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# New Methods and Reagents in Organic Synthesis. 60.[1)] A New Synthesis of Aromatic and Heteroaromatic Amines Using Diphenyl Phosphorazidate (DPPA)[2)]

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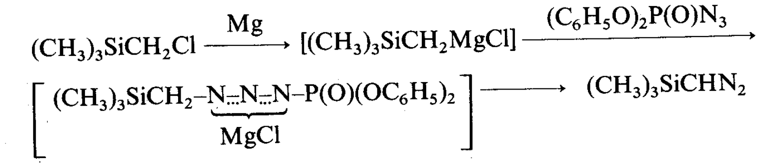
Aromatic and heteroaromatic organometallics (Grignard and lithium compounds) react with diphenyl phosphorazidate (DPPA) to give labile phosphinyltriazenes. A study of the conversion of phosphinyltriazenes into amines has revealed that reductive work-up with aluminum hydride gives much better results than acidic or alkaline work-up. Sequential treatment of aromatic and heteroaromatic organometallics with DPPA, followed by aluminum hydride provides a convenient new method for the preparation of aromatic and heteroaromatic amines.

**Keywords**——organometallic; aromatic halide; Grignard compound; organolithium compound; aromatic amine; heteroaromatic amine; phosphinyltriazene; hydride reduction; diphenyl phosphorazidate

We have already demonstrated[3)] that the Grignard reagent from chloromethyltrimethylsilane readily undergoes the diazo transfer reaction with diphenyl phosphorazidate (DPPA, $(C_6H_5O)_2P(O)N_3$) to give trimethylsilyldiazomethane[4)] in good yield *via* the triazene intermediate,[5)] as shown in Chart 1.



$$(CH_3)_3SiCH_2Cl \xrightarrow{Mg} [(CH_3)_3SiCH_2MgCl] \xrightarrow{(C_6H_5O)_2P(O)N_3} \left[ (CH_3)_3SiCH_2\text{-}\underbrace{N{\cdots}N{\cdots}N}_{MgCl}\text{-}P(O)(OC_6H_5)_2 \right] \longrightarrow (CH_3)_3SiCHN_2$$

Chart 1

We now wish to report a simple and convenient preparation of aromatic and heteroaromatic amines by using methodology similar to the above. We have found that aromatic and heteroaromatic amines **1** can be easily prepared in a one-pot process by treatment of the corresponding Grignard or lithium derivatives **2** with DPPA followed by reductive (or acidic) work-up of the resulting triazene derivatives **3**.[5)] The overall process is depicted in Chart 2.[6,7)]

First, the reaction conditions for the formation of the triazene derivative **4** were investigated by the use of phenylmagnesium bromide. As shown in Table I, diethyl ether seems to be a better solvent than tetrahydrofuran. The triazene **4**[8)] was obtained in excellent yield by the reaction at −70 °C for 2 h, followed by warming to −20 °C during 40 min (run 6 in Table I).

Decomposition of the triazene **4** was next investigated under alkaline and acidic conditions. While alkaline conditions gave aniline in modest yield, acidic conditions gave much better results, as shown in Table II. Treatment of the triazene **4** with methanolic hydrogen chloride at room temperature for 3 h and 20 min afforded aniline hydrochloride in 79% yield.

By using the most favorable reaction conditions for the triazene formation (run 6 in

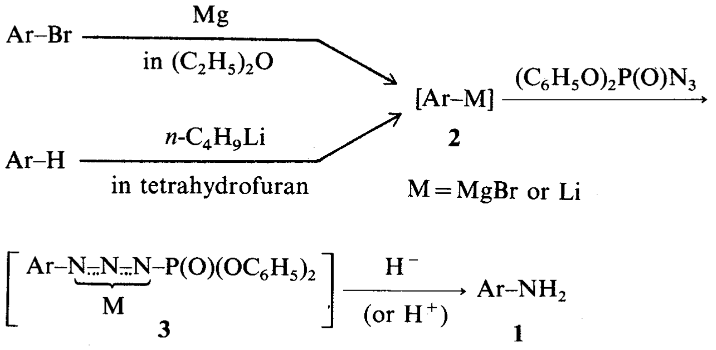
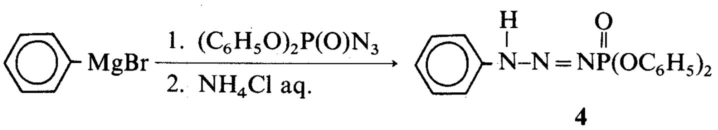


