Synthesis of a New Tricyclic Compound, 5*H*-2-Methoxycarbonyl-4-oxofuro[2,3-*b*][1,5]benzothiazepine

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Methyl 5-(2-carboxyphenylthio)-2-furancarboxylate (2) was obtained by the nucleophilic substitution of methyl 5-nitro-2-furancarboxylate (3) with the disodium salt of thiosalicylic acid, and chlorination of 2 gave methyl 5-(2-chloroformylphenylthio)-2-furancarboxylate (4). The reaction of 4 with sodium azide gave methyl 5-(2-azidocarbonylphenylthio)-2-furancarboxylate (8) and methyl 5-(2-isocyanatophenylthio)-2-furancarboxylate (9) was obtained by the thermolysis of 8. Finally, the new tricyclic compound, 5*H*-2-methoxycarbonyl-4-oxofuro[2,3-*b*][1,5]benzothiazepine (1) was obtained by cyclization of 9 in the presence of aluminum chloride.

Key words furobenzothiazepine; furothiochromone; Curtius rearrangement; nucleophilic substitution; phenyl furyl sulfide; phenyl furyl ether

Many tricyclic compounds have biological activities and are used as drugs in various fields of clinical medicine. We have synthesized furo[3,2-*b*]indoles[1] and studied the biological activities of their derivatives, some of which were found to have anti-inflammatory activity.[2] In the present study, to investigate the properties of tricyclic compounds in which the benzene ring of the antidepressant amoxapine or the psychotherapeutic agent clothiapine had been exchanged for a furan ring, the synthesis of furobenzothiazepines and furobenzoxazepines was attempted (Fig. 1). We also studied the nucleophilic substitution of nitrofurans,[3] phenyl furyl sulfides and phenyl furyl ethers, obtained as synthetic intermediates.

Nagarajan *et al.*[4] synthesized 10*H*-11-oxodibenz[*b*,*f*]-[1,4]thiazepine by the Beckmann rearrangement of thioxanthone oxime. First, the synthesis of 5*H*-2-methoxycarbonyl-4-oxofuro[2,3-*b*][1,5]benzothiazepine (**1**) was attempted by the Beckmann rearrangement of the corresponding furothiochromone oxime (**6**). Methyl 5-(2-carboxyphenylthio)-2-furancarboxylate (**2**) was obtained from the nucleophilic substitution of methyl 5-nitro-2-furancarboxylate (**3**) using the disodium salt of thiosalicylic acid. In this reaction, the nitro group of **3** is a good leaving group.[3] Furthermore, **2** was treated with thionyl chloride to give methyl 5-(2-chloroformylphenylthio)-2-furancarboxylate (**4**), followed by the intramolecular Friedel–Crafts reaction of **4**, yielding 2-methoxycarbonylfuro[2,3-*b*]thiochromone (**5**). However, 2-methoxycarbonylfuro[2,3-*b*]thiochromone oxime (**6**) was not obtained from the reaction of **5** with hydroxylamine under various conditions. Nagarajan *et al.*[4] synthesized thioxanthone oxime from the reaction of thioxanthione with hydroxylamine. Compound **5** was treated with phosphorus pentasulfide to afford 2-methoxycarbonylfuro[2,3-*b*]thiochromthione (**7**), which was allowed to react with hydroxylamine to give **6** in moderate yield. The Beckmann rearrangement of **6** was attempted under various conditions (phosphorus pentachloride, polyphosphate ester, and formic acid), but the corresponding furobenzothiazepine was not obtained (Chart 1).

