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Studies on Organometallic Compounds. II.[1] Facile and Convenient Method for the Synthesis of Iodoazines through Iododestannation of Trimethylstannylazines[2]

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Trimethylstannyl and bis(trimethylstannyl) derivatives of pyridine, quinoline, and isoquinoline were prepared from the corresponding halo and dihalo (chloro or bromo) derivatives and trimethylstannyl sodium, which was generated *in situ* from chlorotrimethylstannane and sodium. These stannyl derivatives readily underwent iododestannation on treatment with iodine to produce the corresponding iodo and diiodo derivatives of pyridine, quinoline, and isoquinoline, respectively, in good yields.

Keywords——trimethylstannylazine; bis(trimethylstannyl)azine; iodoazine; diiodoazine; iododestannation; trimethylstannyl sodium; chlorotrimethylstannane

Iodoazines, which have potential utility in the synthetic chemistry of *N*-heteroaromatics, can be obtained by several methods (for example, Sandmeyer reaction of aminoazines,[3] reactions of nitroaminopyridines with phosphorus triiodide,[4] and halogen interconversion of chloro derivatives with iodides.[5] However, all of the above procedures possess limitations in their scope. On the other hand, halodemetallation has been well investigated theoretically and practically.[6] We were thus interested in the application of halodemetallation as an additional methodology offering some advantages in the synthesis of iodoazines. This paper deals with a general method for the synthesis of trimethylstannylazines and iodoazines through the iododestannation of the trimethylstannylazines.

Synthesis of Trimethylstannyl- and Bis(trimethylstannyl)-azines

To our knowledge there is only one example of synthesis of organostannyl substituted *N*-heteroaromatics in the literature: Anderson and Webster[7] have synthesized 2-trimethylstannylpyridine (**3a**) in 12% yield from 2-chloropyridine (**1a**) and chlorotrimethylstannane in the presence of magnesium. In our study, this method was modified in order to prepare trimethylstannyl substituted pyridines, quinolines, and isoquinolines. Employment of trimethylstannyl sodium instead of the system of chlorotrimethylstannane and magnesium in the stannation reaction resulted in an effective procedure for the formation of 2-trimethylstannylpyridine (**3a**). Thus, treatment of 2-chloropyridine (**1a**) in 1,2-dimethoxyethane (DME) with trimethylstannyl sodium (**2**) prepared *in situ* from chlorotrimethylstannane and

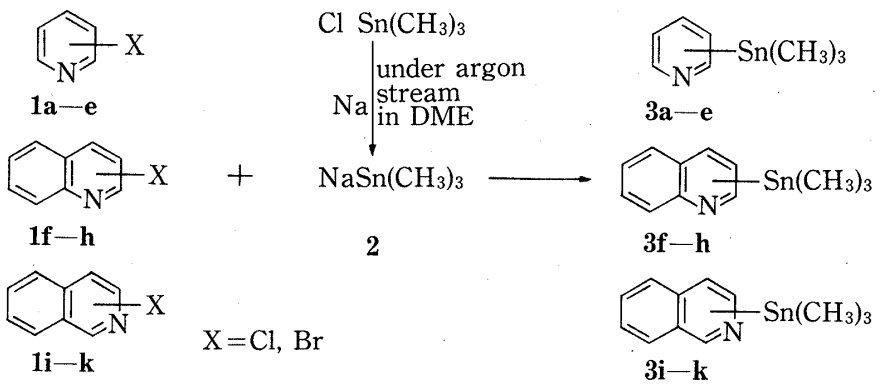
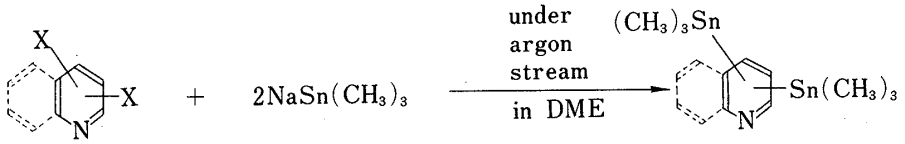


Chart 1



1l—r X=Cl, Br **2** **3l—r**

Chart 2

TABLE I. Preparation of Trimethylstannylazines (3a—k)[a]

