Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

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Published online: 21 Aug 2006.

To cite this article: Koyo Matsuda , Isao Yanagisawa , Yasuo Isomura , Toshiyasu Mase & Tadao Shibanuma (1997) Alternative Synthesis of Dibenzo-and Dipyrido-[1,3]Diazepines from Thioamides and o,o′-Diaminobiaryls, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 27:14, 2393-2402, DOI: 10.1080/00397919708004101

To link to this article: http://dx.doi.org/10.1080/00397919708004101

SYNTHETIC COMMUNICATIONS, 27(14), 2393-2402 (1997)

ALTERNATIVE SYNTHESIS OF DIBENZO-AND DIPYRIDO-[1,3]DIAZEPINES FROM THIOAMIDES AND o,o'-DIAMINOBIARYLS

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Abstract: Thioamides were treated with iodomethane, and then o,o'-diaminobiphenyl or bipyridyl to afford 6-substituted-5H-dibenzo- or dipyrido [1,3]diazepines in good yields.

Dibenzo- and dipyrido[1,3]diazepines are of pharmaceutical interest, and known to be prepared from imino ester salts and o,o'-diaminobiaryls.[1] Dipyrido[1,3]diazepines have been reported to have activities on central nervous systems.[1c] In the course of our studies on hypolipidemic agents, we prepared these ring systems in a different way from the imino ester method, that is the thioimino ester method as shown below.

The imino methyl thioester salts were prepared by methylation of thioamides with iodomethane at 60° in acetone. The reaction was completed in 5

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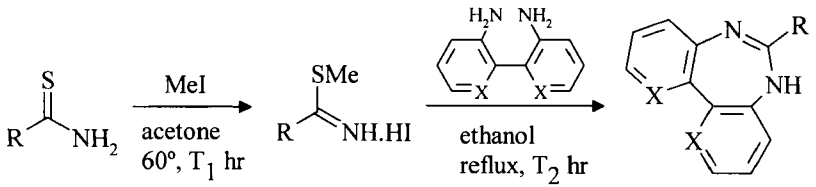
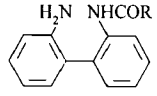


Table

R	T_1(hr)	X	T_2(hr)	Yield(%)	Lit.Yield(%)[a]
Phenyl	3	CH	1	94	68[1a]
Phenyl	3	N	1	77	85[1c]
4-Chlorophenyl	4	CH	1	93	51[1b]
4-Chlorophenyl	4	N	1	87	
3-Chloro-4-fluorophenyl	5	CH	2	98	
2-Chlorophenyl	4	CH	17	87	
4-Methylphenyl	2	CH	3	81	17.5[1b]
4-Trifluoromethylphenyl	5	CH	2	92	
4-Trifluoromethylphenyl	5	N	2	96	
4-Biphenylyl	2	CH	2	84	
2-Naphthyl	3	CH	3	89	60[1a]
2-Thienyl	5	CH	2	72	72[1b]
2-Thienyl	5	N	3	90	
3-Thienyl	3	CH	2	84	
2-Methylthiazol-4-yl	3	CH	2	84[b]	
Ethyl	1	CH	1	83[b]	

a) Lit. Yields are by the imino ester method.
b) N-Monoacylated diaminobiphenyl derivative was obtained.



hr. to provide the imino methyl thioester hydroiodides quantitatively. This process appears to be easier than the typical imino ester formation from nitriles with gaseous hydrogen chloride which is slower with an occasional low yield.[2]

These salts reacted with o,o'-diaminobiaryls in refluxing ethanol to form [1,3]diazepine derivatives in yields comparative to those obtained by the imino

ester method (Table). The reaction worked on aromatic and several heteroaromatic substituents such as phenyl, 4- or 2-chlorophenyl, 3-chloro-4-fluorophenyl, 4-methylphenyl, 4-trifluoromethylphenyl, 4-biphenylyl, 2-naphthyl, and 2- or 3-thienyl. The cyclization was completed in 3 hr. except for 2-chlorophenyl substituent which needed a longer reaction time because of a slow reaction, in which the ratios of the product and the starting diaminobiphenyl by HPLC after refluxing 1, 2, 4, and 17 hr. were 0.94, 1.38, 2.68, and 42.35, respectively.