Next, the synthesis of **1** was attempted *via* another route. Compound **4**, obtained as already described, was treated with sodium azide to give methyl 5-(2-azidocarbonylphenylthio)-2-furancarboxylate (**8**). The thermolysis of **8** was then attempted in chlorobenzene (130 °C), but the expected product **1** was not obtained. Only the stable isocyanate, methyl 5-(2-isocyanatophenylthio)-2-furancarboxylate (**9**) was obtained. As **9** has the electron-withdrawing group at the 2-position of the furan ring, the reactivity of the furan ring at the 4-position was decreased; **1** was not obtained by the cyclization of **9**. Effenberger and Gleiter[5] reported that a Lewis acid (aluminum chloride) is useful for the acetylation of benzene derivatives by isocyanates. Therefore, **9** was heated in *o*-dichlorobenzene (150 °C) in the presence of aluminum chloride to give the new tricyclic compound **1** in 25% yield (Chart 2). The ^{1}H-NMR spectrum of **1** showed the furan ring proton at δ 7.44 (s, 1H) and the amide group proton at δ 10.51 (br s, 1H, exchangeable proton). The structure of **1** was supported by the ^{1}H-NMR, IR, and MS data and the results of elemental analyses.

Synthesis of 5*H*-2-methoxycarbonyl-4-oxofuro[2,3-*b*]-[1,5]benzoxazepine (**10**) was then attempted in a manner similar to that used for the synthesis of **1**. Methyl 5-(2-carboxyphenoxy)-2-furancarboxylate (**11**) could not be obtained by the reaction of **3** using the sodium salt of salicylic acid under various conditions. This may have been due to the difference in nucleophilicity between benzenethiolate and phenolate. It may also be difficult to form the disodium salt of salicylic acid, because the salicylate anion is very stable (Fig. 2).

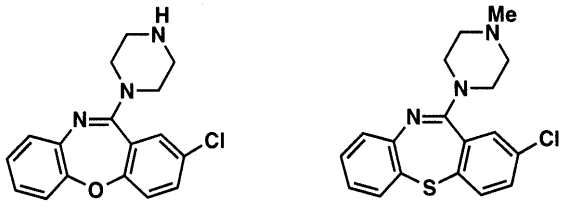

Fig. 1

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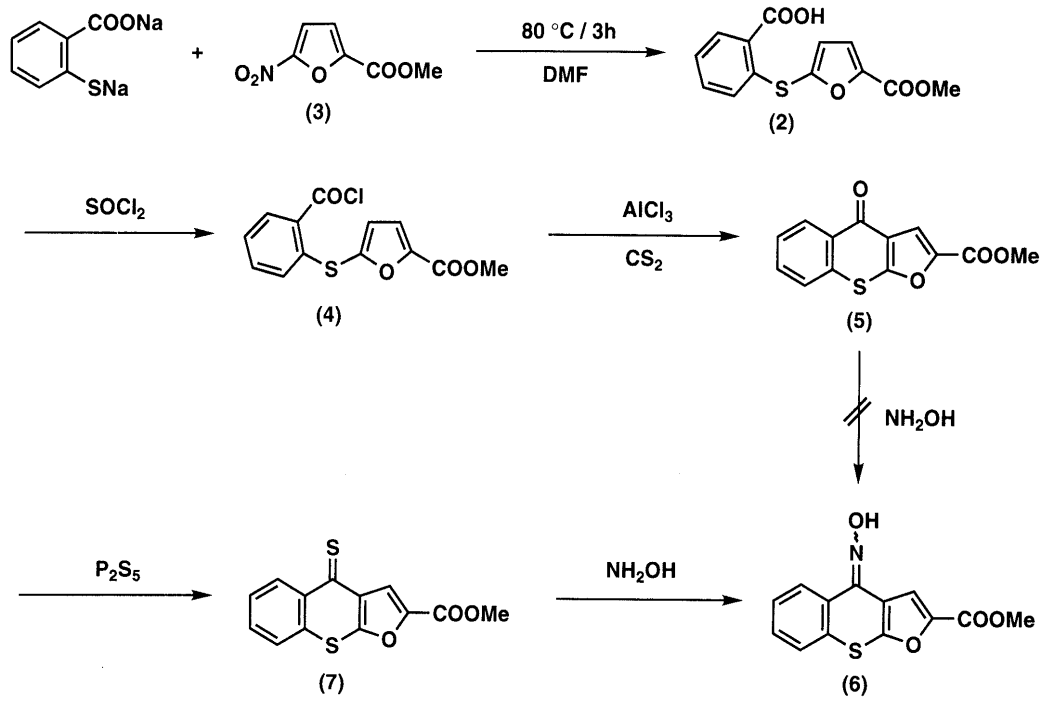