Chart 2

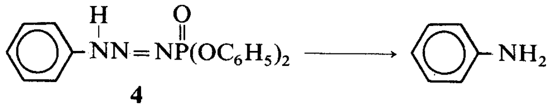
TABLE I. Reaction of Phenylmagnesium Bromide and DPPA



| Run | Reaction conditions | | | Yield,[a] % |
|---|---|---|---|---|
| | Solvent | Temp. (°C) | Time | |
| 1 | $\mathrm{Et_2O}$ | −16—−10 | 2 h | 63 |
| 2 | $\mathrm{Et_2O}$ | −78—−68 | 2 h 20 min | 64 |
| 3 | $\mathrm{Et_2O}$ | −78—−70 | 8 h | 65 |
| 4 | THF | −73—−69 | 2 h 10 min | 53 |
| 5 | $\mathrm{Et_2O}$ | −74 | 2 h | 77 (76)[b] |
| | | −74→−20 | 10 min | |
| 6 | $\mathrm{Et_2O}$ | −73—−69 | 2 h | 91 |
| | | −70→−20 | 40 min | |
| 7[c] | $\mathrm{Et_2O}$ | −71—−68 | 2 h | 37 |

*a*) Yield from DPPA determined with the TLC scanner. *b*) Isolated yield. *c*) Reversed order of addition.

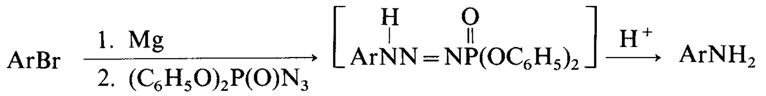
TABLE II. Decomposition of 1-Phenyl-3-diphenoxyphosphinyltriazene (4)



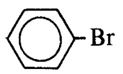
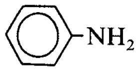
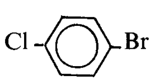
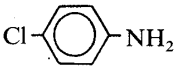
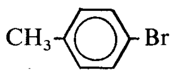
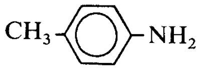
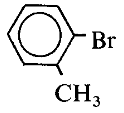
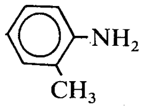
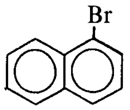
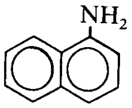
| Run | Reaction conditions | | | | Yield, % |
|---|---|---|---|---|---|
| | Reagent | Solvent | Temp. (°C) | Time | |
| 1 | 30% KOH–MeOH | — | r.t. | 4 d | 49 |
| 2 | 30% KOH–MeOH | THF | r.t. | 4 d | 54 |
| 3 | Silica gel 60 | Benzene | r.t. | 4 d | 0 |
| 4 | 5% HCl–MeOH | — | r.t. | 2 h 30 min | 47 |
| 5 | 5% HCl–MeOH | MeOH | r.t. | 15 h | 71[a] |
| 6 | 10% HCl–MeOH | MeOH | r.t. | 3 h 20 min | 79[a] |
| 7 | 10% HCl–MeOH | MeOH | 35 | 40 min | 73[a] |

r.t.; room temperature. *a*) Isolated as the hydrochloride.

Table III. Conversion of Aromatic Bromides to Aromatic Amines

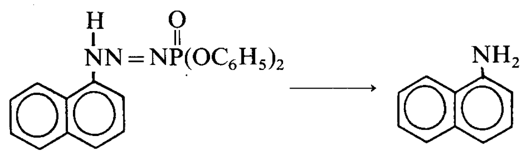


$$\mathrm{ArBr}\xrightarrow[2.\ (C_6H_5O)_2P(O)N_3]{1.\ Mg}\left[\mathrm{Ar}\overset{H}{\underset{|}{\mathrm{N}}}\mathrm{N{=}N}\overset{O}{\underset{\|}{\mathrm{P}}}(OC_6H_5)_2\right]\xrightarrow{H^+}\mathrm{ArNH_2}$$

| Run | ArBr | Product | Yield,[a] % |
|---|---|---|---|
| 1 |  |  | 51 |
| 2 |  |  | 63 |
| 3 |  |  | 33 |
| 4 |  |  | 33 |
| 5 |  |  | 28[b] |

*a*) Isolated as the hydrochloride; yield based on DPPA. *b*) Isolated as the free amine.