In the case of 2-methylthiazol-4-yl and ethyl substituents, N-monoacylated diaminobiphenyl derivatives which seemed to be resulted by hydrolysis of the reaction intermediate were obtained (Table). In these two reactions, the starting materials were consumed smoothly to form a sole product which could not be isolated in pure form but changed gradually during work-up, presumably hydrolysis, to give the N-monoacylated product. The reaction did not give the cyclized product even with a longer reaction time. The formation of N-propionyl diaminobiphenyl is consistent with the result by the imino ester method in which the similar N-monoacylated product was obtained for aliphatic substituents.[1b] On the other hand, the difference between 2-methylthiazol-4-ly and 2- or 3-thienyl is interesting, because the former gave the N-acylated product while the latter gave the cyclized products.

Although these reaction conditions were applied to thionicotinamide, it did not give the product, while the corresponding imino ester method gave the [1,3]diazepine derivative.[1a]

EXPERIMENTAL

Melting points were determined on a Yanaco MP-500D apparatus and are uncorrected. IR spectra were recorded as KBr pellets on a HITACHI 270-30 spectrometer. NMR spectra were measured on a JEOL-EX-90, JNM-EX-400, or JNM-EX-500 spectrometers and the chemical shifts are expressed in ppm with TMS as an internal standard. MS spectra were recorded on a HITACHI M-80 or JEOL-JMS-DX300 apparatus. Column chromatography was carried out using Merck Kieselgel 60(230-400 mesh). TLC was done on Merck DC-Fertigplatten Kieselgel 60 F_{254}. And HPLC was carried out using TOSOH Tsk gel ODS-80T_M column with HITACHI L-6200 Intelligent Pump, L-4000 UV Detecter, AS-2000 Autosampler, and D-2500 Chromato-Integrator under the following conditions (solvent: MeCN/0.01M KH_2PO_4=7/3; flow rate: 1.00 ml/min.; UV: 254 nm).

2,2'-Diaminobiphenyl was prepared by reduction of commercially available 2,2'-dinitrobiphenyl in quantitative yield using tin(II) chloride dihydrate(conc. HCl/ethanol at room temperature), mp. 74-75°; lit.[3] mp. 79-80°; IR: 3412, 3298, 3202, 1638, 1569, 1500, 1488, 1449, 1305, 1293, 1257, 864, 753, and 699 cm^{-1}; ^1H NMR ($CDCl_3$): δ 3.55(4H, br-s), 6.80(4H, m), and 7.17(4H, m). 3,3'-Diamino-2,2'-bipyridyl was prepared from 2-chloro-3-nitropyridine in two steps according to a reported procedure.[4] 2-Chloro-3-nitropyridine was coupled in the presence of copper in N,N-dimethylformamide at 150°(bath temp.) for 2 hr. to give 3,3'-dinitro-2,2'-bipyridyl in 83% yield which was reduced as above to afford 3,3'-diamino-2,2'-bipyridyl in 47% yield, mp. 130-132°; IR: 3382, 3238, 1587, 1446, 1434, 1317, 1272, 1149, 1062, 798, 729, 639, and 540 cm^{-1}; ^1H NMR ($CDCl_3$): δ 6.27(4H, Br-s), 7.03(4H, s), and 7.97(2H, s); MS (FAB): m/e 187($(M+H)^+$, base peak); *Anal.* Calcd. for $C_{10}H_{10}N_4$: C, 64.50; H, 5.41; N, 30.09, Found: C, 64.38; H, 5.30; N, 30.02. 4-Phenylthiobenzamide, 2-thionaphthamide, and thiophene-3-thiocarboxamide were prepared from the corresponding nitrile with diethyl dithiophosphate[5] in 4N HCl/ethyl acetate at room temperature overnight in 99, 85, and 84% yield, respectively. 4-Phenylthiobenzamide, mp. 228-229°; lit.[6] mp. 218°; IR: 3358, 3292, 3166, 1635, 1416, 1302, 882, 846, 768, 726, and 693 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.3-7.6(3H, m), 7.73(4H, m), 8.01(2H, m), 9.54(1H, s), and 9.89(1H, s); MS (FAB): m/e 214($(M+H)^+$, base peak). 2-Thionaphthamide, mp. 152-153.5°; IR: 3382, 3304, 3178, 1629, 1407, 1368, 1353, 1317, 1284, 1098, 912, 903, 867, 834, 816, 747, and 720 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.60(2H, m)