Starting material	No.	Product	No.	bp, °C (Torr)	Yield (%)
2-Chloro-Py.	**1a**	2-TMSn-Py.	**3a**	81—83 [b] (10.0)	88
3-Bromo-Py.	**1b**	3-TMSn-Py.	**3b**	116—118 (16.0)	87
4-Chloro-Py.	**1c**	4-TMSn-Py.	**3c**	88—90 (14.0)	60
4-Chloro-2-methyl-Py.	**1d**	2-Methyl-4-TMSn-Py.	**3d**	104—106 (10.0)	61
4-Chloro-2,6-dimethyl-Py.	**1e**	2,6-Dimethyl-4-TMSn-Py.	**3e**	109—112 (11.0)	67
2-Chloro-Q.	**1f**	2-TMSn-Q.	**3f**	124—126 (1.0)	80
3-Bromo-Q.	**1g**	3-TMSn-Q.	**3g**	124—126 [c] (1.0)	81
4-Bromo-Q.	**1h**	4-TMSn-Q.	**3h**	128—131 (1.0)	74
1-Chloro-I.Q.	**1i**	1-TMSn-I.Q.	**3i**	120—121 (4.5)	77
3-Bromo-I.Q.	**1j**	3-TMSn-I.Q.	**3j**	118—120 (4.0)	70
4-Bromo-I.Q.	**1k**	4-TMSn-I.Q.	**3k**	147—149 (3.0)	78

a) The following abbreviations are used: Py.=pyridine; Q.=quinoline; I.Q.=isoquinoline; and TMSn=trimethylstannyl.
b) Ref. 7, 75°C (4 Torr). *c*) **3g** solidified after distillation; mp 42—44°C.

TABLE II. Preparation of Bis(trimethylstannyl)azines (3l—r)[a]

Starting material	No.	Product	No.	bp, °C (Torr) or mp, °C	Yield (%)
2,3-Dibromo-Py.	**1l**	2,3-Bis(TMSn)-Py.	**3l**	125—127 (3.0)	70
2,4-Dichloro-Py.	**1m**	2,4-Bis(TMSn)-Py.	**3m**	115—116 (4.5)	71
2,5-Dibromo-Py.	**1n**	2,5-Bis(TMSn)-Py.	**3n**	115—116	88
2,6-Dichloro-Py.	**1o**	2,6-Bis(TMSn)-Py.	**3o**	132—134 (10.0)	83
3-Bromo-4-chloro-Py.	**1p**	3,4-Bis(TMSn)-Py.	**3p**	116—118 (3.5)	78
3,5-Dichloro-Py.	**1q**	3,5-Bis(TMSn)-Py.	**3q**	118—120 (3.5)	86
4-Bromo-2-chloro-Q.	**1r**	2,4-Bis(TMSn)-Q.	**3r**	145—147 (0.5)	65

a) The following abbreviations are used: Py.=pyridine; Q.=quinoline, TMSn=trimethylstannyl.

metallic sodium under an argon stream readily afforded **3a** in a high yield. The product **3a** was identified by comparison of its infrared (IR) spectrum with that of an authentic sample prepared by Anderson's method.

By this procedure, trimethylstannyl derivatives (**3b—k**) of pyridine, quinoline, and isoquinoline were successfully synthesized from the corresponding chloro or bromo derivatives (**1b—k**). IR spectra of these stannyl derivatives (**3a—k**) showed the characteristic Sn–C stretching in the region of 750—790 cm^{-1}. In the nuclear magnetic resonance (NMR) spectra (CCl_4), distinctive singlet signals due to nine protons of the trimethylstannyl group were observed at δ 0.34—0.48. Table I summarizes the results obtained.

In addition, bis(trimethylstannyl) derivatives (**3l—r**) of pyridine and quinoline were similarly prepared from the corresponding dihalo (chloro or bromo) derivatives (**1l—r**) using two equivalent amounts of **2** (Table II).

Synthesis of Iodoazines and Diiodoazines

Iododestannation of **3a—k** was found to proceed smoothly leading to iodoazines (**4**) in high to excellent yields: for example, treatment of **3f** in chloroform ($CHCl_3$) with an equimolar amount of iodine at room temperature for 15 min produced 2-iodoquinoline (**4f**) in a quantitative yield. In a similar manner, iodo derivatives (**4a—k**) of pyridine, quinoline, an disoquinoline were simply synthesized. The results obtained are summarized in Table III.