Chart 1

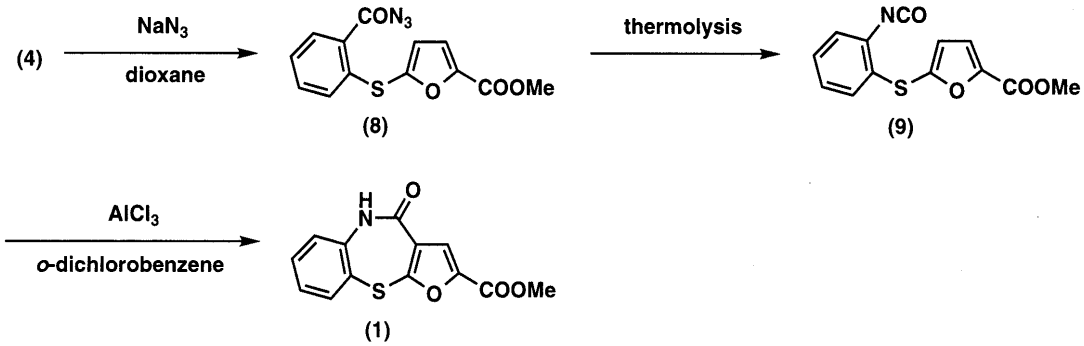
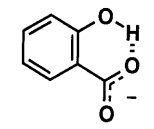


Chart 2



salicylate anion

Fig. 2

However, compound **3** was treated with the sodium salt of benzyl salicylate to give methyl 5-(2-benzyloxycarbonylphenoxy)-2-furancarboxylate (**12**) in 69% yield. Subsequent catalytic reduction of **12** gave the desired carboxylic acid **11**. Compound **11** was allowed to react with thionyl chloride to give methyl 5-(2-chloroformylphenoxy)-2-furancarboxylate (**13**) (Chart 3).

However, the desired methyl 5-(2-azidocarbonylphenoxy)-2-furancarboxylate (**14**) was not obtained by the reaction of **13** with sodium azide, but an intractable complex mixture was obtained. Compound **13** was treated with sodium azide in aqueous solution to give 2,2′-bis(5-methoxycarbonyl-2-furyloxy)carbanilide (**15**). As the azide compound **14** may be unstable, **14** was subjected to Curtius rearrangement to give the isocyanate compound (**16**), and **16** was then reacted with water to give methyl 5-(2-aminophenoxy)-2-furancarboxylate (**17**). Finally, **15** was obtained by the reaction of **16** with **17**. The ^{1}H-NMR spectrum of **15** showed the furan ring proton at δ 6.92 (d, 2H) and δ 5.22 (d, 2H), and the amide group proton at δ 8.26 (br s, 2H, exchangeable proton). The structure of **15** was supported by the ^{1}H-NMR, IR, and MS data and the results of elemental analyses. Compound **10** was not obtained *via* this route (Chart 4).

In conclusion, the new tricyclic compound **1** was obtained from **2**, which was prepared by nucleophilic substitution of **3** with the disodium salt of thiosalicylic acid, in four steps. Further, the furothiochromone **5** was synthesized by intramolecular Friedel–Crafts reaction in good yield. This method is also very useful for the synthesis of furothiochromones. Further studies of the chlorination of **1** are in progress in our laboratory to prepare tricyclic compounds with secondary amines at the 4-position of **1**. These results will be reported in due course.