7.95(2H, m), 8.04(2H, m), 8.45(1H, s), 9.67(1H, s), and 9.98(1H, s); MS (FAB): m/e 188($(M+H)^+$, base peak). Thiophene-3-thiocarboxamide, mp. 109-110°; lit.[7] mp. 111°; IR: 3352, 3304, 3172, 3082, 1635, 1515, 1437, 1398, 1353, 1302, 1218, 1140, 945, 846, 804, 786, and 675 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.17(1H, br-s), 7.32(1H, dd, J=3.1 and 5.2 Hz), 7.50(1H, dd, J=1.3 and 5.2 Hz), 7.64(1H, br-s), and 7.99(1H, dd, J=1.3 and 3.1 Hz); MS (FAB): m/e 188($(M+H)^+$, base peak).

Commercially available thiobenzamide, 4-chlorothiobenzamide, 3-chloro-4-fluorothiobenzamide, 2-chlorothiobenzamide, 4-methylthiobenzamide, 4-trifluoromethylthiobenzamide, thiophene-2-thiocarboxamide, 2-methylthiazole-4-thiocarboxamide, and thiopropionamide were used without a further purification.

Preparation of [1,3]diazepines and N-monoacylated diaminobiphenyls (general procedure).-Methanethiol formed *in situ* is highly toxic. All manipulations should be performed under a well ventilated hood. A mixture of thioamide (2.4 mmol) and iodomethane (7.2 mmol) in acetone (12 ml) was heated at 60°(bath temp.) for T_1 hr. (shown in Table), then the solvents were evaporated to leave the imino methyl thioester salt (^1H NMR spectral data were shown below). This salt was refluxed with o,o'-diaminobiaryl (2.0 mmol) in ethanol (20 ml) for T_2 hr. (shown in Table). Chloroform (100 ml) was added and the mixture was washed with 1N NaOH (60 ml x 1) and sat. NaCl (50 ml x 1), dried over anhydrous $MgSO_4$, and evaporated. The residue was crystallized from hexane-diethyl ether, collected and dried *in vacuo* to afford [1,3]diazepine in yield shown in Table. As for N-monoacylated diaminobiphenyl derivative, the residue was chromatographed on silica gel with chloroform as eluent, then crystallized from hexane-diethyl ether to give the product .

^1H NMR spectral data of imino methyl thioester salt intermediate: R=phenyl, ^1H NMR (DMSO-d_6): δ 2.88(3H, s), 7.5-8.0(5H, m), and 11.6(2H, br); R=4-chlorophenyl, ^1H NMR (DMSO-d_6): δ 2.88(3H, s), 7.75(2H, d, J=9.0 Hz), 7.92(2H, d, J=9.0 Hz), and 11.37(2H, br); R=3-chloro-4-fluorophenyl, ^1H NMR (DMSO-d_6): δ 2.84(3H, s), 7.8-8.3(3H, m), and 9.60(2H, br); R=2-chlorophenyl, ^1H NMR (DMSO-d_6): δ 2.84(3H, s), 7.73(4H, m), and 11.99(2H, br); R=4-methylphenyl, ^1H NMR (DMSO-d_6): δ 2.44(3H, s), 2.86(3H, s), 7.49(2H, d, J=8.2 Hz), 7.81(2H, d, J=8.2 Hz), and 11.46(2H, br); R=4-trifluoromethylphenyl, ^1H NMR (DMSO-d_6): δ 2.90(3H, s), 8.07(4H, s), and 10.47(2H, br); R=biphenyl-4-yl, ^1H NMR (DMSO-d_6): δ 2.88(3H, s), 7.52(3H, m), 7.81(2H, m), 8.00(4H, s), and 11.71(2H, br); R=2-naphthyl, ^1H NMR (DMSO-d_6): δ 2.93(3H, s), 7.76(2H, m),

7.89(1H, m); 8.11(1H, m), 8.21(2H, m), 8.62(1H, m), and 11.81(2H, br); R=2-thienyl, ^{1}H NMR (DMSO-d_6): δ 2.91(3H, s), 7.24(1H, m), 8.23(2H, m), and 9.50(2H, br); R=3-thienyl, 2.86(3H, s), 7.69(1H, m), 7.92(1H, m), 8.76(1H, m), and 11.49(2H, br); R=2-methylthiazol-4-yl, ^{1}H NMR (DMSO-d_6): δ 2.77(3H, s), 2.81(3H, s), 8.90(1H, s), and 11.60(2H, br); R=ethyl, ^{1}H NMR (DMSO-d_6): δ 1.45(3H, t, J=7.7 Hz), 2.96(3H, s), 3.11(2H, q, J=7.7 Hz), and 11.26(2H, br).