Table IV. Decomposition of 1-(1-Naphtyl)-3-diphenoxyphosphinyltriazene (**5**)



**5**

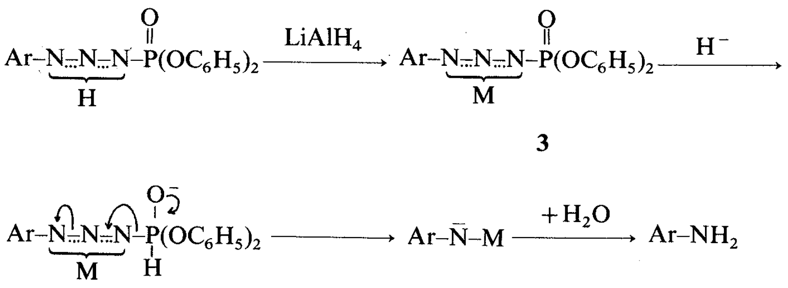
| Run | Reaction conditions | | | | Yield, % |
|---|---|---|---|---|---|
| | Reagent | Solvent | Temp. (°C) | Time | |
| 1 | $SiO_2$[a] | Benzene | r.t. | 24 h | 0 |
| 2 | $Al_2O_3$[b] | Benzene | r.t. | 31 h | 0 |
| 3 | 30% KOH–MeOH | THF | r.t. | 22 h | 56 |
| 4 | 10% HCl–MeOH | MeOH | 2—3<br>r.t. | 1 h<br>Overnight | 17 |
| 5 | $H_2$/5% Pd–C | EtOH | r.t. | 2 d | 0 |
| 6 | $LiAlH_4$ | $Et_2O$ | r.t. | Overnight | 64 |

*a*) Mallinckrodt silicic acid (100 mesh). *b*) Merck aluminum oxide 90.

Table I) and for the acidic decomposition (run 6 in Table II), some representative aromatic halides were converted to corresponding amines without purification of the triazene intermediates. The yields were mostly modest, as shown in Table III.

Since the conversion of 1-bromonaphthalene to 1-naphthylamine proceeded sluggishly, we further investigated the reaction conditions for the decomposition of the corresponding 1-naphthyltriazene **5**.[8] As shown in Table IV, neither alkaline nor acidic conditions afforded satisfactory results. Reductive decomposition of **5** with lithium aluminum hydride in diethyl ether, however, gave 1-naphthylamine in most satisfactory yield.

The reaction mechanism of this hydride reduction is presumably as shown in Chart 3.



Ar = 1-naphthyl
M = Al<

Chart 3

On the basis of this mechanism, we considered that the magnesium bromide salt of the triazene (**3**, M = MgBr, in Chart 3) would undergo analogous hydride reduction. Furthermore, the isolation of the triazenes was thought to be not advantageous, since phosphinyltriazenes in general are rather labile. Indeed, the Grignard reagent from 1-bromonaphthalene was easily converted in a single vessel to 1-naphthylamine in 82% yield by successive treatment with DPPA and lithium aluminum hydride. Replacement of lithium aluminum hydride with sodium bis(2-methoxyethoxy)aluminum hydride gave 1-naphthylamine in 89% yield.

Application of this one-pot procedure to various aromatic Grignard reagents derived from the corresponding bromides afforded aromatic amines in good yields, as summarized in Table V. 2-Bromodibenzofuran also underwent the amination smoothly. Furthermore, the lithiated compounds, prepared from the arenes and heteroarenes by the ortho lithiation procedure,[9)] afforded the corresponding amines in good to modest yields under analogous conditions using tetrahydrofuran as a reaction solvent.

As shown in Table V, the efficiency of our amination procedure is either superior or comparable to those of the reported methods.[6,7)] Thus, amination of aromatic and heteroaromatic organometallics (Grignard and lithium compounds) by the successive use of DPPA and aluminum hydride appears to be a most promising procedure because of its generality, mildness, rapidity, and high yield.

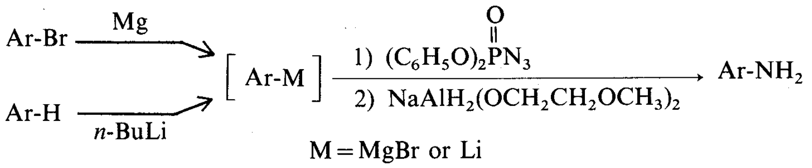
DPPA is already known as a versatile synthetic reagent.[10)] The work described above has confirmed the function of DPPA as a $^+NH_2$ synthon for the organometallics, and has extended the utility of DPPA in organic synthesis.

