Synthesis of C-13-Alkylated 8-Oxoberbines

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C-13-alkylated methoxy-8H-dibenzo[a,g]quinolizin-8-ones 2a-e were synthesized by photocyclization of 1-alkylidene-N-benzoyl-1,2,3,4-tetrahydroisoquinolines 1. Moreover, condensation of 1,2,3,4-tetrahydro-6,7-dimethoxy-1-oxo-isoquinoline with homophthalic acid anhydrides 7a and b leads to the C-13-alkylated 8-oxoberbines 2b and c and improves the yields compared with those of the photocyclization method.

In connection with our investigations of compounds with cytostatic activity and affinity to steroid receptors we synthesized a number of 8-oxoberbines\(^5\). These compounds were either prepared according to Lenz\(^3\) or to Ninomiya\(^4\) by photocyclization or using Haimova's strategy\(^5\). In this paper we describe the synthesis of C-13-alkylated 8-oxoberbines, because the C-13-substituent was regarded as a lipophilic anchor for the estrogen receptor.

a) Photochemical synthesis of C-13-alkylated 8-oxoberbines

C-13-alkyl-8-oxoberbines with one methoxy group in each aromatic ring were obtained only by photocyclization, because alkylated mono-methoxy homophthalic acid anhydrides, necessary according to method b) (see below), are accessible with difficulties only. This photocyclization starts with enamides, often used for the preparation of protobereines\(^3,4\). On account of the formation of a variety of products, quite often the reaction is not controllable because of the excess of energy used during photocyclization, resulting in poor yields and extensive purification. In spite of these handicaps C-13-alkylated 8-oxoberbines can be prepared rather easily by this method\(^6\) and so we synthesized compounds 2a-e according to this procedure.

1-Alkyl-3,4-dihydroisoquinolines 5 obtained via enamides 4 (Scheme 2) are condensed with benzoic acid chlorides 6 affording 1-alkylidene-N-benzoyl-1,2,3,4-tetrahydroisoquinolines 1 which were cyclized to the 8-oxoberbines 2 which were subsequently converted to the acetox derivatives 3 (Scheme 1).

β-(3-Methoxyphenyl)-ethylamine or homoveratrylamine was condensed base catalyzed with the appropriate acid chlorides to get compounds 4. The yields are nearly quantitative and are higher than those of the condensation of these β-phenylethylamines with the pertinent esters\(^7\).

Amides 4 are cyclized to the 1-alkyl substituted 3,4-dihydroisoquinolines 5 by Bischler-Napieralski reaction with POCl\(_3\) in acetonitrile (Scheme 2). The nitritium ion - formed

\(^{5}\) Dedicated to Prof. Dr. Diss. H. Oelschläger, Frankfurt am Main, on the occasion of his 70th birthday appreciating his merits to the development of the Institute of Pharmacy at the University of Regensburg.
zoic acid chlorides. Therefore, we also started from benzoic acid chlorides which were condensed base catalyzed in benzene with compounds 5 affording the 1-alkylidene-N-benzo-3,4-dihydroisoquinolines 1 (Scheme 2). The benzenic solution of the product can be used directly for photocyclization after separation from triethylamine hydrochloride.

The E- and Z-isomers of the enamides 1 arose in different ratios, but in some cases the Z-isomer came up only. Ninomiya(10) explained analogous findings by the different bulkiness of the N-aroyl groups, but also the aromatic protons of the isoquinoline ring system influence the ratio of the isomers(11): C-8-H in ring A of the isoquinoline overlaps according to the Van der Waals-radius in Dreiding-models with the alkyl substituent, and so the formation of the Z-isomer is preferred on account of sterical hindrance (Fig. 1).

