Application of Phosphonium Salts to the Reactions of Various Kinds of Amides

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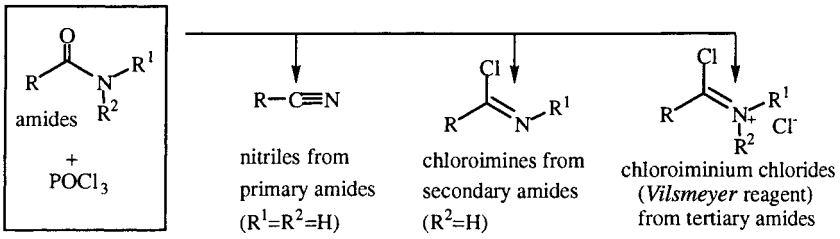
The phosphonium salts **1** and **2** prepared from triphenylphosphine and *N*-halogenosuccinimide proved to be applicable to the conversion of amide compounds. Especially, halogenation of electron-deficient heteroaromatic alcohols with these reagents seems to be a convenient method compared to the halogenation with phosphorus oxyhalides.

Introduction. – Amide compounds are important intermediates in organic chemistry. They can be converted to nitriles, chloroimines, or chloroiminium chlorides with phosphorus oxychloride (= phosphoric trichloride). Phosphorus oxychloride is, however, an irritant and is unwieldy on account of its moisture-sensitive nature. Thus, the development of new reaction modes not dependent on halogenating reagents such as phosphorus oxyhalide would be advantageous for many chemists (*Scheme 1*).

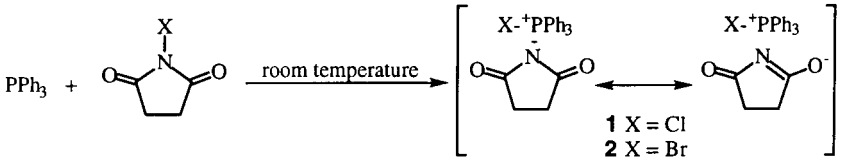
Scheme 1. *Conversion of Amides with Phosphorus Oxychloride* ($POCl_3$)



P-Compounds have a strong affinity for O-atoms and are widely used for the conversion of alcohols or carboxylic acids (halogenation with phosphorus oxyhalide or *Mitsunobu* reaction), or carbonyl compounds (*Wittig* reaction). Recently, conversion of carboxylic acids to acyl halides [1], esters [2], and amides [3] with phosphonium salts **1** or **2**, which were obtained by the reaction of triphenylphosphine with *N*-halogenosuccinimide (*Scheme 2*), was reported by *Froyen*. As the phosphonium salts **1** and **2** were presumed to have a chemical reactivity similar to that of a representative halogenating reagent, phosphorus oxyhalide, their application to the reactions of various kinds of amides was undertaken.

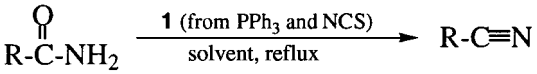
Results and Discussion. – At first, the phosphonium salt **1** was applied to the dehydration of primary amides. A mixture of an amide shown in *Table 1* and **1** was

Scheme 2. *Preparation of Phosphonium Salts* **1** *and* **2**



heated to reflux to give the corresponding nitrile in good yield. Best results were achieved with 2 equiv. of **1** and heating to reflux for 4 h. Both dioxane and tetrahydrofuran (THF) were suitable as a solvent for this reaction.