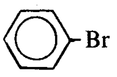
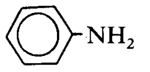
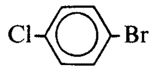
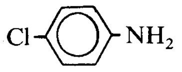
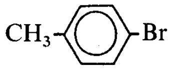
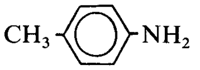
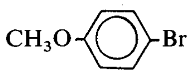
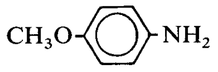
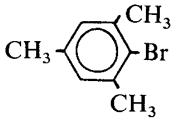
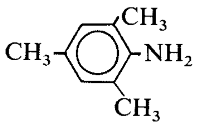
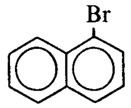
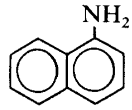
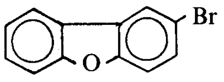
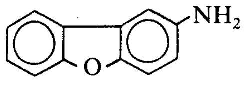
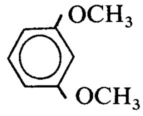
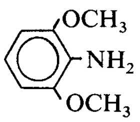
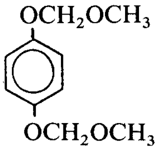
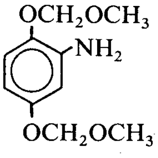
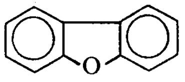
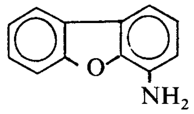
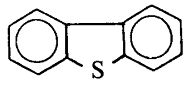
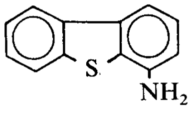
## Experimental

Melting points are uncorrected. Infrared (IR) spectra were recorded on a JASCO IRA-2 spectrometer (Nujol mulls or potassium bromide disks for crystals and films for oils). Proton nuclear magnetic resonance ($^1$H-NMR) spectra were recorded on a JEOL PMX-60 or MH-100 spectrometer with tetramethylsilane as an internal standard in deuteriochloroform. Silica gel (Mallinckrodt CC-7 special) was used for column chromatography. Preparative layer chromatography (PLC) was carried out on plates (20 × 20 cm, 2 mm thickness) precoated with silica gel (60F$_{254}$, Merck Art.5717). Quantitative analysis by thin layer chromatography (TLC) was carried out on silica gel plates (60F$_{254}$, 5 × 5 cm, 0.25 mm, Merck Art. 5715) in conjunction with a Shimadzu CS-920 high-speed TLC scanner. Each spot was detected under ultraviolet light (254 nm).

Most of the starting materials for the amination were purchased, distilled, and dried over molecular sieves 4A. 2-Bromodibenzofuran[11)] and hydroquinone bis(methoxymethyl) ether[12)] were prepared according to the literature. Preparation of organometallics, reaction with DPPA, and treatment with hydride reagents were carried out under argon.

Table V. Amination of Aromatic and Heteroaromatic Organometallics Using DPPA and Sodium Bis(2-methoxyethoxy)aluminum Hydride



| Run | Starting material | Product | Reaction solvent | Yield, % | Reported Yield, % |
|---|---|---|---|---|---|
| 1 |  |  | $Et_2O$ | 73$^{a)}$ | 67.5,$^{6b)}$ 68,$^{6c)}$ 80—85,$^{6e)}$ 72,$^{6f)}$ 90,$^{7a)}$ 67,$^{7c)}$ 35$^{7d)}$ |
| 2 |  |  | $Et_2O$ | 79$^{a)}$ | 49,$^{6a)}$ 92$^{6f)}$ |
| 3 |  |  | $Et_2O$ | 88$^{a)}$ | 66$^{6a)}$ |
| 4 |  |  | THF | 84 | 51,$^{6a)}$ 50,$^{6b)}$ 69$^{6f)}$ |
| 5 |  |  | $Et_2O$ | 67 | — |
| 6 |  |  | $Et_2O$ | 89 | 31$^{7c)}$ |
| 7 |  |  | THF | 71 | — |
| 8 |  |  | THF | 72 | 50—67,$^{6b)}$ 70$^{6c)}$ |
| 9 |  |  | THF | 47 | — |
| 10 |  |  | THF | 58$^{b)}$ (88)$^{c)}$ | — |
| 11 |  |  | THF | 62$^{b)}$ (99)$^{c)}$ | 55$^{7a)}$ |

a) Isolated as the hydrochloride. b) Yield based on starting material. c) Yield based on consumed starting material.

