+] (41), 134 [M^+-CH_2OH] (100); $C_{10}H_{15}NO$ (165.2); calcd.: C 72.69, H 9.15, N 8.47; found: C 72.66, H 9.22, N 8.30.

Acknowledgments

This work was supported by the *Deutsche Forschungsgemeinschaft*. We would like to thank Dr. *S. Schrader* for helpful comments and discussions. *U.S.* wishes to express his sincere gratitude to Professor *P. Jutzi* for his continued support and interest in this work.

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Received December 5, 1994. Accepted December 9, 1994