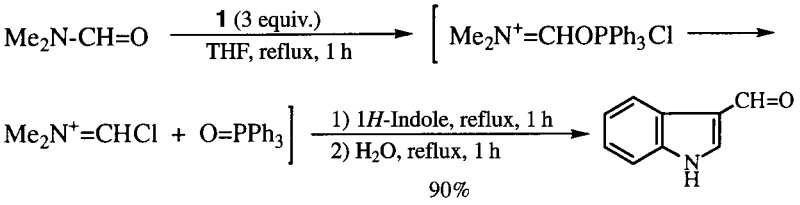
Table 1. *Dehydration of Amides*



R	Solvent	Amount of **1**	Reaction time [h]	Yield [%]
p-$MeOC_6H_4$	dioxane	1 equiv.	1	70
p-$MeOC_6H_4$	dioxane	1 equiv.	4	74
p-$MeOC_6H_4$	dioxane	2 equiv.	4	92
p-$MeOC_6H_4$	THF	2 equiv.	4	93
Ph	dioxane	2 equiv.	4	64
Ph	THF	2 equiv.	4	72
$PhCH_2$	dioxane	2 equiv.	4	83
$PhCH_2$	THF	2 equiv.	4	92

Next, formylation of 1*H*-indole with **1** was carried out. A mixture of *N*,*N*-dimethylformamide and 3 equiv. of **1** in THF was heated to reflux for 1 h, followed by addition of 1*H*-indole to give 1*H*-indole-3-carbaldehyde (*Scheme 3*). This result shows that the reaction of tertiary amides and **1** gives the corresponding iminium salts.

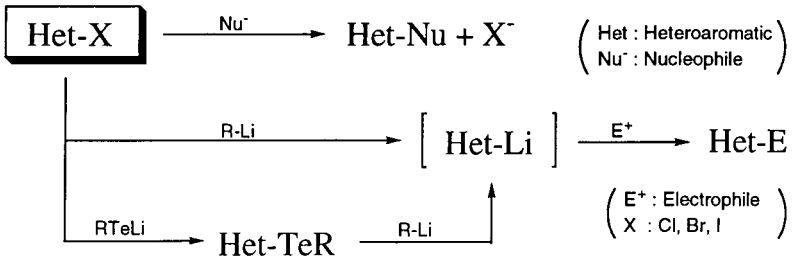
Scheme 3. *Formylation of 1*H*-Indole*



Electron-deficient halogenoheteroaromatics are useful intermediates in organic synthesis. They react with nucleophiles such as amines or alkoxides to give the corresponding substituted products, and are lithiated to introduce electrophiles *via* a halogen-lithium exchange reaction [4] or a tellurium-lithium exchange reaction [5–7] (*Scheme 4*).

While the reactivity of heteroaromatics has been studied in detail, the study of their basic synthetic reactions like halogenation [8] has remained at a standstill. It is well-known that heteroaromatic alcohols are chlorinated with phosphorus oxychloride to

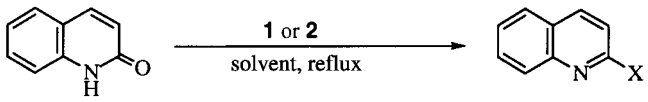
Scheme 4. *Reaction of Electron-Deficient Haloheteroaromatics* (Het-X)



give chloroheteroaromatics [9–12]. Similarly, bromoheteroaromatics can be obtained with phosphorus oxybromide [12–14]. These reagents are, however, irritants and are unwieldy on account of their moisture-sensitive nature. Furthermore, phosphorus oxybromide is too expensive for synthetic applications. We speculated that the phosphonium salts **1** could be applied to chlorination of heteroaromatic alcohols. In a preceding paper [15], the convenient halogenation of some heteroaromatic alcohols with triphenylphosphine and *N*-halogenosuccinimide was reported. We applied this method to a number of other heteroaromatic alcohols and developed a one-pot iodination using a combination of the chlorination and a Cl/I-exchange reaction.