6-Phenyl-5H-dibenzo[d,f][1,3]diazepine, Yellow needles (benzene) mp. 165.5-166.5°; lit.[1a] mp. 165°; IR: 3262, 3070, 3034, 1638, 1581, 1482, 1437, 1287, 1251, 771, 756, 744, and 696 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.12(4H, m), 7.34(4H, m), 7.52(3H, m), 7.98(2H, m), and 8.45(1H, s); MS (FAB): m/e 271($(M+H)^+$, base peak).

Anal. Calcd. for $C_{19}H_{14}N_2$: C, 84.42; H, 5.22; N, 10.36

Found: C, 84.62; H, 5.00; N, 10.39

6-Phenyl-5H-dipyrido[3,2-d:2',3'-f][1,3]diazepine, Yellow needles (chloroform) mp. 305-306°; lit.[1c] mp. >300°; IR: 3472, 3190, 3070, 2998, 1641, 1566, 1515, 1464, 1452, 1440, 1419, 1293, 813, and 699 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.3-7.6(7H, m), 7.99(2H, m), 8.41(2H, m), and 8.79(1H, s); MS (FAB): m/e 273($(M+H)^+$, base peak).

Anal. Calcd. for $C_{17}H_{12}N_4$: C, 74.98; H, 4.44; N, 20.57

Found: C, 75.07; H, 4.42; N, 20.55

6-(4-Chlorophenyl)-5H-dibenzo[d,f][1,3]diazepine, Yellow needles (benzene). mp. 163-164°; lit.[1b] mp. 157-158°; IR: 3286, 1638, 1593, 1482, 1437, 1320, 1299, 1107, 1095, 762, 753, and 723 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.10(4H, m), 7.29(2H, m), 7.37(2H, m), 7.57(2H, d, J=8.6 Hz), 8.00(2H, d, J=8.6 Hz), and 8.51(1H, s); MS: m/e 304(M^+), 167(base peak).

Anal. Calcd. for $C_{19}H_{13}N_2Cl$: C, 74.88; H, 4.30; N, 9.19; Cl, 11.63

Found: C, 74.98; H, 4.25; N, 9.24; Cl, 11.89

6-(4-Chlorophenyl)-5H-dipyrido[3,2-d:2',3'-f][1,3]diazepine, Golden yellow needles (chloroform-ethanol). mp. 321.5-322.5°; lit[1b] mp. 157-158°; IR: 3232, 3064, 3004, 1647, 1572, 1518, 1461, 1440, 1416, 1314, 1293, 1272, 1104, 1095, 822, 810, and 732 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.37(2H, m), 7.44(1H, m), 7.50(1H, m), 7.60(2H, m), 8.01(2H, m), 8.38(1H, m), 8.44(1H, m), and 8.85(1H, s); MS (FAB): m/e 307($(M+H)^+$, base peak).

Anal. Calcd. for $C_{17}H_{11}N_4Cl$: C, 66.56; H, 3.61; N, 18.26; Cl, 11.56

Found: C, 66.61; H, 3.59; N, 18.37; Cl, 11.80

6-(3-Chloro-4-fluorophenyl)-5H-dibenzo[d,f][1,3]diazepine, Pale yellow needles (benzene). mp. 181.5-182.5°; IR: 3214, 3070, 1641, 1626, 1599, 1581, 1497, 1485, 1437, 1323, 1302, 1266, 819, 756, and 723 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.1-7.4(8H, m), 7.57(1H, t, J=9.0 Hz), 8.01(1H, m), 8.15(1H, m), and 8.56(1H, s); MS: m/e 322(M^+), 167(base peak).

Anal. Calcd. for $C_{19}H_{12}N_2ClF$: C, 70.70; H, 3.75; N, 8.68; Cl, 10.98; F, 5.89
Found: C, 70.62; H, 3.63; N, 8.76; Cl, 11.25; F, 5.79

6-(2-Chlorophenyl)-5H-dibenzo[d,f][1,3]diazepine, Yellow needles (hexane-benzene). mp. 160-161°; IR: 3394, 3214, 3104, 3070, 1641, 1614, 1584, 1521, 1482, 1437, 1390, 1302, 1272, 1257, 1110, 762, 726, and 693 cm^{-1}; ^1H NMR (DMSO-d_6): δ 6.86(1H, d, J=8.4 Hz), 6.95(1H, d, J=7.2 Hz),7.05(1H, t, J=7.2 Hz), 7.1-7.6(9H, m), and 8.44(1H, s); MS (FAB): m/e 305((M+H)$^+$, base peak).