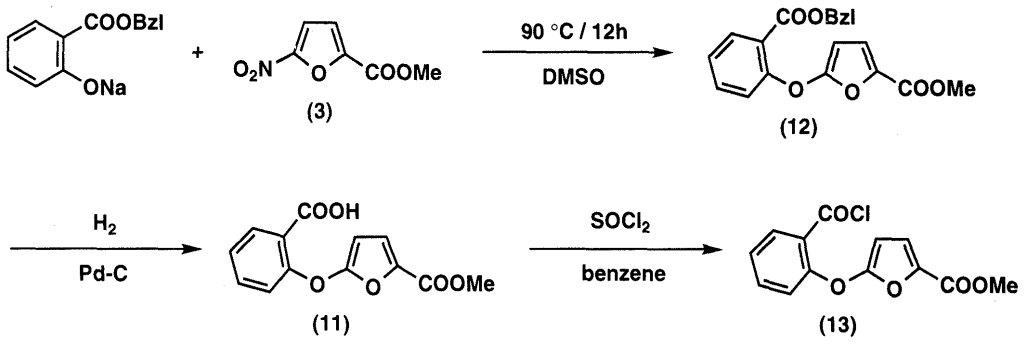


Chart 3

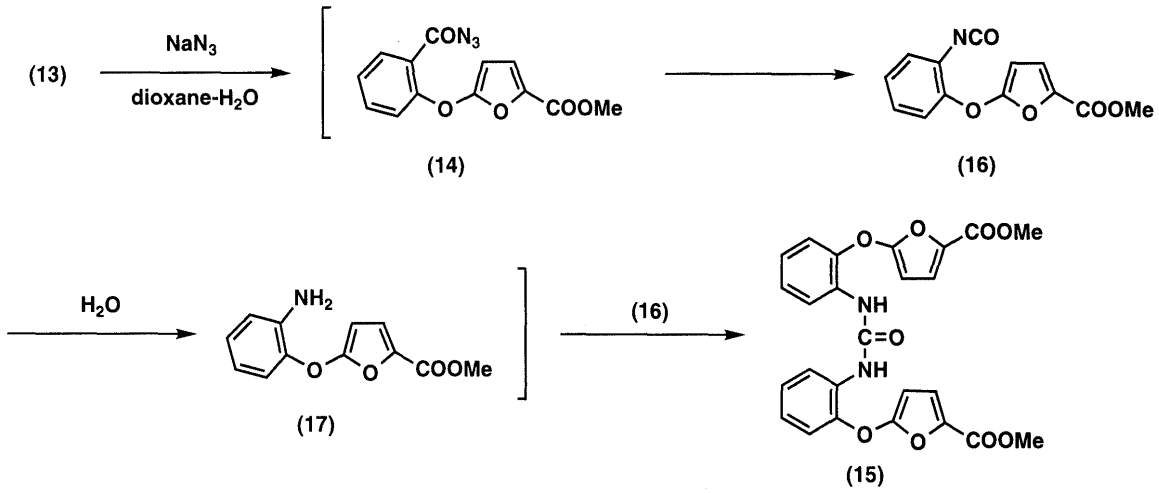


Chart 4

Experimental

All melting points (open capillaries) were determined using a Yamato MP-21 and are uncorrected. The ^{1}H-NMR spectra were determined at 60 MHz using a Nippon Denshi JNM PMR 60SI NMR spectrometer with tetramethylsilane (TMS) as an internal reference. The ^{1}H-NMR spectra of **1** and **15** were also determined at 270 MHz using a Nippon Denshi JEOL GX270FT NMR spectrometer. The IR spectra were measured using a JASCO IR-810 spectrometer. The MS were obtained on a Nippon Denshi DX-300 spectrometer at 70 eV. Chromatography was carried out using silica gel (Wakogel C-200, Wako Pure Chemical Industries, Ltd.). Ligroin refers to the fraction of bp 75—120 °C and petroleum benzin refers to the fraction of bp 50—90 °C.