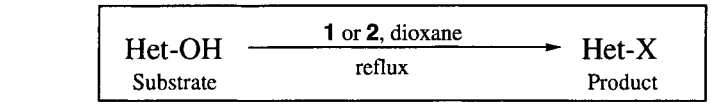
The results of chlorination of quinolin-2(1*H*)-one with **1** and bromination with **2** are reported in *Table 2*. Thus, a mixture of quinolin-2(1*H*)-one and 1 equiv. of **1** in dioxane was heated to reflux to give 2-chloroquinoline in 35–39% yield (*Entries 1–3*). When the amount of **1** was increased, the yield of 2-chloroquinoline was improved (*Entries 4* and *5*). Bromination of quinolin-2(1*H*)-one with 5 equiv. of phosphonium salt **2** resulted in 90% yield of 2-bromoquinoline after 4 h reflux in dioxane (*Entry 9*). In THF (b.p. 66°) as solvent, the yield was lower (*Entries 6* and *10*), this halogenation presumably requiring a certain temperature. Iodination of quinolin-2(1*H*)-one with *N*-iodosuccinimide gave 2-iodoquinoline in low yield. This result may be due to the instability of the iodinating reagent being heated.

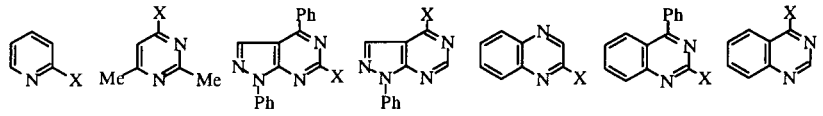
Table 2. *Halogenation of Quinolin-2(1*H*)-one*



Entry	X	Solvent	Amount of **1** or **2**	Reaction time [h]	Yield [%]
1	Cl	dioxane	**1** (1 equiv.)	2	35
2	Cl	dioxane	**1** (1 equiv.)	4	39
3	Cl	dioxane	**1** (1 equiv.)	8	35
4	Cl	dioxane	**1** (2 equiv.)	4	58
5	Cl	dioxane	**1** (5 equiv.)	4	63
6	Cl	THF	**1** (5 equiv.)	6	31
7	Br	dioxane	**2** (1 equiv.)	4	46
8	Br	dioxane	**2** (2 equiv.)	4	71
9	Br	dioxane	**2** (5 equiv.)	4	90
10	Br	THF	**2** (5 equiv.)	11	0

Table 3. *Halogenation of Some Heteroaromatic Alcohols*





A B C D E F G

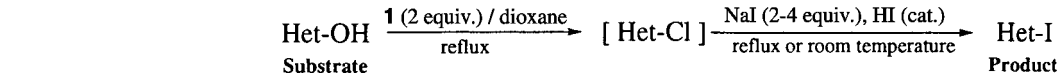
Entry	Substrate	Phosphonium salt **1** or **2**	Reaction time [h]	Product	Yield [%]
1	**A** (X = OH)	**1** (5 equiv.)	4	**A** (X = Cl)	43
2	**A** (X = OH)	**2** (5 equiv.)	4	**A** (X = Br)	54
3	**B** (X = OH)	**1** (2 equiv.)	4	**B** (X = Cl)	74
4	**B** (X = OH)	**2** (5 equiv.)	9.5	**B** (X = Br)	84
5	**C** (X = OH)	**1** (2 equiv.)	4	**C** (X = Cl)	89
6	**C** (X = OH)	**2** (7 equiv.)	4	**C** (X = Br)	71
7	**D** (X = OH)	**1** (5 equiv.)	7	**D** (X = Cl)	56
8	**D** (X = OH)	**2** (5 equiv.)	8.5	**D** (X = Br)	50
9	**E** (X = OH)	**1** (5 equiv.)	4	**E** (X = Cl)	88
10	**E** (X = OH)	**2** (5 equiv.)	4	**E** (X = Br)	43
11	**F** (X = OH)	**1** (5 equiv.)	4	**F** (X = Cl)	82
12	**F** (X = OH)	**2** (5 equiv.)	4	**F** (X = Br)	49
13	**G** (X = OH)	**1** (5 equiv.)	4	**G** (X = Cl)	73
14	**G** (X = OH)	**2** (2 equiv.)	1	**G** (X = Br)	5
15	**G** (X = OH)	**2** (2 equiv.)	4	**G** (X = Br)	1