Anal. Calcd. for $C_{19}H_{13}N_2Cl$: C, 74.88; H, 4.30; N, 9.19; Cl, 11.63
Found: C, 74.83; H, 4.35; N, 9.26; Cl, 11.53

6-(4-Methylphenyl)-5H-dibenzo[d,f][1,3]diazepine, Yellow needles (hexane-benzene). mp. 138.5-139.5°; lit.[1b] mp. 135-136°; IR: 3334, 3040, 1635, 1614, 1587, 1572, 1476, 1437, 1428, 1317, 1284, 1248, 816, 750, 723, and 672 cm^{-1}; ^1H NMR (DMSO-d_6): δ 2.38(3H, s), 7.0-7.5(10H, m), 7.89(2H, d, J=8.8 Hz), and 8.38(1H, s); MS: m/e 284(M^+), 167(base peak).

Anal. Calcd. for $C_{20}H_{16}N_2$: C, 84.48; H, 5.67; N, 9.85
Found: C, 84.68; H, 5.73; N, 9.78

6-(4-Trifluoromethylphenyl)-5H-dibenzo[d,f][1,3]diazepine, Yellow prisms (hexane-benzene). mp. 166.5-167.5°; IR: 3268, 1635, 1581, 1482, 1437, 1326, 1293, 1173, 1122, 1083, 1065, 1014, 834, 750, 729, and 681 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.0-7.5(8H, m), 7.88(2H, d, J=8.4 Hz), 8.16(2H, d, J=8.4 Hz), and 8.62(1H, s); MS (FAB): m/e 339((M+H)$^+$, base peak).

Anal. Calcd. for $C_{20}H_{13}N_2F_3$: C, 71.00; H, 3.87; N, 8.28; F, 16.85
Found: C, 70.89; H, 3.93; N, 8.39; F, 16.58

6-(4-Trifluoromethylphenyl)-5H-dipyrido[3,2-d:2',3'-f][1,3]diazepine, Yellow needles (hexane-chloroform). mp. 264.5-265.5°; IR: 3238, 1653, 1527, 1464, 1443, 1419, 1332, 1320, 1293, 1167, 1128, 1113, 1065, 1017, 810, 741, and 687 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.39(2H, dd, J=8.2 and 4.2 Hz), 7.50(2H, d, J=8.2 Hz), 7.90(2H, d, J=8.0 Hz), 8.19(2H, d, J=8.0 Hz), 8.43(2H, m), and 8.98(1H, s); MS (FAB): m/e 341((M+H)$^+$, base peak).

Anal. Calcd. for $C_{18}H_{11}N_4F_3$: C, 63.53; H, 3.26; N, 16.46; F, 16.75
Found: C, 63.41; H, 3.32; N, 16.56; F, 16.45

6-(Biphenyl-4-yl)-5H-dibenzo[d,f][1,3]diazepine, Yellow needles (benzene) mp. 181-182°; IR: 3328, 3070, 1635, 1611, 1584, 1479, 1425, 1293, 759, 747, 726, and 696 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.1-7.6(11H, m), 7.76(2H, m), 7.82(2H, d, J=8.6 Hz), 8.09(2H, d, J=8.6 Hz), and 8.52(1H, s); MS (FAB): m/e 347($(M+H)^+$, base peak).

Anal. Calcd. for $C_{25}H_{18}N_2$: C, 86.68; H, 5.24; N, 8.09

Found: C, 86.52; H, 5.18; N, 8.05

6-(2-Naphthyl)-5H-dibenzo[d,f][1,3]diazepine, Yellow prisms (benzene) mp. 171-172°; lit.[1a] 176-178°; IR: 3268, 3070, 1626, 1578, 1482, 1434, 1287, 1272, 1251, 1233, 1191, 810, 759, 750, 729, and 693 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.1-7.5(8H, m), 7.62(2H, m), 7.9-8.2(4H, m), 8.56(1H, s), and 8.61(1H, s); MS: m/e 320(M^+), 167(base peak).