These considerations, however, can not fully explain our observations:
1) The C-3'-OCH₃-singlet is shifted upfield in the Z-isomer for 0.2-0.3 ppm, as compared with the E-isomer: when the aroyl rest is rotating around the N-CO-bond the methoxy group is influenced by the alkylidene moiety.
2) Rotation of the aroyl rest around this N-CO-bond leads to broad signals in the 250-MHz-spectra; at -50°C, however, the molecules are "frozen" and well resolved signals can be seen (Fig. 2).
3) In 1-ethyldiene derivatives, not brominated in the aroyl ring system (1a, e.g. - Scheme 1), only the Z-isomer arises because of the interaction described above (Fig. 1). If, however, the aroyl ring system contains bromine (1d, e.g. - Scheme 3) also the E-isomer is formed on account of the rotation of the C-2'-brominated aroyl increment (mono-methoxy substitution: E/Z = 1:1; di-methoxy substitution: E/Z = 3:7).
4) In 1-propyldiene substituted isoquinolines (1c or 1e, e.g.), independent of the bromine substitution at the aroyl ring system, only the Z-isomer is formed; the sterical interaction of the propyldiene group with the aromatic C-8-H is stronger than the sterical hindrance by the substituents of the benzoyl group so preventing the formation of the E-isomer.

Compound 1c/Z-isomer (Fig. 2) exhibits the triplet of the propyldiene group at δ = 0.72 ppm, the pertinent methylene group resonates as a multiplet at δ = 2.02 ppm. One C-3-H leads to a broad signal at δ = 2.82 ppm. The protons of the C-4-methylene group also show a multiplet in the 250-MHz-spectrum at -50°C at δ = 3.22 ppm. As described above the C-3'-methoxy group is shifted upfield, while the other methoxy groups form singlets near δ = 3.95 ppm. The second C-3-H, extremely shifted downfield, resonates as a multiplet at δ = 5.11 ppm. This effect can be explained by the anisotropic effect of the carbonyl group. By this chemical shift the signals of the different isomers can be assigned (Fig. 1): In the Z-isomer of e.g. 1d the methyl-doublet (δ = 1.53 ppm) is shifted upfield as compared to the E-isomer (δ = 1.92 ppm) because of this interaction, while the quartet of the vinylic proton is shifted downfield (Z-isomer: δ = 6.23 ppm, E-isomer: δ = 5.71 ppm). The signals of the aromatic protons in Z-1c come up between δ = 6.6 ppm and 7.2 ppm (Fig. 2).
The mechanisms of the photocyclizations of the enamides, either brominated or not brominated, have been discussed by Lenz\textsuperscript{2,9} or Ninomiya\textsuperscript{4,10}, respectively. We used the oxidative photocyclization of enamides 1, not substituted in the ortho-position, for the preparation of compounds 2a-4. On account of the rotation around the N-CO-bond of the 3,10- and 3,12-dimethoxy-8-oxoberbines 2a and 2d as regioisomers. The tetramethoxy derivatives 2b or 2c were low on account of the regioisomers mentioned above we switched from this oxidative photocyclization to the "refined" methods of Ninomiya\textsuperscript{9} or Lenz\textsuperscript{2} making use of ortho-brominated benzoyl increments in the enamides 1d-f (Scheme 3). Here elimination of the ortho-group by irradiation takes place.

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\begin{align*}
\text{Scheme 3: Photocyclization of the brominated enamides 1d-f} \\
\text{b) Synthesis of C-13-alkylated 8-oxoberbines by condensation of 2,3,4-tetrahydro-6-methoxy-1-oxo-isoquinolines with C-4-alkylated homophthalic acid anhydrides}
\end{align*}
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Haimova used this condensation for the synthesis of 2,3,10,11-tetramethoxy-8-oxoberbine \textsuperscript{5}. It had to be shown whether C-4-alkylated homophthalic acid anhydrides 7 could also be used for this strategy (Scheme 4). We synthesized the tetramethoxy-8-oxoberbines 2b and 2c by this method. As C-4-alkylated 6-mono-methoxy-substituted homophthalic acid anhydrides are not easily available, 8-oxoberbines with C-11-mono-methoxy substitution in ring D were not prepared by this route.

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\begin{align*}
\text{Scheme 4} \\
\text{Compounds 7 were synthesized via the dimethoxylactones 10a and 10b. First the alkylated dimethoxyphenylacetoni-} \\
\text{triles 8a and 8b are hydrolysed to the phenylacetic acid derivatives 9a and 9b (Scheme 5). According to Finkelstein} \\
\text{and Brossi} \textsuperscript{13} \text{the methylene group ortho to the acetic acid increment was introduced into compounds 9 affording the lactones 10, which are transformed by alkaline oxidation with KMnO}_4 \text{to the corresponding homophthalic acids. Under} \\
\text{these conditions the alkyl groups are not oxidized. Ring closure to the homophthalic acid anhydrides 7 was effected by} \\
\text{refluxing the acids with acetyl chloride (Scheme 5).}
\end{align*}
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\begin{align*}
\text{Scheme 5} \\
\text{This extension of Haimova's strategy} \textsuperscript{5} \text{simplifies known methods, including photocyclization, for the preparation of C-13-alkylated 8-oxoberbines, improves the yields and,} \\
\text{probably, the total synthesis of alkaloids with a berbine skeleton. Pharmacological tests are described in a forthcoming} \\
\text{publication} \textsuperscript{2}. \\
\end{align*}
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**Experimental Part**