The halogenation was then applied to some other heteroaromatic alcohols to confirm its generality, and was successful with most of the substrates shown in *Table 3*. Pyridin-2(1*H*)-one (**A**), 2,6-dimethylpyrimidin-4(3*H*)-one (**B**), 1,4-diphenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-6(7*H*)-one (**C**), 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4(5*H*)-one (**D**), quinoxalin-2(1*H*)-one (**E**), and 4-phenylquinazolin-2(1*H*)-one (**F**) were halogenated with **1** or **2** to give the corresponding halide in good to fair yields (*Entries 1–12*). Bromination of quinazolin-4(3*H*)-one (**G**) gave 4-bromoquinazoline in low yield because of the instability of the product (*Entries 14* and *15*).

As the direct iodination of heteroaromatic alcohols with triphenylphosphine and *N*-iodosuccinimide was found to be difficult, the one-pot synthesis of some iodoheteroaromatics from the corresponding heteroaromatic alcohols by chlorination with **1** followed by Cl/I exchange was developed as shown in *Table 4*. It should be noted that the isolation of the intermediary chloro compounds synthesized with **1** is not necessary in this iodination of heteroaromatic alcohols, whereas the isolation of the chloro compounds synthesized with $POCl_3$ is mandatory.

Conclusion. – In this study, the conversion of amides into nitriles, haloimines, or chloroiminium salts was accomplished with the phosphonium salts **1** or **2**. Especially the halogenation of electron-deficient heteroaromatic alcohols with **1** or **2** seems to be a convenient and inexpensive method compared to the halogenation with phosphorus oxyhalides.

Table 4. *Iodination of Some Heteroaromatic Alcohols by the Cl/I Exchange Reaction*



Substrate (Het-OH)	Product (Het-I)	Yield [%]
quinolin-2(1*H*)-one	2-iodoquinoline	53
2,6-dimethylpyrimidin-4(3*H*)-one (**B**)	4-iodo-2,6-dimethylpyrimidine	60
1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4(5*H*)-one (**D**)	4-iodo-1-phenyl-1*H*-pyrazolo[3,4-*d*]-pyrimidine	31
quinoxalin-2(1*H*)-one (**E**)	2-iodoquinoxaline	41

Experimental Part

1. *General*. CC = column chromatography. M.p.: not corrected. ^{1}H-NMR Spectra: *Hitachi-R-90 H* spectrometer; δ in ppm rel. to $SiMe_4$ as an internal standard, J in Hz.

2. *Dehydration of Amides: General Procedure* (*Table 1*): *N*-Chlorosuccinimide (1335 mg, 10.0 mmol) was added little by little to a soln. of PPh_3 (2623 mg, 10.0 mmol) in an appropriate solvent (100 ml) and stirred at r.t. for 30 min. The amide (5–10 mmol) was added to the suspension, and the mixture was heated to reflux. After the mixture was neutralized with Et_3N, the solvent was evaporated and the residue submitted to CC (SiO_2) to give the corresponding nitriles.

4-Methoxybenzonitrile: CC (SiO_2, hexane/AcOEt 5:1). ^{1}H-NMR ($CDCl_3$): 3.85 (*s*, MeO); 6.94 (*d*, J = 9.0, 2 arom. H); 7.59 (*d*, J = 9.0, 2 arom. H).

Benzonitrile: CC (SiO_2, hexane/AcOEt 10:1). ^{1}H-NMR ($CDCl_3$): 7.40–7.75 (*m*, 5 arom. H).

Benzeneacetonitrile: CC (SiO_2, hexane/AcOEt 5:1). ^{1}H-NMR ($CDCl_3$) 3.73 (*s*, CH_2); 7.34 (*s*, 5 arom. H).

