SYNTHESIS OF 2,3-DIALKOXY-1*H*-PYRROLE *VIA* REDUCTION OF DIOXOPYRROLINE WITH SODIUM HYDROSULFITE[1]

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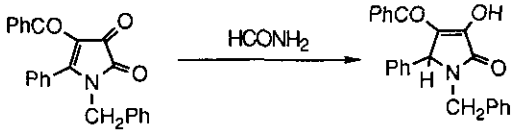
Abstract - Reduction of 1*H*-pyrrole-2,3-dione (**3** and **7**) with sodium hydrosulfite took place selectively in a 1,4-manner. Methylation of the product with diazomethane gave 1,5-dihydro-3-methoxy-2*H*-pyrrol-2-one (**5** and **8**) in good yield. Treatment of the methyl ether (**5** or **8**) with triethyloxonium tetrafluoroborate caused ethylation of the lactam carbonyl with concomitant deprotonation to give 2,3-dialkoxy-1*H*-pyrrole (**9**). This conversion provides a simple synthetic method of 2,3-dialkoxypyrroles.

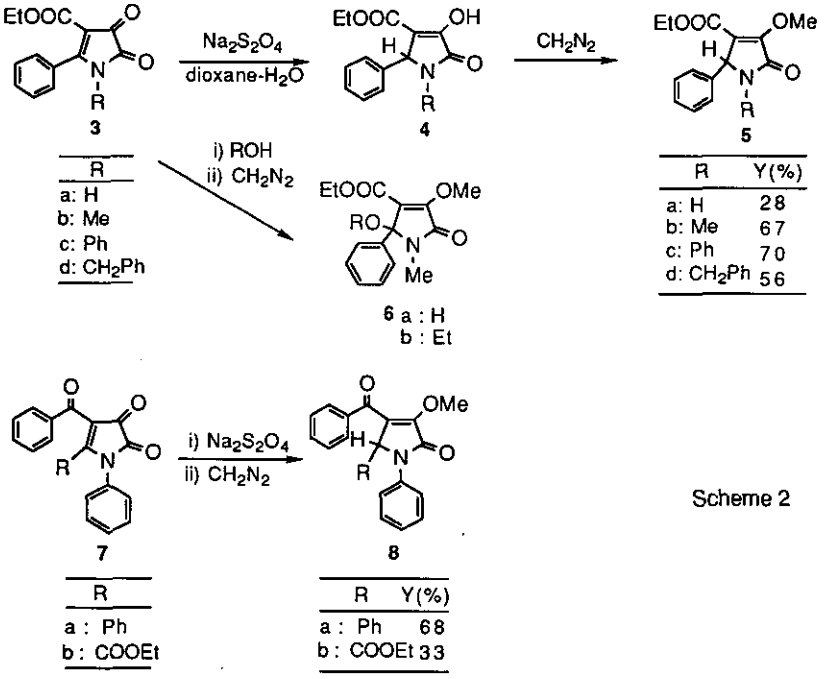
The electrophilic property of 1*H*-pyrrole-2,3-dione (dioxopyrroline) has been shown by its vulnerability to protic solvents. The high reactivity of the ring system towards nucleophiles is attributable to its non-aromatic character.[2] However, the reaction for other nucleophiles except protic solvent is seldom reported. For reduction of dioxopyrroline, Andreichikov's group[3] reported that reaction of 4-benzoyl-1-benzyl-5-phenyldioxopyrroline (**1**) with formamide happened to cause reduction giving 1,5-dihydro-3-hydroxy-2*H*-pyrrol-2-one (**2**) in good yield. In this paper we report conversion of dioxopyrrolines into 2,3-dialkoxy-1*H*-pyrroles *via* the reduction with hydrosulfite.