Melting points: Büchi 510 apparatus, uncorrected.- Elemental analyses: Mikroanalytisches Laboratorium, University of Regensburg.- IR-spectra: Beckman Acculab III; KBr.- 1H-NMR-spectra: Varian EM 390 (90 MHz), Bruker WM 250 (250 MHz); TMS as internal standard.- UV-spectra: Uvicron 810 (Kontron); solvent: acetonitrile.- Mass-spectra: Varian MAT CH 5.

N-Benzoyl-1,2,3,4-tetrahydroisoquinolines 1a-4, general procedure

20 mmole of the acid chloride 6 in benzene (25 ml) are added to 2.2 g of triethylamine and 20 mmole of the 3,4-dihydroisoquinoline 5 in benzene (50 ml), then the solution is refluxed for 2 h. The precipitate is separated, the product in the remaining benzenic solution is used without purification.- Purified for identification by CC (SiO\textsubscript{2}, CHCl\textsubscript{3}/ether 1:1 or EtOAc).- Yields: 60-75% and small amounts of benzamide-derivatives.

1-Ethylidene-1,2,3,4-tetrahydro-6-methoxy-2-(3-methoxybenzoyl)isoquinoline (1a)

Prepared from 5a and 6b\textsuperscript{6}.

2-(3,4-Dimethoxybenzoyl)-1-ethylidene-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (1b)

From 5c and 6c; colorless crystals, m.p. 156-158°C (ether).- Z-isomer: C\textsubscript{2}H\textsubscript{2}H\textsubscript{2}NO\textsubscript{3} (338.4) Calc. C 68.5 H 6.47 N 3.67 Found C 68.9 H 6.52 N 3.77.- IR (KBr): 1625 (CO) cm\textsuperscript{-1}.- 1H-NMR (250 MHz, -50°C, CDCl\textsubscript{3}); δ (ppm) = 1.30 (d; J = 7 Hz; 3H; CH\textsubscript{3}), 2.60-3.33 (m; 3H; CH\textsubscript{2}), 3.71 (s; 3H; CH\textsubscript{3}), 3.91, 3.93, 3.94 (s; 9H, OCH\textsubscript{3}), 5.11 (m; 1H; H-3), 5.68 (q; J = 7 Hz; 1H vinyl), 6.60-7.22 (m; 5H arom.).
N-[β-(3,4-Dimethoxy-6-[l-oxopropyl]phenyl)ethyl]-3,4-dimethoxybenzamide

Isolation by CC (SiO2, EtOAc). m.p. 128-130°C (ether). C23H27NO5 (410.3). IR (KBr): 3300 (NH), 1690 (CO), 1640 (CO-NH) cm⁻¹. UV (MeOH): λ max (log ε) = 290 (4.02), 260 (4.25), 205 nm (4.64); no change by addition of HCl. MS: m/z = 401 (5%, M⁺), 383 (4, (M - H2O)+), 372 (1), 220 (24), 191 (16), 165 (23), 43 (100).

* This compound has probably come up by hydrolysis of the pertinent 3,4-dihydroisquinoline derivative of Ib.

1,2,3-Tetrahydro-6,7-dimethoxy-2-(3,4-dimethoxybenzoyl)-1-propyldiene-isouquinoline (1c)

From 5d and 6c; colorless crystals; m.p. 134-136°C (ether). Z-isomer: C23H27NO5 (397.5) Calc. 69.5 H 6.65 N 3.5 Found C 69.5 H 6.85 N 3.5.


General Procedure for the Photocyclization

The benzenic solution of the enamide 1 is degassed with N2 in a preparative photoresactor for 20-40 h. Then the solution is irradiated with a 125 W Hg vapor lamp for 20-40 h and evaporated. The remaining oil is purified by CC (SiO2, EtOAc or EtOAc/ether), yields 20-40%.