3. *Formylation of 1*H*-Indole* (*Scheme 3*): *N*-Chlorosuccinimide (2003 mg, 15.0 mmol) was added little by little to a soln. of PPh_3 (3934 mg, 15.0 mmol) in THF (150 ml) and stirred at r.t. for 30 min. *N*,*N*-Dimethylformamide (2193 mg, 30.0 mmol) was added to the suspension, and the mixture was heated to reflux for 1 h. Then 1*H*-indole (586 mg, 5.00 mmol) was added, and the mixture was heated to reflux for 1 h. The THF was evaporated, H_2O (50 ml) added, and the mixture heated to reflux for 1 h and then alkalinized with NaOH soln. After extraction with AcOEt (200 ml), the residue of the org. phase was submitted to CC (SiO_2, hexane/AcOEt 3:2): 653 mg (90%) of *1*H*-indole-3-carboxaldehyde* [16]. White solid. M.p. 196–197° ([16]: 194–196°). ^{1}H-NMR ($CDCl_3$): 7.15–7.66 (*m*, H–C(5), H–C(6), H–C(7)); 7.83 (*d*, J = 3.1, H–C(2)); 8.17–8.46 (*m*, H–C(4)); 8.55–9.45 (br., NH); 10.07 (*s*, CH=O).

4. *Halogenation of Heteroaromatic Alcohols: General Procedure* (*Tables 2* and *3*): *N*-Halogenosuccinimide was added little by little to a soln. of PPh_3 in an appropriate solvent and stirred at r.t. for 30 min. The heteroaromatic alcohol was added to the suspension, and the mixture was heated to reflux. After the mixture was alkalinized with Et_3N, dioxane and Et_3N were evaporated, and the residue was submitted to CC (SiO_2) to give the corresponding halogenoheteroaromatics.

2-Chloroquinoline [17]: CC (SiO_2, hexane/AcOEt 6:1). White solids. M.p. 31–32° ([17]: 37–38°). ^{1}H-NMR ($CDCl_3$): 7.26–8.18 (*m*, 6 arom. H).

2-Bromoquinoline [18]: CC (SiO_2, hexane/CH_2Cl_2 1:1). White solid. M.p. 49° ([18]: 48–49°). ^{1}H-NMR ($CDCl_3$): 7.40–8.13 (*m*, 6 arom. H).

2-Chloropyridine [19]: CC (SiO_2, hexane/CH_2Cl_2 2:1). Colorless oil. ^{1}H-NMR ($CDCl_3$): 7.06–7.44 (*m*, H–C(3), H–C(5)); 7.65 (*td*, J = 7.5, 2.0, H–C(4)); 8.40 (*d*, J = 5.0, H–C(6)).

2-Bromopyridine [20]: CC (SiO_2, hexane/CH_2Cl_2 3:2). Colorless oil. ^{1}H-NMR ($CDCl_3$): 7.02–7.36 (*m*, H–C(5)); 7.36–7.71 (*m*, H–C(3), H–C(4)); 8.20–8.50 (*m*, H–C(6)).

4-Chloro-2,6-dimethylpyrimidine [21]: CC (SiO_2, with hexane/AcOEt 10:1 → 3:1). Yellow oil. ^{1}H-NMR ($CDCl_3$): 2.48 (*s*, 1 Me); 2.67 (*s*, 1 Me); 7.04 (*s*, 1 arom. H).

4-Bromo-2,6-dimethylpyrimidine: CC (SiO_2, hexane/AcOEt 10:1 → 2:1). White needles. M.p. 54°. ^{1}H-NMR ($CDCl_3$): 2.46 (*s*, 1 Me); 2.67 (*s*, 1 Me); 7.21 (*s*, 1 arom. H). Anal. calc. for $C_6H_7BrN_2$: C 38.53, H 3.77, N 14.98; found: C 38.50, H 3.76, N 14.73.

*6-Chloro-1,4-diphenyl-1*H-*pyrazolo[3,4-*d*]pyrimidine* [22]: CC (SiO_2, hexane/CH_2Cl_2 1:1). Pale yellow needles (recryst. from hexane/AcOEt). M.p. 184–186°. ^{1}H-NMR ($CDCl_3$): 7.25–7.72 (*m*, 6 H, Ph); 8.04–8.32 (*m*, 4 H, Ph); 8.50 (*s*, H–C(3)).