In order to make the procedure simpler, we developed a one-pot synthesis: 2-chloroquinoline (**1f**) was treated with **2** in DME for 3 h in an ice-salt bath. To the resulting

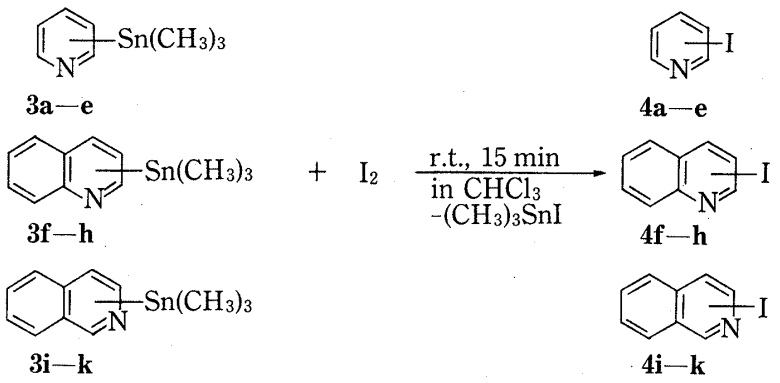


Chart 3

TABLE III. Preparation of Iodoazines (**4a—k**)[a]

Starting material	Product	No.	bp, °C (Torr)	mp, °C	Yield,[b] %
3a	2-Iodo-Py.	**4a**	100—102(25.0)[c]		75 (69)
3b	3-Iodo-Py.	**4b**	90—92(14.0)	48—50[d]	78 (71)
3c	4-Iodo-Py.	**4c**		101(subl.)[e]	76
3d	4-Iodo-2-methyl-Py.	**4d**	102—103(25.0)	42—44[f]	79.5(54)
3e	4-Iodo-2,6-dimethyl-Py.	**4e**		101—103[g]	78.5(60)
3f	2-Iodo-Q.	**4f**	98—100(0.35)	53—55[h]	97 (70)
3g	3-Iodo-Q.	**4g**	105—107(0.25)	60—62[i]	96 (77)
3h	4-Iodo-Q.	**4h**	108—111(0.27)	98—100[j]	91 (64)
3i	1-Iodo-I.Q.	**4i**		74—76[k]	96
3j	3-Iodo-I.Q.	**4j**		59.5—60.5	94
3k	4-Iodo-I.Q.	**4k**		93—94[l]	95

a) The following abbreviations are used: Py.=pyridine; Q.=quinoline; I.Q.=isoquinoline.
b) The yields of the one-pot procedure are shown in the parentheses.
c) Ref. 3*a*, 93°C (13 Torr). *d*) Ref. 3*b*, 50°C. *e*) Ref. 3*c*, 100°C (dec.). *f*) Ref. 3*e*, 43—45°C. *g*) Ref. 3*e*, 101—102°C.
h) Ref. 5*b*, 54—55°C. *i*) Ref. 3*d*, 61—62°C. *j*) Ref. 5*c*, 97—99°C. *k*) Ref. 5*a*, 70—72°C. *l*) Ref. 8, 99°C.

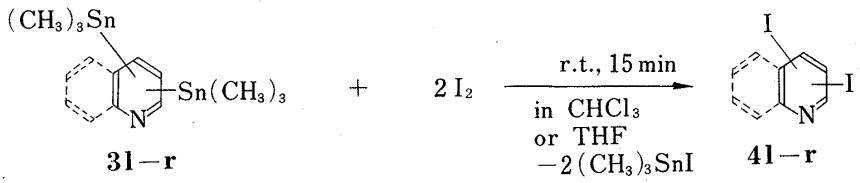


Chart 4

TABLE IV. Preparation of Diiodoazines (**4l—r**)[a]

Starting material	Product	No.	Reaction conditions: Time	Reaction conditions: Solvent	mp, °C	Yield,[b] %
3l	2,3-Diiodo-Py.	**4l**	1 h	THF	63—65	86
3m	2,4-Diiodo-Py.	**4m**	2 h	THF	78—80[c]	90
3n	2,5-Diiodo-Py.	**4n**	30 min	$CHCl_3$	154—156	93
3o	2,6-Diiodo-Py.	**4o**	30 min	$CHCl_3$	196—197[d]	80(68)
3p	3,4-Diiodo-Py.	**4p**	2 h	THF	106 (dec.)	92
3q	3,5-Diiodo-Py.	**4q**	30 min	$CHCl_3$	170—172	91
3r	2,4-Diiodo-Q.	**4r**	30 min	THF	140—142[e]	80(52)

a) The following abbreviations are used: Py.=pyridine; Q.=quinoline.
b) The yields by the one-pot procedure are shown in parentheses.
c) Ref. 9, 74°C.
d) Ref. 10, mp was not given.
e) Ref. 5*d*, 131—132°C.

mixture, an equimolar amount of iodine was added at room temperature to give **4f** in a high yield. The yields obtained in this fashion are also shown in Tables III and IV.