1 **2**

Scheme 1

When a dioxane solution of *N*-nonsubstituted 4-ethoxycarbonyl-5-phenyldioxopyrroline (**3a**) was treated with an aqueous solution of large excess of sodium hydrosulfite (about 20 mole equivalent), reduction took place immediately to give a 1,5-dihydro-3-hydroxy-2*H*-pyrrol-2-one (**4a**). This was very unstable to air oxidation even in solid state regenerating the dioxopyrroline. Methylation of the crude product with diazomethane (CH_2N_2) gave the methyl ether (**5a**) in 28% yield, which was stable and fully characterized. The structure of **5a** was clarified by spectral data, specially by analogy of the uv spectrum to the known 1,5-dihydro-3-hydroxy-5-methoxy-2*H*-pyrrol-2-one (**6b**).[2] In the following experiments the product obtained from reduction of dioxopyrroline was characterized as the corresponding methyl ether after methylation with CH_2N_2.



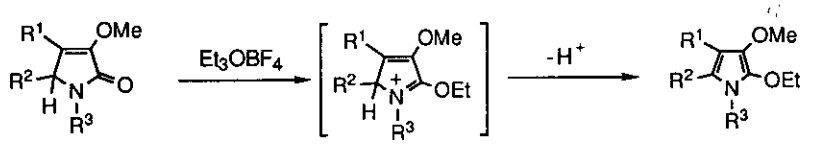
Scheme 2

Reaction of *N*-methyl-, *N*-phenyl-, and *N*-benzyldioxopyrrolines (**3b**, **3c**, and **3d**) with large excess of sodium hydrosulfite in aqueous dioxane solution caused the same 1,4-reduction to give the 1,5-dihydro-3-methoxy derivatives (**5b**, **5c**, and **5d**) in good yield, respectively, as shown in Scheme 2. Reduction of 4-benzoyldioxopyrrolines (**7a** and **7b**) with sodium hydrosulfite similarly took place to give the dihydro derivatives (**8a** and **8b**) in 58 and 63% yields, respectively. Thus, sodium hydrosulfite selectively reduced the dioxopyrroline ring in a 1,4-manner to yield 1,5-dihydro-3-hydroxy-2*H*-pyrrol-2-one regardless of substituent at 1, 4, and 5 positions. The reductant, sodium hydrosulfite, should be used in large excess to

avoid solvolitic change of the dioxopyrroline that could competitively take place in the medium. For example, treatment of **3b** in ethanol with an aqueous solution of 1.5 molar eq. of sodium hydrosulfite gave the ethanol adduct (**6b**) as a major product (23%) along with the water adduct (**6a**) (1.5%) and the reduced product (**5b**) (15%). On the other hand, reduction of **3a** in ethanol with 20 molar eq. of sodium hydrosulfite gave **5a** in 50% yield, no solvolytic product being isolated from the latter reaction.

Reduction of the dioxopyrroline (**3**) with other reducing methods such as sodium borohydride in ethanol, tetra-n-butylammonium borohydride in dichloromethane, and lithium borohydride in THF gave, after methylation with CH_2N_2, the methyl ether (**5**) in 2-15% yield and no other characterizable products were isolated from their reaction mixture. For example, reduction of **3a** and **3b** with tetra-n-butylammonium borohydride in dichloromethane gave **5a** and **5b** in 8% and 16% yield, respectively. A major reason decreasing the yield in hydride reductions may be due to over reduction, although no evidence was available.

The 1,5-dihydro-3-methoxy-2*H*-pyrrol-2-ones (**5** and **8**) were readily converted into the 2,3-dialkoxy-1*H*-pyrroles (**9**). Treatment of **5** or **8** with triethyloxonium tetrafluoroborate (Meerwein reagent) in dichloromethane at room temperature gave the 2-ethoxy-3-methoxy-1*H*-pyrrole (**9**) in good yield as shown in Scheme 3. The formation of pyrrole is rationalized in terms of 1,5-hydride shift and/or deprotonation from the iminium ion (**10**) formed by *O*-ethylation of lactam carbonyl with Meerwein réagent.