*6-Bromo-1,4-diphenyl-1*H-*pyrazolo[3,4-*d*]pyrimidine:* CC (SiO_2, hexane/CH_2Cl_2 1:1). Colorless needles (recryst. from hexane/AcOEt). M.p. 194°. ^{1}H-NMR ($CDCl_3$): 7.28–7.82 (*m*, 6 H, Ph); 8.06–8.40 (*m*, 4 H, Ph); 8.51 (*s*, H–C(3)). Anal. calc. for $C_{17}H_{11}BrN_4$: C 58.14, H 3.16, N 15.95; found: C 58.30, H 3.14, N 15.83.

*4-Chloro-1-phenyl-1*H-*pyrazolo[3,4-*d*]pyrimidine* [10]: CC (SiO_2, hexane/AcOEt 10:1). White solid. M.p. 131–132° ([10]: 126–127°). ^{1}H-NMR ($CDCl_3$): 7.32–7.70 (*m*, 3 H, Ph); 8.07–8.29 (*m*, 2 H, Ph); 8.34 (*s*, H–C(3)); 8.87 (*s*, H–C(6)).

*4-Bromo-1-phenyl-1*H-*pyrazolo[3,4-*d*]pyrimidine* [23]: CC (SiO_2, hexane/CH_2Cl_2 1:1). White solid. M.p. 137–139° ([23]: 135°). ^{1}H-NMR ($CDCl_3$): 7.32–7.69 (*m*, 3 H, Ph); 8.06–8.30 (*m*, 2 H, Ph); 8.27 (*s*, H–C(3)); 8.81 (*s*, H–C(6)).

2-Chloroquinoxaline [9]: CC (SiO_2, hexane/AcOEt 6:1). Pale yellow solid. M.p. 47–47.5° ([9]: 46–47°). ^{1}H-NMR ($CDCl_3$): 7.67–7.93 (*m*, 2 arom. H); 7.93–8.23 (*m*, 2 arom. H); 8.78 (*s*, H–C(3)).

2-Bromoquinoxaline [14]: CC (SiO_2, hexane/CH_2Cl_2 3:2). Pale yellow solid. M.p. 58° ([14]: 58–59°). ^{1}H-NMR ($CDCl_3$): 7.62–7.90 (*m*, 2 arom. H); 7.90–8.23 (*m*, 2 arom. H); 8.86 (*s*, H–C(3)).

2-Chloro-4-phenylquinazoline [24]: CC (SiO_2, hexane/CH_2Cl_2 1:1). Pale yellow solid. M.p. 111–112° ([24]: 113°). ^{1}H-NMR ($CDCl_3$): 7.45–8.28 (*m*, 9 arom. H).

2-Bromo-4-phenylquinazoline: CC (SiO_2, hexane/CH_2Cl_2 1:1). Colorless needles (recryst. from hexane/AcOEt). M.p. 130–131°. ^{1}H-NMR ($CDCl_3$): 7.44–8.23 (*m*, 9 arom. H). Anal. calc. for $C_{14}H_9BrN_2$: C 58.97, H 3.18, N 9.82; found: C 58.79, H 3.13, N 9.80.

4-Chloroquinazoline [24]: CC (SiO_2, hexane/AcOEt 8:1). White solid. M.p. 97–98° ([24]: 96.5–97.5°). ^{1}H-NMR ($CDCl_3$): 7.78 (*qd*, J = 8.1, 7.9, H–C(6)); 7.91–8.17 (*m*, H–C(7), H–C(8)); 8.28 (*dd*, J = 8.1, 0.8, H–C(5)).