Bis(trimethylstannyl) derivatives **3l—r** likewise underwent iododestannation to afford the corresponding diiodo derivatives (**4l—r**) in good yields (Table IV). In these reactions, the use of tetrahydrofuran (THF) as a solvent gave better results than that of $CHCl_3$.

This stannation–iodination method appears to be very attractive in the following respects in comparison with the methods presently used: a) the yields are in most cases high, b) the procedure is simple, c) the reaction conditions are mild, d) iodination at any position on the pyridine nucleus is possible.

Experimental

2-Trimethylstannylpyridine (**3a**)——A typical preparation of trimethylstannylazines (Table I) was carried out as follows. A solution of chlorotrimethylstannane (62.3 g, 313 mmol) in 50 ml of DME was added to a stirred suspension of small cubes (*ca.* 2 mm cube) of metallic sodium (21.6 g, 940 mg atom) in 200 ml of DME under an argon stream in an ice bath over a 20 min period. When the addition was complete, the mixture was stirred and chilled in an ice-salt bath for 2 h. The color changed to green. The unreacted sodium was removed by filtration through a fritted-glass filter funnel with large porosity. A solution of 2-chloropyridine (**1a**, 28.4 g, 250 mmol) in 150 ml of DME was added dropwise to the filtrate in an ice-salt bath. The resulting solution was stirred for 3 h at the same temperature and then allowed to warm to room temperature. After removal of the solvent *in vacuo* at ambient temperature, the residue was extracted with ether. Concentration of the ether layer followed by distillation gave 2-trimethylstannylpyridine (**3a**), bp 81—83°C (10 Torr). Yield: 53.3 g (88%). Table V summarizes the spectral and analytical data for the trimethylstannylazines (**3a—k**).

Bis(trimethylstannyl)azines (**3l—r**)——Bis(trimethylstannyl)azines (**3l—r**) were prepared from the corresponding dihaloazines (**1l—r**) indicated in Table II, chlorotrimethylstannane, and metallic sodium in the molar ratio of 1: 2.6: 7.8 by the same procedure as given above for **3a—k**. Table VI summarizes the spectral and analytical data for **3l—r**.

General Procedure for Preparation of Iodoazines (**4a—k**)——Iodine (5 mmol) was added in small portions to a stirred solution of **3** (5 mmol) in $CHCl_3$ (30 ml). The reaction mixture was stirred for 15 min at room temperature, and then saturated potassium fluoride solution (20 ml) was added. The resulting mixture was made alkaline with potassium carbonate and extracted with $CHCl_3$. The $CHCl_3$ layer was washed succes-

TABLE V. IR, NMR, MS, and Analysis Data for Trimethylstannylazines (**3a—k**)