5 or **8** **10** **9**

	R_1	R_2	R_3
5a	COOEt	Ph	H
5b	COOEt	Ph	Me
5c	COOEt	Ph	Ph
5d	COOEt	Ph	$PhCH_2$
8a	PhCO	Ph	Ph
8b	PhCO	COOEt	Ph

	R_1	R_2	R_3	Y(%)
a	COOEt	Ph	H	76
b	COOEt	Ph	Me	69
c	COOEt	Ph	Ph	85
d	COOEt	Ph	$PhCH_2$	72
e	PhCO	Ph	Ph	84
f	PhCO	COOEt	Ph	64

Scheme 3

EXPERIMENTAL

Unless otherwise stated, the following procedures were adopted. All melting points were taken on a Yanagimoto micro hot-stage melting point apparatus and are uncorrected. Ir spectra were measured with a Hitachi 260-10 and are given in ν_{max} cm^{-1}. Uv spectra were measured with a Hitachi 200-10 spectropho-

tometer in dioxane and given in λ_{max} nm (ε). Nmr spectra were taken on a JEOL FX-100 NMR spectrometer (^1H; 100 MHz, ^{13}C; 25.0 MHz) in $CDCl_3$ using tetramethylsilane (TMS) as an internal standard. The chemical shifts are given in δ values. Low resolution mass spectra (LRms) and high resolution mass spectra (HRms) were determined with a JEOL JMS-D 300 spectrometer at 30 eV.

Reduction of Dioxopyrroline (1) with Sodium Hydrosulfite (General Procedure) A freshly prepared solution of sodium hydrosulfite (20 mol eq.) in hot water (50 ml) was added in one portion to a solution of dioxopyrroline (**3** or **7**) (1 g, 2.83-4.08 mmol) in dioxane (50 ml), and the resulting solution was stirred at room temperature for 30 min. After filtration of insoluble materials, the filtrate was concentrated *in vacuo* and extracted with CH_2Cl_2. The extract was washed with water, dried over Na_2SO_4, and concentrated *in vacuo* to afford a crystalline powder, which was dissolved in CH_2Cl_2 and methylated by the addition of an excess ethereal CH_2N_2 to give **5** or **8** after usual work-up procedure and purification by SiO_2 column chromatography.

5a: 528 mg (28%). Colorless prisms from acetone-ether, mp 130-132.5°C. Ir (Nujol): 3180, 3080, 1730, 1710, 1640. ^1H-Nmr: 1.09 (3H, t, *J*=7 Hz, $CO_2CH_2C\underline{H}_3$), 4.06 (2H, q, *J*=7 Hz, $CO_2C\underline{H}_2CH_3$), 4.28 (3H, s, $OC\underline{H}_3$), 5.24 (1H, br s, NC<u>H</u>Ph), 7.28 (5H, s, ArH). ^{13}C-Nmr: 13.9 (q), 58.0 (d), 60.2 (q), 60.6 (t), 118.8 (s), 127.3 (dx2), 128.4 (d), 128.5 (dx2), 136.5 (s), 153.2 (s), 161.9 (s), 167.1 (s). Uv: 248 (11500). LRms (*m/z*): 261 (M^+). *Anal.* Calcd for $C_{14}H_{15}NO_4$: C, 64.36; H, 5.79; N, 5.36. Found: C, 64.45; H, 5.65; N: 5.20.