5,6-Dihydro-3,10-dimethoxy-13-methyl-8H-dibenzo[a,g]quinolizin-8-one (2a)

From 1a or 1d; colorless crystals, m.p. 178-180°C (EtOH). C29H19NO3 x 1/2 EtOH (344.4) Calc. C 73.2 H 6.44 N 4.1 Found C 73.2 H 6.31 N 4.1.

6,5-Dihydro-2,3,10,11-tetramethoxy-13-methyl-8H-dibenzo[a,g]quinolizin-8-one (2b)

From Ib or 1b if photocyclization.

5,6-Dihydro-2,3,10,11-tetramethoxy-13-methyl-8H-dibenzo[a,g]quinolizin-8-one (2c)

From 1e by photocyclization.

13-Ethyl-5,6-dihydro-2,3,10,11-tetramethoxy-8H-dibenzo[a,g]quinolizin-8-one (2d)

From 5e and 6d; yellow needles, m.p. 179-180°C (EtOH). C29H19NO3 x 1/2 EtOH (341.8) Calc. C 68.9 H 6.74 N 3.4 Found C 68.9 H 6.74 N 3.2.

5,6-Dihydro-3,12-dimethoxy-13-methyl-8H-dibenzo[a,g]quinolizin-8-one (2e)

From 1a; colorless crystals, m.p. 177-179°C (EtOH). C29H19NO3 x 1/2 EtOH (344.4) Calc. C 73.2 H 6.44 N 4.1 Found C 73.3 H 6.48 N 4.0.

13-Ethyl-5,6-dihydro-3,10-dimethoxy-8H-dibenzo[a,g]quinolizin-8-one (2f)

From 1e; yellow foam, m.p. 130-131°C (EtOH). C29H19NO3 (335.4) Calc. C 75.2 H 6.31 N 4.2 Found C 74.4 H 6.30 N 4.1.
2.80 (t; J = 6 Hz; 2H; CH\textsubscript{2}-H-5); 2.98 (q; J = 7.5 Hz; 2H; CH\textsubscript{2}-CH\textsubscript{3}); 3.82 (s; 3H; OCH\textsubscript{3}); 3.93 (s; 3H; OCH\textsubscript{3}); 4.23 (t; J = 6 Hz; 2H; CH\textsubscript{2}-H-6); 6.73-6.98 (m; 2H arom.); 7.20-7.83 (m; 3H arom.); 7.93 (d; J = 2.5 Hz; 1H arom; H-9).- UV: λ max (log ε) = 322 (4.23), 216 nm (4.54).

2,10,11-Tetraacetoxy-5,6-dihydro-8H-dibenzo[a,g]quinolinizin-2-one (3a)

From 2c according to lit.1, m.p. 225-227°C (MeOH).- C\textsubscript{23}H\textsubscript{22}NO\textsubscript{8} x 1 H\textsubscript{2}O (525.5) Calc. C 61.7 H 5.18 N 2.7 Found C 61.4 H 5.00 N 2.7.- IR (KBr): 1760, 1770 (CO-CH\textsubscript{3}), 1650 (CO) cm\textsuperscript{-1}.- CD: δ (ppm) = 1.43 (s; 3H; CH\textsubscript{3}); 2.35 (s; 3H; H-C-CO); 2.76-3.18 (m; 4H; CH\textsubscript{2}-CH\textsubscript{3} and CH\textsubscript{2}-H-5); 4.28 (t; J = 6 Hz; 2H; CH\textsubscript{2}-H-6); 7.20, 7.53, 7.70, 8.38 (s; 4H arom.).- UV: λ max (log ε) = 330 (4.30), 213 nm (4.52).