4-Bromoquinazoline: CC (SiO_2, hexane/AcOEt 2:1). Pale orange prisms (recryst. from hexane). M.p. 95–97° (dec.). ^{1}H-NMR ($CDCl_3$): 7.60–8.33 (*m*, H–C(5), H–C(6), H–C(7), H–C(8)); 8.98 (*s*, H–C(2)). Anal. calc. for $C_8H_5BrN_2$: C 45.96, H 2.41, N 13.40; found: C 46.33, H 2.62, N 13.73.

5. *Iodination of Heteroaromatic Alcohols by Cl/I-Exchange: General Procedure. N*-Chlorosuccinimide was added to a soln. of PPh_3 in dioxane and stirred at r.t. for 30 min. Starting material was added to the suspension, and the mixture was stirred under the *Conditions 1* (see below). NaI and a cat. amount of hydroiodic acid were added to the mixture, and the mixture was stirred under the *Conditions 2* (see below). Dioxane was evaporated and the residue dissolved in CH_2Cl_2 and alkalinized with Et_3N. The crude product was purified with by CC (SiO_2).

2-Iodoquinoline [25]: According to the *General Procedure* (*Conditions 1:* reflux, 6 h; *Conditions 2:* reflux, 10 h), from quinolin-2(1*H*)-one (726 mg, 5.00 mmol), PPh_3 (2623 mg, 10.0 mmol), *N*-chlorosuccinimide (1335 mg, 10.0 mmol), and NaI (1500 mg, 10.0 mmol): 670 mg (53%) of yellow solid. M.p. 44–45° ([25]: 52–53°). ^{1}H-NMR ($CDCl_3$): 7.29–7.90 (*m*, 5 arom. H); 8.05 (*d*, J = 7.6, 1 arom. H).

4-Iodo-2.6-dimethylpyrimidine [26]: According to the *General Procedure* (*Conditions 1:* reflux, 9 h, *Conditions 2*, reflux, 9 h), from 2,6-dimethylpyrimidin-4(3*H*)-one (372 mg, 3.00 mmol), PPh_3 (1574 mg, 6.00 mmol), *N*-chlorosuccinimide (801 mg, 6.00 mmol), and NaI (1800 mg, 12.0 mmol): 421 mg (60%) of white powder. M.p. 115° ([26]: 112°). ^{1}H-NMR ($CDCl_3$): 2.40 (*s*, Me–C(2)); 2.65 (*s*, Me–C(6)); 7.48 (*s*, H–C(5)).

*4-Iodo-1-phenyl-1*H-*pyrazolo[3,4-*d*]pyrimidine:* According to the *General Procedure* (*Conditions 1:* reflux, 8 h, *Conditions 2:* r.t. 14 h), from 1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4(5*H*)-one (637 mg, 3.00 mmol), PPh_3 (1574 mg, 6.00 mmol), *N*-chlorosuccinimide (801 mg, 6.00 mmol), and NaI (900 mg, 6.00 mmol): 300 mg (31%) of white powder (recryst. from hexane/AcOEt). M.p. 165°. ^{1}H-NMR ($CDCl_3$): 7.24–7.74 (*m*, 3 H, Ph); 7.99–8.34 (*m*, 2 H, Ph); 8.11 (*s*, H–C(3)); 8.72 (*s*, H–C(6)). Anal. calc. for $C_{11}H_7IN_4$: C 41.02, H 2.19, N 17.39; found: C 41.07, H 2.18, N 17.21.

2-Iodoquinoxaline [10]: According to the *General Procedure* (*Conditions 1:* reflux, 10 h; *Conditions 2:* reflux, 6 h), from quinoxalin-2(1*H*)-one (731 mg, 5.00 mmol), PPh_3 (2623 mg, 10.0 mmol), *N*-chlorosuccinimide (1335 mg, 10.0 mmol), and NaI (3000 mg, 20.0 mmol): pale yellow powder. M.p. 104–105° ([10]: 104–105°). ^{1}H-NMR ($CDCl_3$): 7.55–7.93 (*m*, 2 arom. H); 7.93–8.36 (*m*, 2 arom. H); 8.99 (*s*, H–C(3)).

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