5b: 713 mg (67%). Colorless prisms from AcOEt-hexane, mp 75-76°C. Ir (Nujol): 1720, 1700, 1600. ^1H-Nmr: 1.08 (3H, t, *J*=7 Hz, $CO_2CH_2C\underline{H}_3$), 2.76 (3H, s, N-$C\underline{H}_3$), 4.05 (2H, dq, *J*=1 and 7 Hz, $CO_2C\underline{H}_2CH_3$), 4.33 (3H, s, $OC\underline{H}_3$), 5.02 (1H, s, NC<u>H</u>Ph), 7.0-7.6 (5H, m, ArH). ^{13}C-Nmr: 13.9 (q), 27.6 (q), 60.1 (q), 60.5 (t), 63.4 (d), 116.8 (s), 127.6 (d), 128.4 (dx4), 135.2 (s), 153.9 (s), 161.8 (s), 164.6 (s). Uv: 247 (12000). LRms (*m/z*): 275 (M^+), 202 (base peak). HRms (*m/z*): Calcd for $C_{15}H_{17}NO_4$ (M^+): 275.1154. Found: 275.1143

5c: 732 mg (70%). Colorless needles from AcOEt-hexane, mp 82-83°C. Ir (KBr): 1688, 1649, 1600. ^1H-Nmr (90 MHz): 1.14 (3H, t, *J*=7 Hz, $CO_2CH_2C\underline{H}_3$), 4.11 (2H, q, *J*=7 Hz, $CO_2C\underline{H}_2CH_3$), 4.35 (3H, s, $OC\underline{H}_3$), 5.74 (1H, s, NC<u>H</u>Ph), 6.9-7.5 (5H, m, ArH), 7.21 (5H, s, ArH). ^{13}C-Nmr: 14.0 (q), 60.3 (q), 60.8 (t), 62.4 (d), 117.6 (s), 122.7 (dx2), 125.8 (d), 127.7 (dx2), 128.5 (dx3), 128.8 (dx2), 135.4 (s), 136.3 (s), 152.5 (s), 161.8 (s), 163.8 (s). Uv: 225 (13800), 256 (11000), 286 (6200, sh). LRms (*m/z*): 337 (M^+), 264 (base peak). HRms (*m/z*): Calcd for $C_{20}H_{19}NO_4$ (M^+): 337.1311. Found: 337.1301.

5d: 581 mg (56%). Colorless needles from ether-hexane, mp 61-62°C. Ir (KBr): 1692, 1636. ^{1}H-Nmr (90 MHz): 1.02 (3H, t, *J*=7 Hz, $CO_2CH_2C\underline{H}_3$), 3.50, 5.12 (each 1H, d, *J*=15 Hz, $NC\underline{H}_2Ph$), 4.00 (2H, dq, *J*=1 and 7 Hz, $CO_2C\underline{H}_2CH_3$), 4.36 (3H, s, $OC\underline{H}_3$), 4.92 (1H, s, $NC\underline{H}Ph$), 7.0-7.6 (10H, m, Ar-H). ^{13}C-Nmr: 13.9 (q), 44.0 (t), 60.2 (q), 60.5 (t), 60.7 (d), 117.2 (s), 127.8 (d), 128.0 (dx3), 128.5 (dx2), 128.8 (dx4), 135.0 (s), 136.4 (s), 153.5 (s), 161.8 (s), 164.5 (s). Uv: 246 (12000). LRms (*m/z*): 351 (M^+), 91 (base peak). HRms (*m/z*): Calcd for $C_{21}H_{21}NO_4$ (M^+): 351.1470. Found: 351.1480.

8a: 714 mg (68%). Colorless needles from $CHCl_3$, mp 177-178°C. Ir (KBr): 1696, 1626, 1603. ^{1}H-Nmr (90 MHz): 3.95 (3H, s, $OC\underline{H}_3$), 6.06 (1H, s, $NC\underline{H}Ph$), 7.0-7.8 (15H, m, ArH). ^{13}C-Nmr: 60.0 (q), 62.7 (d), 119.8 (s), 122.0 (d), 125.5 (d), 126.2 (d), 127.0 (dx2), 128.8 (dx3), 128.9 (dx3), 129.1 (dx3), 133.4 (d), 134.7 (s), 136.4 (s), 137.6 (s), 148.8 (s), 162.4 (s), 190.6 (s). Uv: 262 (14200), 322 (6400, sh). LRms (*m/z*): 369 (M^+), 105 (base peak). HRms (*m/z*): Calcd for $C_{24}H_{19}NO_3$ (M^+): 369.1365. Found: 369.1395.