3,10-Diacetoxy-5,6-dihydro-8H-dibenzo[a,g]quinolinizin-8-one (3b)

From 2a according to lit.1, m.p. 232-234°C (EtOH).- C\textsubscript{23}H\textsubscript{22}NO\textsubscript{8} (378.4) Calc. C 69.0 H 5.07 N 3.7 Found C 68.9 H 5.08 N 3.7.- IR (KBr): 1760 (CO-CH\textsubscript{3}), 1650 (CO) cm\textsuperscript{-1}.- H-NMR (250 MHz, CDC\textsubscript{13}) δ (ppm) = 0.95 (t; J = 7.5 Hz; 3H; CH\textsubscript{3}); 1.38-1.82 (m; 2H; CH\textsubscript{2}-CH\textsubscript{2}-CH\textsubscript{3}); 3.62 (q; J = 7.5 Hz; 2H; CH\textsubscript{2}-CH\textsubscript{3}), 6.57-6.87 (m; 3H arom.); 7.07-7.33 (m; 1H arom.).- UV: λ max (log ε) = 274 (3.73), 249 (3.70), 229 nm (3.58).- UV (plus HCl): λ max (log ε) = 316 (3.81), 307 (3.81), 236 nm (3.70).

Methoxypheylethylamides 4a-d, general procedure

100 mmoles of propionic or butyric acid chloride are added under reflux to 100 mmoles β-(3-methoxyphenyl)ethylamine or homoveratrylamine and 10.0 g triethylamine in 60 ml absol. CH\textsubscript{2}Cl\textsubscript{2}. The mixture is stirred for 2 h. Then excess of POCl\textsubscript{3} and acetoni­cles are distilled off. The resulting acid chlorides are purified by distillation or used without purification.- Yields 80-90%.

N-β-[3-Methoxypheylethyl]propionamide (4a)

From 1-amino-2-(3-methoxypheylethyl)ethane (Alldrich) and propionic acid chloride; colorless oil, b.p. 96-97°C, 0.05 T., lit. 97°C, 0.05 T.0.

N-β-[3-Methoxypheylethyl]butanamide (4b)

From 1-amino-2-(3-methoxypheylethyl)ethane (Alldrich) and butyric acid chloride; colorless oil, b.p. 185-187°C, 0.1 T.; IR (KBr): 3290 (NH), 1650 (CO) cm\textsuperscript{-1}.- H-NMR (CDCl\textsubscript{3}) δ (ppm) = 0.90 (t; J = 7.5 Hz; 3H; CH\textsubscript{3}); 1.37-1.82 (m; 2H; CH\textsubscript{2}-CH\textsubscript{2}-CH\textsubscript{3}); 2.10 (t; J = 7.5 Hz; 2H; CH\textsubscript{2}-CH\textsubscript{2}-CH\textsubscript{3}); 2.77 (t; J = 7.5 Hz; 2H; CH\textsubscript{2}-CH\textsubscript{2}-NH+); 3.62 (q; J = 7.5 Hz; 2H; CH\textsubscript{2}-CH\textsubscript{2}-N; t after D-exchange), 3.73 (s; 3H; OCH\textsubscript{3}); 6.22 (s [broad]; 1H; NH), 6.57-6.87 (m; 3H arom.), 7.07-7.33 (m; 1H arom.).

N-β-[3,4-Dimethoxypheylethyl]propionamide (4c)

From homoveratrylamine and propionic acid chloride; colorless crystals, m.p. 54-55°C (ether), lit.: 57.5-59°C.0.

3,10-Diacetoxy-8H-dibenzo[a,g]quinolinizin-8-one (3c)

From 2a according to lit.1, m.p. 195-197°C (MeOH).- C\textsubscript{23}H\textsubscript{22}NO\textsubscript{8} x 1 H\textsubscript{2}O (409.5) Calc. C 67.5 H 5.66 N 3.4 Found C 67.9 H 5.88 N 3.5.- IR (KBr): 1760 (CO-CH\textsubscript{3}), 1640 (CO) cm\textsuperscript{-1}.- H-NMR (250 MHz, CDC\textsubscript{13}) δ (ppm) = 0.78 (s; 3H; CH\textsubscript{3}); 1.36-1.82 (m; 2H; CH\textsubscript{2}-CH\textsubscript{2}-CH\textsubscript{3}), 6.60-6.83 (m; 2H arom.), 7.42 (d; J = 7.5 Hz; 2H; CH\textsubscript{2}-CH\textsubscript{2}-N), 3.80 (s; 3H; OCH\textsubscript{3}), 6.57-6.87 (m; 2H arom.), 7.42 (d; J = 7.5 Hz; 1H arom.).- UV: λ max (log ε) = 316 (3.81), 307 (3.81), 236 nm (3.70).
6,7-Dimethoxy-4-methyl-isochroman-1,3-dione (7a)

From 10a via 4,5-dimethoxy-α-methyl-homophthalic acid\(^{(13)}\), which was heated with CH\(_3\)COCl\(^{13}\).