Compd. No.	IR ν_{max}^{neat} cm^{-1}	NMR (CCl_4) (J=Hz)	MS (m/e)	Formula	Analysis (%) Calcd (Found) C	H	N
3a	1570 (C=N) 770 [–Sn(CH_3)$_3$]	0.30 (9H, s), 6.8—7.6 (3H, m), 8.5—8.6 (1H,m).	243(M^++1) 242(M^+)	$C_8H_{13}NSn$	39.72 (39.51	5.42 5.32	5.79 5.57)
3b	1560 (C=N) 770 [–Sn(CH_3)$_3$]	0.33 (9H, s), 6.8—7.2 (1H, m), 7.5—7.7 (1H, m), 8.2—8.5 (2H, m).	243(M^++1) 242(M^+)	$C_8H_{13}NSn$	39.72 (39.44	5.42 5.38	5.79 5.75)
3c	1575 (C=N) 770 [–Sn(CH_3)$_3$]	0.30 (9H, s), 7.11 (2H, d, J=5), 8.25 (2H, d, J=5).	243(M^++1) 242(M^+)	$C_8H_{13}NSn$	39.72 (39.50	5.42 5.60	5.79 5.73)
3d	1575 (C=N) 770 [–Sn(CH_3)$_3$]	0.30 (9H, s), 2.43 (3H, s), 6.9—7.2 (2H, m), 8.26 (1H, d, J=4).	257(M^++1) 256(M^+)	$C_9H_{15}NSn$	42.24 (42.39	5.91 5.85	5.47 5.49)
3e	1575 (C=N) 770 [–Sn(CH_3)$_3$]	0.28 (9H, s), 2.37 (6H,s), 6.81 (2H, s).	271(M^++1) 270(M^+)	$C_{10}H_{17}NSn$	44.50 (44.42	6.35 6.29	5.19 5.09)
3f	1580 (C=N) 770, 750 [–Sn(CH_3)$_3$]	0.37 (9H, s), 7.2—8.2 (6H, m).	292(M^+)	$C_{12}H_{15}NSn$	49.37 (49.13	5.18 5.20	4.80 4.74)
3g	1565 (C=N) 790—750 [–Sn(CH_3)$_3$]	0.35 (9H, s), 7.2—8.1 (5H, m), 8.82 (1H, d, J=2).	293(M^++1) 292(M^+)	$C_{12}H_{15}NSn$	49.37 (49.36	5.18 5.11	4.80 4.80)
3h	1560 (C=N) 760 [–Sn(CH_3)$_3$]	0.43 (9H, s), 7.2—8.1 (5H, m), 8.68 (1H, d, J=4).	293(M^++1) 292(M^+)	$C_{12}H_{15}NSn$	49.37 (49.32	5.18 5.14	4.80 4.64)
3i	1620 (C=N) 770 [–Sn(CH_3)$_3$]	0.48 (9H, s), 7.3—8.0 (5H, m), 8.54 (1H, d, J=6).	293(M^++1) 292(M^+)	$C_{12}H_{15}NSn$	49.37 (49.19	5.18 5.15	4.80 4.67)
3j	1625 (C=N) 750 [–Sn(CH_3)$_3$]	0.40 (9H, s), 7.2—8.0 (5H, m), 9.26 (1H, s).	293(M^++1) 292(M^+)	$C_{12}H_{15}NSn$	49.37 (49.15	5.18 5.17	4.80 4.86)
3k	1620 (C=N) 770, 750 [–Sn(CH_3)$_3$]	0.45 (9H, s), 7.3—8.0 (4H, m), 8.46 (1H, s), 9.09 (1H, s).	293(M^++1) 292(M^+)	$C_{12}H_{15}NSn$	49.37 (49.39	5.18 5.29	4.80 4.89)

TABLE VI. IR, NMR, MS, and Analysis Data for Bis(trimethylstannyl)azines (**3l—r**)

Compd. No.	IR ν_{max}^{neat} cm^{-1}	NMR (CCl_4) (J=Hz)	MS (m/e)	Formula	Analysis (%) Calcd (Found) C	H	N
3l	1550 (C=N) 765 [–Sn(CH_3)$_3$]	0.33 (18H, s), 6.91 (1H, ABX), 7.52 (1H, ABX),8.48 (1H,ABX).	407(M^++2) 406(M^++1)	$C_{11}H_{21}NSn_2$	32.65 (32.41	5.23 5.05	3.46 3.52)
3m	1555 (C=N) 760 [–Sn(CH_3)$_3$]	0.30 (18H, s), 7.08 (1H, d, J=4), 7.31 (1H, s), 8.42 (1H, d, J=4).	407(M^++2) 406(M^++1) 405(M^++1)	$C_{11}H_{21}NSn_2$	32.65 (32.55	5.23 5.25	3.46 3.33)
3n	1550 (C=N)[a] 765 [–Sn(CH_3)$_3$]	0.31 (18H, s), 7.28 (1H, d, J=6.5), 7.50 (1H, dd, J=2, 6.5), 8.63 (1H, s).	407(M^++2) 406(M^++1) 405(M^+)	$C_{11}H_{21}NSn_2$	32.65 (32.48	5.23 5.19	3.46 3.47)
3o	1555 (C=N) 765 [–Sn(CH_3)$_3$]	0.31 (18H, s), 7.0—7.3 (3H, AB_2).	407(M^++2) 406(M^++1) 405(M^+)	$C_{11}H_{21}NSn_2$	32.65 (32.39	5.23 5.00	3.46 3.63)
3p	1550 (C=N) 765 [–Sn(CH_3)$_3$]	0.32 (9H, s), 0.34 (9H, s), 7.21 (1H, d, J=4), 8.23 (1H, d, J=4), 8.36 (1H, s).	407(M^++2) 406(M^++1) 405(M^+)	$C_{11}H_{21}NSn_2$	32.65 (32.79	5.23 5.18	3.46 3.32)
3q	1540 (C=N) 765 [–Sn(CH_3)$_3$]	0.33 (18H, s), 7.66 (1H, A_2B), 8.38 (2H, A_2B).	407(M^++2) 406(M^++1) 405(M^+)	$C_{11}H_{21}NSn_2$	32.65 (32.73	5.23 5.38	3.46 3.65)
3r	1555 (C=N) 760 [–Sn(CH_3)$_3$]	0.41 (9H, s), 0.46 (9H, s), 7.1—8.2 (5H, m).	457(M^++2) 456(M^++1) 455(M^+)	$C_{15}H_{23}NSn_2$	39.62 (39.65	5.10 5.36	3.08 2.88)