8b: 381 mg (33%). Light yellow prisms from hexane-ether-acetone, mp 89-93°C. Ir (KBr): 1744, 1713 (br), 1640, 1599. ^{1}H-Nmr (90 MHz): 1.01 (3H, t, *J*=7 Hz, $CO_2CH_2C\underline{H}_3$), 3.99 (3H, s, $OC\underline{H}_3$), 4.04 (2H, q, *J*=7 Hz, $CO_2C\underline{H}_2CH_3$), 5.58 (1H, s, $NC\underline{H}Ph$), 7.1-8.0 (10H, m, ArH). ^{13}C-Nmr: 13.8 (q), 59.6 (q), 61.6 (d), 62.3 (t), 119.5 (s), 121.4 (dx3), 126.1 (d), 128.5 (dx2), 129.2 (dx3), 133.7 (d), 136.7 (s), 137.4 (s), 150.6 (s), 163.8 (s), 167.3 (s), 190.0 (s). Uv: 262 (13500). LRms (*m/z*): 365 (M^+), 105 (base peak). HRms (*m/z*): Calcd for $C_{21}H_{19}NO_5$ (M^+): 365.1263. Found: 365.1288.

Reduction of 3a with Sodium Hydrosulfite in EtOH A freshly prepared solution of sodium hydrosulfite (14.3 g, 20 mol eq.) in hot water (50 ml) was added to a solution of **3a** (1 g, 4.08 mmol) in EtOH (70 ml), and the resulting solution was stirred at room temperature for 2 h. After removal of insoluble materials, the filtrate was concentrated *in vacuo* to dryness to afford a crude gum. Methylation of this with CH_2N_2 in CH_2Cl_2 and chromatography of the product over SiO_2 gave **5a** (528 mg, 50%).

Reduction of 3b with Sodium Hydrosulfite in EtOH A freshly prepared solution of sodium hydrosulfite (0.62 g, 1 mol eq.) in hot water (5 ml) was added to a solution of **3b** (1 g, 3.86 mmol) in EtOH (40 ml) and ether (40 ml) at room temperature. After 30 min, a solution of sodium hydrosulfite (0.3 g, 0.5 mol eq.) in hot water (0.5 ml) was further added to this mixture, and the mixture was stirred at room temperature for 30 min. After removal of insoluble materials by filtration, filtrate was diluted with CH_2Cl_2, washed with water, dried over Na_2SO_4, and concentrated *in vacuo* to give a crude product (652 mg). This was methylated with CH_2N_2 and the product was purified by MPLC eluting with AcOEt-hexane (1:2) to give the ethanol-adduct (**6b**) (285 mg, 23%) as a pale yellow oil.[2] The reduced product

(**5b**) (149 mg, 15%), and the H_2O-adduct (**6a**) (19 mg, 1.8%) as colorless needles from AcOEt-hexane (1:2), mp 146-148°C.[2]

Reduction of 3a or 3b with Tetra-n-butylammonium Borohydride Tetra-n-butylammonium borohydride (530 mg, 0.5 mol eq. for **3a** and 496 mg, 0.5 mol eq. for **3b**) was added to a solution of **3a** or **3b** (1.0 g,) in dry CH_2Cl_2 (40 ml) at -40°C with stirring. After 1 min, excess hydride was decomposed by addition of ice-water. The mixture was extracted with CH_2Cl_2. The organic extract was washed with brine, dried over Na_2SO_4, and concentrated *in vacuo* to afford the crude product which was treated with CH_2N_2. After a usual work-up, the product was filtered through a short SiO_2 column eluting with AcOEt-hexane (1:1), and the eluate was purified by MPLC [AcOEt-hexane (1:1)] to give **5a** (86 mg, 8.0%) or **5b** (169 mg, 16%).