Anal. Calcd. for $C_{23}H_{16}N_2$: C, 86.22; H, 5.03; N, 8.74

Found: C, 86.35; H, 5.09; N, 8.63

6-(2-Thienyl)-5H-dibenzo[d,f][1,3]diazepine, Yellow needles (benzene) mp. 161-162°; lit.[1b] mp. 164°; IR: 3394, 3100, 1635, 1584, 1467, 1431, 1416, 1281, 1239, 771, 753, and 723 cm^{-1}; ^1H NMR ($CDCl_3$): δ 6.16(1H, s), 6.76(1H, m), and 7.1-7.5(10H, m); MS (FAB): m/e 277($(M+H)^+$, base peak).

Anal. Calcd. for $C_{17}H_{12}N_2S$: C, 73.88; H, 4.38; N, 10.14 S, 11.60

Found: C, 73.88; H, 4.38; N, 10.27 S, 11.30

6-(2-Thienyl)-5H-dipyrido[3,2-d:2',3'-f][1,3]diazepine, Yellow needles (hexane-chloroform). mp. 297-298°; IR: 3196, 3112, 3004, 1638, 1572, 1518, 1464, 1428, 1416, 1317, 1290, 813, and 714 cm^{-1}; ^1H NMR ($CDCl_3$): δ 6.33(1H, s), 7.10(2H, m), 7.20(1H, m), 7.28(1H, m), 7.48(3H, m), and 8.55(2H, m); MS (FAB): m/e 279($(M+H)^+$, base peak).

Anal. Calcd. for $C_{15}H_{10}N_4S$: C, 64.73; H, 3.62; N, 20.13; S, 11.52

Found: C, 64.45; H, 3.57; N, 20.01; S, 11.62

6-(3-Thienyl)-5H-dibenzo[d,f][1,3]diazepine, Yellow prisms (hexane-benzene) mp. 151-152°; IR: 3382, 3100, 3064, 1647, 1584, 1476, 1428, 1275, 1239, 801, 762, 750, 726, and 678 cm^{-1}; ^1H NMR (DMSO-d_6): δ 7.12(4H, m), 7.27(2H, m), 7.37(2H, m), 7.62(1H, m), 7.67(1H, m), 8.22(1H, s), and 8.34(1H, m); MS (FAB): m/e 277($(M+H)^+$, base peak).

Anal. Calcd. for $C_{17}H_{12}N_2S$: C, 73.88; H, 4.38; N, 10.14 S, 11.60

Found: C, 74.03; H, 4.21; N, 10.09 S, 11.42

2-(2-Methylthiazol-4-ylcarbonylamino)-2'-aminobiphenyl, Colorless needles (benzene) mp. 151-152°; IR: 3340, 1677, 1629, 1611, 1587, 1545, 1515, 1497,

1452, 1317, 1299, 1179. 792, and 756 cm^{-1}; ^1H NMR (DMSO-d_6): δ 2.57(3H, s), 4.74(2H, s), 6.70(1H, m), 6.88(1H, d, J=8.0 Hz), 6.97(1H, m), 7.1-7.3(3H, m), 7.42(1H, m), 8.20(1H, s), 8.30(1H, d, J=8.0 Hz), and 9.73(1H, s); MS (FAB): m/e 310($(M+H)^+$, base peak).

Anal. Calcd. for $C_{17}H_{15}N_3OS$: C, 66.00; H, 4.89; N, 13.58 S, 10.36

Found: C, 66.05; H, 4.80; N, 13.49 S, 10.32

2-(Propionylamino)-2'-aminobiphenyl, Colorless prisms (diethyl ether) mp. 65-66°; IR: 3334, 2986, 1701, 1683, 1614, 1584, 1524, 1497, 1476, 1449, 1296, 1281, 1191, 759, 681, 621, and 501 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.08(3H, t, J=7.2 Hz), 2.23(2H, q, J=7.2 Hz), 3.61(2H, br), 6.8-6.9(2H, m), 7.08(1H, m), 7.1-7.3(3H, m), 7.39(1H, m), 7.51(1H, br), and 8.25(1H, d, J=7.6 Hz); MS (FAB): m/e 241($(M+H)^+$, base peak).

Anal. Calcd. for $C_{15}H_{16}N_2O$: C, 74.97; H, 6.71; N, 11.66

Found: C, 75.00; H, 6.80; N, 11.64

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(Received in Japan 10 January 1997)