Methyl 5-(2-Carboxyphenylthio)-2-furancarboxylate (2) A solution of thiosalicylic acid (30.8 g, 0.2 mol) in *N*,*N*-dimethylformamide (DMF) (240 ml) was added to a suspension of 60% sodium hydride (16.0 g, 0.4 mol) in DMF (240 ml) with stirring below 5 °C, and the mixture was warmed to 80 °C. To this mixture, a solution of **3** (34.2 g, 0.2 mol) in DMF (80 ml) was added and the whole was stirred for 3 h at 80 °C. It was then cooled to room temperature and poured into ice-cold water. The solution was made acidic with 10% hydrochloric acid and the resulting product was collected by filtration and purified by recrystallization from methanol to give 28.9 g (52%) of **2** as brown needles, mp 224—225 °C. IR (KBr): 3200—2800 (OH), 1725 (COOMe), 1680 (COOH) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 12.93 (1H, brs, OH, exchangeable proton), 7.97 (1H, dd, J=2, 8 Hz, Ph), 7.48—7.17 (2H, m, Ph), 7.42 (1H, d, J=4 Hz, F-3), 7.15 (1H, d, J=4 Hz, F-4), 6.67 (1H, dd, J=2, 8 Hz, Ph), 3.83 (3H, s, CH_3). MS *m/z*: 278 (M^+), 260 (M^+-H_2O), 247 (M^+−OMe). *Anal.* Calcd for $C_{13}H_{10}O_5S$: C, 56.11; H, 3.62. Found: C, 55.96; H, 3.67.

Methyl 5-(2-Chloroformylphenylthio)-2-furancarboxylate (4) A solution of **2** (20.0 g, 71.9 mmol) in thionyl chloride (100 ml) was refluxed with stirring for 30 min, then any excess thionyl chloride was evaporated under reduced pressure. The resulting product was purified by recrystallization from benzene–hexane to give 17.8 g (84%) of **4** as white needles, mp 115 °C. IR (KBr): 1760 (COCl), 1730 (COOMe) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 8.25 (1H, m, Ph), 7.57—7.33 (2H, m, Ph), 7.25 (1H, d, J=4 Hz, F-3), 6.90 (1H, d, J=4 Hz, F-4), 6.78 (1H, m, Ph), 3.87 (3H, s, CH_3). MS *m/z*: 298 (M^++2), 296 (M^+). *Anal.* Calcd for $C_{13}H_9ClO_4S$: C, 52.62; H, 3.06. Found: C, 52.82; H, 3.13.

2-Methoxycarbonylfuro[2,3-*b*]thiochromone (5) Aluminum chloride (10.8 g, 81 mmol) was added to a solution of **4** (12.0 g, 40.5 mmol) in carbon disulfide (120 ml), then the mixture was refluxed with stirring for 7 h and poured into ice-cold water. The resulting product was collected by filtration and purified by recrystallization from methanol to give 9.7 g (92%) of **5** as white needles, mp 164—165 °C. IR (KBr): 1740 (COOMe), 1645 (C=O) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 8.57 (1H, m, Ph), 7.80 (1H, s, F-3), 7.62—7.43 (3H, m, Ph), 3.93 (3H, s, CH_3). MS *m/z*: 260 (M^+), 229 (M^+−OMe). *Anal.* Calcd for $C_{13}H_8O_4S$: C, 59.99; H, 3.10. Found: C, 60.03; H, 3.20.

2-Methoxycarbonylfuro[2,3-*b*]thiochromthione (7) Phosphorus pentasulfide (4.0 g, 18 mmol) was added to a solution of **5** (4.0 g, 15.4 mmol) in pyridine (80 ml), and the mixture was heated with stirring at 120 °C for 40 min in an atmosphere of argon. At the end of the reaction, pyridine was evaporated under reduced pressure, then water was added to the residue. The resulting product was collected by filtration and purified by recrystallization from chloroform–hexane to give 4.1 g (98%) of **7** as a brown powder, mp 116 °C. IR (KBr): 1725 (COOMe) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 9.03 (1H, m, Ph), 8.00 (1H, s, F-3), 7.62—7.42 (3H, m, Ph), 3.93 (3H, s, CH_3). MS *m/z*: 276 (M^+), 245 (M^+−OMe). *Anal.* Calcd for $C_{13}H_8O_3S_2$: C, 56.51; H, 2.92. Found: C, 56.37; H, 2.99.