4-Ethyl-6,7-dimethoxy-isochroman-1,3-dione (7b)

From 10b via 4,5-dimethoxy-α-ethyl-homophthalic acid\(^{(13)}\), which was heated with CH\(_3\)COCl\(^{13}\).

6,7-Dimethoxy-4-methyl-isochroman-3-one (10a)

From 9a; colorless crystals, m.p. 122-124°C (EtOH).- C\(_{13}\)H\(_{16}\)O\(_4\) (222.2) Calc. C 64.8 H 6.35 Found C 64.6 H 6.43.- IR (KBr): 1740 (CO) cm\(^{-1}\).

6,7-Dimethoxy-4-ethyl-isochroman-3-one (10b)

From 9b; light-brown crystals, m.p. 84-85°C (EtOH).- C\(_{13}\)H\(_{18}\)O\(_4\) (236.3) Calc. C 66.1 H 6.83 Found C 65.7 H 6.84.- IR (KBr): 1740 (CO) cm\(^{-1}\).- 1H-NMR (CDCl\(_3\)): \(\delta\) (ppm) = 1.07 (d; J = 7 Hz; 3H; CH\(_3\)), 1.77-2.17 (m; 2H; CH\(_2\)-CH\(_3\)), 3.50 (t; J = 7 Hz; 1H; CH-CH\(_2\)), 3.87 (s; 6H; OCH\(_3\)), 5.30 (d; J = 5 Hz; 2H; CH\(_2\)-H-1), 6.68, 6.73 (s; 2H arom.).

2-(3,4-Dimethoxyphenyl)propionic acid (9a)

From 8a according to Jeffreys\(^{(19)}\), yellow oil; lit. m.p. 50°C\(^{(19)}\).

2-(3,4-Dimethoxyphenyl)butyric acid (9b)

From 8b according to Jeffreys\(^{(19)}\); colorless crystals, m.p. 90-92°C (H\(_2\)O); lit. 103°C\(^{(20)}\).

2-(3,4-Dimethoxyphenyl)propionitrile (8a)

Prepared from 3,4-dimethoxyphenylacetonitrile (Merck) according to Chavdarian\(^{(12)}\) by the carboxylation procedure.- Colorless crystals; yield 87%; m.p. 67-69°C (toluene), lit. 67-69°C\(^{(13)}\).

2-(3,4-Dimethoxyphenyl)butyronitrile (8b)

2.52 ml (18 mmole) of diisopropylamine are dissolved in 25 ml of absol. THF and cooled to -20°C. 7.74 ml of 2.3-molar n-BuLi (17.8 mmole) are added, the temp. should not exceed -10°C. Then the mixture is stirred for 15 min at -20°C and cooled to -50°C. 16.9 mmole of 3,4-dimethoxyphenylacetonitrile (Fa. Aldrich) in 10 ml of absol. THF are added below -40°C.

After stirring for 5 min at -50°C, 1.45 ml (17.8 mmole) of C\(_2\)H\(_4\)I in 20 ml of absol. THF are added and the mixture is stirred again for 1 h at -60°C, then overnight at room temp, and poured on 50 ml of 2N HCl. The solution is extracted for a few times with ether, the org. layers are washed with H\(_2\)O, dried and evaporated.- Purification: CC (SiO\(_2\), EtOAc), yield 80-90%.- Colorless oil, b.p. 90-95°C/0.1 T.- Lit. 120.\(^{(20)}\); m.p. 56-57°C.- C\(_{13}\)H\(_{12}\)NO\(_2\) (205.3) Calc. C 70.2 H 7.37 N 6.8 Found C 70.1 H 7.30 N 6.7.- IR (film): 2260 (CN) cm\(^{-1}\).- 1H-NMR (CDCl\(_3\)): \(\delta\) (ppm) = 1.03 (t; J = 7 Hz; 3H; CH\(_3\)), 1.93 (quin; J = 7.5 Hz; 2H; CH\(_2\)-CH\(_3\)), 3.63 (t; J = 7.5 Hz; 1H; CH-CH\(_3\)), 3.87 (s; 6H; OCH\(_3\)), 6.67-6.90 (m; 3H arom.).

References