a) KBr tablet.

Table VII. IR, NMR, MS and Analysis Data for Iodo-(**4j**) and Diiodoazines (**4l** and **4n—q**)

Compd. No.	IR $\nu_{max}^{CHCl_3}$ cm^{-1}	NMR (CCl_4) (J=Hz)	MS (m/e)	Formula	Analysis (%) Calcd (Found) C	H	N
4j	1560 (C=N)	7.3—8.0 (4H, m), 8.12 (1H, s), 8.96 (1H, s).	256(M^++1) 255(M^+)	C_9H_6IN	42.38 (42.33	2.37 2.26	5.49 5.33)
4l	1495 (C=N)	6.95 (1H, ABX), 7.94 (1H, ABX), 8.25 (1H, ABX).	332(M^++1) 331(M^+)	$C_5H_3I_2N$	18.15 (18.35	0.91 0.80	4.23 4.26)
4n	1540—1505 (C=N)	7.3—7.8 (2H, ABX), 8.5—8.7 (ABX).	332(M^++1) 331(M^+)	$C_5H_3I_2N$	18.15 (18.13	0.91 0.85	4.23 4.14)
4o	1550—1510 (C=N)	6.92 (1H, A_2B), 7.68 (2H, A_2B)	332(M^++1) 331(M^+)	$C_5H_3I_2N$	18.15 (18.27	0.91 0.98	4.23 4.19)
4p	1540 (C=N)	7.79 (1H, d, J=5), 8.11 (1H, d, J=5), 8.85 (1H, s).	332(M^++1) 331(M^+)	$C_5H_3I_2N$	18.15 (18.25	0.91 0.92	4.23 4.28)
4q	1530 (C=N)	8.33 (1H, A_2B), 8.72 (2H, A_2B).	332(M^++1) 331(M^+)	$C_5H_3I_2N$	18.15 (18.36	0.91 0.88	4.23 4.18)

sively with saturated potassium fluoride solution and saturated sodium thiosulfate solution, dried, and concentrated under reduced pressure. The residue was purified by recrystallization or distillation *in vacuo*.

General Procedure for Preparation of Diiodoazines (4l—r)——A stirred solution of bis(trimethylstannyl)-azines (**3l—r**) in $CHCl_3$ or THF was treated with two equivalent amounts of iodine. The reaction mixture was stirred for more than 40 min at room temperature, and then saturated potassium fluoride solution (*ca.* 20 ml) was added (THF was removed *in vacuo* and replaced by $CHCl_3$, when THF was used). The resulting mixture was made alkaline with potassium carbonate and subsequently extracted with $CHCl_3$. The $CHCl_3$ layer was washed successively with saturated potassium fluoride solution and saturated sodium thiosulfate solution, dried, and concentrated under reduced pressure. The residue was purified by recrystallization. Table VII summarizes the spectral and analytical data for the new iodo- and diiodo-azines.

One-Pot Procedure for Preparation of Iodoazines (4a, b, d—h) and Diiodoazines (4o, r)——A solution of a haloazine (**1a, b, d—h**, 10 mmol) or a dihaloazine (**1o, r**, 5 mmol) in DME (10 ml) was added dropwise with stirring under an argon stream to an ice-salt cooled solution of trimethylstannyl sodium in DME, prepared from chlorotrimethylstannane (2.49 g, 12.5 mmol) and metallic sodium (1.15 g, 50 mg atom) in the manner described for **3a—k**. The mixture was stirred for 2 h at the same temperature, then iodine (3.05 g, 12 mmol) was added. The resulting mixture was allowed to warm to room temperature, stirred for an additional 15 min, and then concentrated under reduced pressure. From the residue, **4a, b, d—h** and **4o, r** were taken up and purified by the same procedure as described above.

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