2-Methoxycarbonylfuro[2,3-*b*]thiochromone Oxime (6) Hydroxylamine hydrochloride (1.6 g, 23 mmol) was added to a solution of **7** (1.8 g, 6.5 mmol) in pyridine (16 ml), then the mixture was heated with stirring at 90 °C for 1 h and poured into ice-cold water. The resulting product was collected by filtration and purified by recrystallization from methanol to give 1.2 g (67%) of **6** as brown needles, mp 222 °C (dec.). IR (KBr): 3360 (OH), 1705 (COOMe) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 11.77 (1H, s, OH, exchangeable proton), 8.25 (1H, m, Ph), 8.08 (1H, s, F-3), 7.56—7.23 (3H, m, Ph), 3.83 (3H, s, CH_3). MS *m/z*: 275 (M^+), 244 (M^+−OMe). *Anal.* Calcd for $C_{13}H_9NO_4S$: C, 56.72; H, 3.30; N, 5.09. Found: C, 56.93; H, 3.36; N, 4.97.

Methyl 5-(2-Azidocarbonylphenylthio)-2-furancarboxylate (8) Sodi-

um azide (1.1 g, 16.9 mmol) was added to a solution of **4** (3.4 g, 11.5 mmol) in dioxane (34 ml), then the mixture was stirred for 24 h at room temperature and poured into ice-cold water. The resulting product was collected by filtration and purified by recrystallization from dichloromethane–ether to give 3.3 g (94%) of **8** as white needles, mp 111 °C (dec.). IR (KBr): 2130 (N_3), 1725 (COOMe), 1685 (CON_3) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 7.98 (1H, dd, J=2, 8 Hz, Ph), 7.53—7.05 (2H, m, Ph), 7.23 (1H, d, J=4 Hz, F-3), 6.90 (1H, d, J=4 Hz, F-4), 6.79 (1H, m, Ph), 3.90 (3H, s, CH_3). MS m/z: 275 ($M^+ - N_2$). *Anal.* Calcd for $C_{13}H_9N_3O_4S$: C, 51.48; H, 2.99; N, 13.85. Found: C, 51.55; H, 3.00; N, 13.61.

Methyl 5-(2-Isocyanatophenylthio)-2-furancarboxylate (9) A suspension of **8** (4.0 g, 13.2 mmol) in chlorobenzene (24 ml) was added to refluxing chlorobenzene (40 ml) over a period of 30 min. The mixture was refluxed with stirring for 30 min, and chlorobenzene was evaporated under reduced pressure. The resulting product was purified by recrystallization from ligroin to give 2.8 g (78%) of **9** as white needles, mp 92—93 °C. IR (KBr): 2220 (NCO), 1725 (COOMe) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.08 (4H, m, Ph), 7.17 (1H, d, J=4 Hz, F-3), 6.63 (1H, d, J=4 Hz, F-4), 3.83 (3H, s, CH_3). MS m/z: 275 (M^+), 244 ($M^+ - OMe$). *Anal.* Calcd for $C_{13}H_9NO_4S$: C, 56.72; H, 3.30; N, 5.09. Found: C, 56.87; H, 3.37; N, 4.81.