Synthesis of 2,3-Dialkoxy-1*H*-pyrrole (9) (General Procedure) An excess triethyloxonium tetrafluoroborate (Et_3OBF_4) and anhydrous K_2CO_3 (50-100 mg) was added to a solution of **5** or **8** (80-300 mg) in dry CH_2Cl_2 (10-40 ml), and the resulting solution was stirred at room temperature under argon atmosphere for 15-48 h. The mixture was poured into 5% $NaHCO_3$ (20-40 ml), and the whole was extracted with CH_2Cl_2, washed with water, and dried over Na_2SO_4. After evaporation of the solvent *in vacuo*, the residue was purified by column chromatography on SiO_2 [elution with AcOEt-hexane (1:3) and (1:1)].

9a: 276 mg (76%) from 330 mg (1.26 mmol) of **5a**. Colorless prisms from ether-hexane, mp 131-134°C. Ir (Nujol): 3180, 1660, 1610, 1590. ^{1}H-Nmr: 1.22, 1.37 (each 3H, t, *J*=7 Hz, $OCH_2C\underline{H}_3$), 3.86 (3H, s, $OC\underline{H}_3$), 4.22, 4.23 (each 2H, q, *J*=7 Hz, $OC\underline{H}_2CH_3$), 7.1-7.6 (5H, m, ArH), 7.71 (1H, br s, N<u>H</u>). ^{13}C-Nmr: 14.1 (q), 15.4 (q), 60.0 (t), 62.5 (q), 69.8 (t), 104.6 (s), 125.4 (s), 127.7 (d), 128.0 (dx3), 128.7 (d), 129.8 (s), 132.4 (s), 134.8 (s), 164.2 (s). Uv: 313 (10000). LRms (*m/z*): 289 (M^+). *Anal.* Calcd for $C_{16}H_{19}NO_4$: C, 66.42; H, 6.62; N, 4.84. Found: C, 66.31; H, 6.73; N: 4.65.

9b: 151 mg (69%) from 200 mg (0.73 mmol) of **5b** as a colorless oil. Ir (Film): 1690, 1600, 1590. ^{1}H-Nmr: 1.00, 1.33 (each 3H, t, *J*=7 Hz, $OCH_2C\underline{H}_3$), 3.13 (3H, s, $NC\underline{H}3$), 3.79 (3H, s, $OC\underline{H}_3$), 4.01, 4.17 (each 2H, q, *J*=7 Hz, $OC\underline{H}_2CH_3$), 7.1-7.4 (5H, m, ArH). ^{13}C-Nmr: 14.0 (q), 15.5 (q), 29.5 (q), 59.1 (t), 62.5 (q), 69.9 (t), 103.9 (s), 127.8 (dx2), 127.9 (s), 129.4 (s), 130.8 (dx3), 131.9 (s), 135.1 (s), 164.0 (s). Uv: 234 (10500), 300 (6100). LRms (*m/z*): 303 (M^+), 274 (base peak). HRms (*m/z*): Calcd for $C_{17}H_{21}NO_4$ (M^+): 303.1471. Found: 303.1472.

9c: 300 mg (85%) from 275 mg (0.82 mmol) of **5c** as a dark orange oil. Ir (Film): 1707, 1613, 1601. ^{1}H-Nmr: 1.17 (6H, t, *J*=7 Hz, $OCH_2C\underline{H}_3$), 3.94 (3H, s, $OC\underline{H}_3$), 3.96, 4.15 (each 2H, q, *J*=7 Hz, $OC\underline{H}_2CH_3$), 7.0-7.4 (5H, m, ArH), 7.15 (5H, s, ArH). ^{13}C-Nmr: 14.0 (q), 15.1 (q), 59.5 (q), 62.3 (t), 70.6 (t), 105.3 (s), 127.2 (dx3), 127.3 (d), 127.9 (s), 128.4 (dx3), 130.5 (s), 131.2 (dx3), 131.4 (sx2), 135.8 (s), 164.0 (s). Uv: 240 (16800), 301 (6900). LRms (*m/z*): 365 (M^+), 336 (base peak). HRms (*m/z*): Calcd for $C_{22}H_{23}NO_4$ (M^+): 365.1627. Found: 365.1660.