5*H*-2-Methoxycarbonyl-4-oxofuro[2,3-*b*][1,5]benzothiazepine (1) A suspension of aluminum chloride (1.2 g, 9.0 mmol) in *o*-dichlorobenzene (40 ml) was warmed to 90—100 °C. To this mixture, a solution of **9** (2.0 g, 7.3 mmol) in *o*-dichlorobenzene (20 ml) was added, and the whole was heated at 150 °C with stirring for 1 h, then poured into ice-cold water. The solution was extracted with dichloromethane. The dichloromethane layer was washed with brine, dried over anhydrous sodium sulfate, and evaporated, and the residue was purified by recrystallization from methanol to give 0.5 g (25%) of **1** as white needles, mp 226—228 °C. IR (KBr): 3180 (NH), 1745 (COOMe), 1675 (CONH) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 10.51 (1H, br s, NH, exchangeable proton), 7.53 (1H, dd, J=1.5, 7.7 Hz, Ph), 7.45 (1H, dt, J=1.5, 7.7 Hz, Ph), 7.44 (1H, s, F-3), 7.33 (1H, dd, J=1.5, 7.7 Hz, Ph), 7.23 (1H, dt, J=1.5, 7.7 Hz, Ph), 3.83 (3H, s, CH_3). MS m/z: 275 (M^+), 244 ($M^+ - OMe$), 216 ($M^+ - COOMe$). *Anal.* Calcd for $C_{13}H_9NO_4S$: C, 56.72; H, 3.30; N, 5.09. Found: C, 56.51; H, 3.32; N, 4.87.

Methyl 5-(2-Benzyloxycarbonylphenoxy)-2-furancarboxylate (12) Sodium hydride (60%, 8.5 g, 212.5 mmol) was added to a solution of benzyl salicylate (40 g, 175.4 mmol) in dimethyl sulfoxide (DMSO) (250 ml) with vigorous stirring. To this mixture, a solution of **3** (25.0 g, 146.2 mmol) in DMSO (50 ml) was added, and the whole was heated at 90 °C with stirring for 12 h and poured into ice-cold water. The solution was next extracted with benzene. The benzene layer was washed with brine and dried over anhydrous sodium sulfate. The extract was evaporated and the residue was purified by chromatography on silica gel with benzene to give 35.5 g (69%) of **12** as a yellowish liquid. Compound **12** could not be distilled even under reduced pressure. IR (neat): 1730 (COOMe and COOBzl) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 7.90 (1H, dd, J=2, 7 Hz, Ph), 7.57—7.10 (3H, m, Ph), 7.28 (5H, s, Ph), 7.00 (1H, d, J=4 Hz, F-3), 5.26 (1H, d, J=4 Hz, F-4), 5.23 (2H, s, CH_2), 3.77 (3H, s, CH_3). MS m/z: 352 (M^+), 321 ($M^+ - OMe$), 261 ($M^+ - Bzl$).

Methyl 5-(2-Carboxyphenoxy)-2-furancarboxylate (11) A solution of **12** (20.0 g, 56.8 mmol) in methanol (350 ml) was hydrogenated over 5% palladium carbon (3.5 g) as a catalyst at atmospheric pressure. The catalyst was filtered off, and the solvent was evaporated under reduced pressure. The residue was purified by recrystallization from methanol–water to give 9.3 g (62%) of **11** as white needles, mp 134—136 °C. IR (KBr): 3130—2825 (OH), 1715 (COOMe), 1690 (COOH) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 13.16 (1H, br s, OH, exchangeable proton), 7.85 (1H, dd, J=2, 8 Hz, Ph), 7.63—7.43 (2H, m, Ph), 7.22 (1H, d, J=4 Hz, F-3), 7.20 (1H, dd, J=2, 8 Hz, Ph), 5.53 (1H, d, J=4 Hz, F-4), 3.73 (3H, s, CH_3); MS m/z: 262 (M^+), 231 ($M^+ - OMe$). *Anal.* Calcd for $C_{13}H_{10}O_6$: C, 59.55; H, 3.84. Found: C, 59.39; H, 3.88.