9d: 62 mg (72%) from 80 mg (0.23 mmol) of **5d** as a yellow oil. Ir (Film): 1707, 1613, 1601. ^{1}H-Nmr: 1.05, 1.20 (each 3H, t, *J*=7 Hz, $OCH_2C\underline{H}_3$), 3.87 (3H, s, $OC\underline{H}_3$), 4.07, 4.08 (each 2H, q, *J*=7 Hz, $OC\underline{H}_2CH_3$), 4.81 (2H, s, N-$C\underline{H}_2$-Ph), 6.8-7.4 (10H, m, Ar-H). ^{13}C-Nmr: 14.0 (q), 15.4 (q), 46.1 (t), 59.2 (t), 62.6 (q), 69.7 (t), 104.6 (s), 126.3 (dx3), 127.1 (d), 127.7 (dx2), 128.0 (s), 128.3 (dx2), 129.3 (s), 131.0 (dx2), 131.8 (s), 135.4 (s), 138.0 (s), 163.9 (s). Uv: 232 (13200), 296 (5700). LRms (*m/z*): 379 (M^+). HRms (*m/z*): Calcd for $C_{23}H_{25}NO_4$ (M^+): 379.1781. Found: 379.1781.

9e: 94 mg (84%) from 100 mg (0.27 mmol) of **8a** as a yellow oil. Ir (CH_2Cl_2): 1752, 1640, 1601. ^{1}H-Nmr: 1.13 (3H, t, *J*=7 Hz, $OCH_2C\underline{H}_3$), 3.74 (3H, s, $OC\underline{H}_3$), 3.97 (2H, q, *J*=7 Hz, $OC\underline{H}_2CH_3$), 6.8-7.9 (10H, m, ArH), 6.96 (5H, s, ArH). ^{13}C-Nmr: 15.4 (q), 62.1 (q), 70.7 (t), 114.4 (s), 126.3 (s), 126.9 (d), 127.4 (dx2), 127.6 (dx2), 128.0 (d), 128.3 (dx2), 128.5 (dx2), 129.7 (s, dx2), 130.7 (s, dx2), 131.8 (d), 135.5 (s), 136.0 (s), 139.0 (s), 192.0 (s). Uv: 249 (18300), 278 (15200), 350 (2800, sh). LRms (*m/z*): 397 (M^+), 368 (base peak). HRms (*m/z*): Calcd for $C_{26}H_{23}NO_3$ (M^+): 397.1676. Found: 397.1651.

9f: 147 mg (64%) from 215 mg (0.59 mmol) of **8b** as light yellow prisms from ether-hexane, mp 38-39°C. Ir (Film): 1715, 1667, 1599. ^{1}H-Nmr: 0.66, 1.16 (each 3H, t, *J*=7 Hz, $OCH_2C\underline{H}_3$), 3.73, 4.11 (each 2H, q, *J*=7 Hz, $OC\underline{H}_2CH_3$), 3.75 (3H, s, $OC\underline{H}_3$), 7.2-8.1 (10H, m, ArH). ^{13}C-Nmr: 13.3 (q), 15.2 (q), 60.0 (t), 62.7 (q), 70.2 (t), 109.8 (s), 122.6 (s), 127.9 (dx2), 128.3 (dx3), 128.5 (dx3), 129.4 (d), 130.4 (s), 133.0 (d), 136.3 (s), 138.3 (s), 139.8 (s), 159.2 (s), 192.7 (s). Uv: 250 (18900), 279 (14100). LRms (*m/z*): 393 (M^+). HRms (*m/z*): Calcd for $C_{23}H_{23}NO_5$ (M^+): 393.1574. Found: 393.1554.

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