Methyl 5-(2-Chloroformylphenoxy)-2-furancarboxylate (13) Thionyl chloride (40 ml) was added to a solution of **11** (8.0 g, 30.5 mmol) in benzene (160 ml), and the mixture was refluxed with stirring for 8 h. The excess thionyl chloride and benzene were evaporated under reduced pressure, and the resulting product was purified by recrystallization from petroleum benzin to give 6.5 g (76%) of **13** as yellowish needles, mp 77 °C. IR (KBr): 1785 (COCl), 1720 (COOMe) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 8.02 (1H, dd, J=2, 8 Hz, Ph), 7.42 (1H, m, Ph), 7.13 (1H, m, Ph), 7.00 (1H, d, J=4 Hz, F-3), 6.98 (1H, dd, J=2, 8 Hz, Ph), 5.58 (1H, d, J=4 Hz, F-4), 3.56 (1H, s, CH_3). MS m/z: 282 ($M^+ + 2$), 280 (M^+). *Anal.* Calcd for $C_{13}H_9ClO_5$: C, 55.63; H, 3.23. Found: C, 55.56; H, 3.29.

2,2′-Bis(5-methoxycarbonyl-2-furyloxy)carbanilide (15) A solution of sodium azide (0.6 g, 9.2 mmol) in water (6 ml) was added to a solution of **13** (2.0 g, 7.1 mmol) in dioxane (20 ml). The mixture was stirred for 24 h at room temperature and poured into ice-cold water. The solution was extracted with ether. The ether layer was washed with brine and then dried over anhydrous sodium sulfate. The extract was evaporated and the residue was purified by recrystallization from methanol–water to give 1.0 g (56%) of **15** as white needles, mp 160—161 °C. IR (KBr): 3375 (NH), 1720 (COOMe), 1690 (NHCONH) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 8.42 (2H, dd, J=1.5, 8.0 Hz, Ph×2), 8.26 (2H, br s, NH×2, exchangeable proton), 7.19 (2H, dt, J=1.5, 8.0 Hz, Ph×2), 7.01 (2H, dd, J=1.5, 8.0 Hz, Ph×2), 6.92 (2H, d, J=3.7 Hz, F-3×2), 6.91 (2H, dt, J=1.5, 8.0 Hz, Ph×2), 5.23 (2H, d, J=3.7 Hz, F-4×2), 3.84 (6H, s, CH_3×2). MS m/z: 492 (M^+). *Anal.* Calcd for $C_{25}H_{20}N_2O_9$: C, 60.98; H, 4.09; N, 5.69. Found: C, 60.89; H, 4.19; N, 5.71.

References and Notes

1) *a*) Tanaka A., Yakushijin K., Yoshina S., *J. Heterocycl. Chem.*, **14**, 975—979 (1977); *b*) *Idem*, *ibid.*, **15**, 123—125 (1978); *c*) *Idem*, *ibid.*, **16**, 785—788 (1979).
2) *a*) Nakashima Y., Kawashima Y., Amanuma F., Sota K., Tanaka A., Kameyama T., *Chem. Pharm. Bull.*, **32**, 4271—4280 (1984); *b*) Nakashima Y., Kawashima Y., Sato M., Okuyama S., Amanuma F., Sota K., Kameyama T., *ibid.*, **33**, 5250—5257 (1985); *c*) Kawashima Y., Sato M., Hatada Y., Okuyama S., Amanuma F., Nakashima Y., Sota K., Moriguchi I., *ibid.*, **34**, 3267—3272 (1986); *d*) Kawashima Y., Hosoda K., Goto J., Sato M., Hatada Y., Asami Y., Nakashima Y., Sato K., *ibid.*, **34**, 5149—5153 (1986); *e*) Kawashima Y., Amanuma F., Sato M., Okuyama S., Nakashima Y., Sato K., Moriguchi I., *J. Med. Chem.*, **29**, 2284—2290 (1986).
3) *a*) Tanaka A., Usui T., Shimadzu M., *Chem. Pharm. Bull.*, **28**, 2846—2849 (1980); *b*) *Idem*, *J. Heterocycl. Chem.*, **18**, 1241—1244 (1981); *c*) Shimadzu M., Ishikawa N., Yamamoto K., Tanaka A., *ibid.*, **23**, 1179—1182 (1986).
4) Nagarajan K., Kulkarni C. L., Venkateswarlu A., *Indian J. Chem.*, **12**, 247—251 (1974).
5) Effenberger F., Gleiter R., *Chem. Ber.*, **97**, 472—479 (1964).