**1-Phenyl-3-diphenoxyphosphinyltriazene (4)**——The Grignard reagent in $Et_2O$ (1.64 ml, 1.1 mmol),$^{13)}$ prepared from bromobenzene and magnesium as described in the general procedure, was added dropwise to DPPA (275 mg, 1.0 mmol) in $Et_2O$ (10 ml) at the temperature indicated in Table I during 3—10 min. The mixture was stirred under the reaction conditions shown in Table I, and cold satd. aq. $NH_4Cl$ was added. The mixture was extracted with benzene–$Et_2O$ (1 : 1). The extracts were washed with water and satd. aq. NaCl, dried over $Na_2SO_4$, and diluted to 100 ml with benzene–$Et_2O$ (1 : 1). An aliquot (1 $\mu$l) was chromatographed on a TLC plate using benzene–hexane–$Et_2O$ (5 : 3 : 5), and the yield of the triazene **4** (*Rf* 0.37) was determined with the TLC scanner.

In run 5, the washed and dried organic extracts were concentrated *in vacuo* and the residue was purified by silica gel column chromatography with benzene–hexane–$Et_2O$ (5 : 2 : 1) to give the triazene **4** (270 mg, 76%) as colorless crystals (from $Et_2O$–hexane), mp 85.5—86.5 °C (dec.), which colored brown on exposure to light. IR $\nu_{max}$ $cm^{-1}$: 3060, 1260. NMR $\delta$: 7.1—7.7 (15H, m), 8.4—9.6 (1H, br s, disappeared on addition of $D_2O$). *Anal.* Calcd for $C_{18}H_{16}N_3O_3P$: C, 61.19; H, 4.56; N, 11.89. Found: C, 61.25; H, 4.56; N, 11.91.

**Decomposition of 1-Phenyl-3-diphenoxyphosphinyltriazene (4)**——The reaction was carried out under shielding from light. (i) Run 2 in Table II. A mixture of the triazene **4** (177 mg, 0.5 mmol) in THF (1 ml) and 30% methanolic KOH (1 ml) was stirred at room temperature for 4 d. After the addition of 10% NaOH (2 ml), the mixture was salted out and extracted with benzene–$Et_2O$. The extracts were washed with satd. aq. NaCl, dried over $Na_2SO_4$, and concentrated *in vacuo* to give aniline (25 mg, 54%).

(ii) Run 3 in Table II. A mixture of the triazene **4** (177 mg, 0.5 mmol) and Silica gel 60 (177 mg, Merck 230—400 mesh) in benzene (2 ml) was stirred for 4 d. Aniline was not detected by TLC in the reaction mixture.

(iii) Run 6 in Table II. A 10% methanolic HCl solution (1 ml) was added to the triazene **4** (353 mg, 1 mmol) in MeOH (1 ml) at 0 °C. The mixture was stirred at room temperature until the triazene was no longer detectable on a TLC plate (3 h and 20 min). After concentration, the residue was washed with benzene and $Et_2O$ to give aniline hydrochloride (102 mg, 79%).

**Conversion of Aromatic Bromides to Aromatic Amines (Table III)**——General procedure. The aromatic Grignard compound [prepared from the aromatic bromide (3.3 mmol) and magnesium (88 mg, 3.63 mg-atom, activated with 1 drop of 1,2-dibromoethane) in $Et_2O$ (5 ml)] was added to DPPA (826 mg, 3 mmol) in $Et_2O$ (30 ml) at −72 °C. The mixture was stirred under shielding from light at −72 °C for 2 h, then warmed to −20 °C during 40 min. Cold aq. $NH_4Cl$ was added, and the mixture was extracted with benzene–$Et_2O$. The extracts were washed with satd. aq. NaCl, dried over $Na_2SO_4$, and concentrated to give the crude triazene.

A 10% methanolic HCl solution (3 ml) was added to the triazene in MeOH (3 ml) at 0 °C under shielding from light. The mixture was stirred at room temperature overnight (2 h in run 1) until the triazene was no longer detectable on TLC, then concentrated *in vacuo*. The residue was basified with 50% KOH and extracted with benzene–$Et_2O$. The extracts were washed with satd. aq. NaCl, dried over $Na_2SO_4$, and concentrated. After addition of 5% methanolic HCl to the residue, the mixture was concentrated and the residue was washed with benzene and $Et_2O$ to give the aromatic amine hydrochloride, which was converted to the free amine and identified by spectral comparisons with an authentic sample.[14,15]

**1-(1-Naphthyl)-3-diphenoxyphosphinyltriazene (5)**——Prepared as described for **4** except that a mixture of $Et_2O$–benzene (2.5 : 1) was used in the preparation of 1-naphthylmagnesium bromide. Purification of the crude triazene was done by silica gel column chromatography with benzene–hexane–$Et_2O$ (5 : 2 : 1) to give **5** (42.5%) as a rather labile, colorless, viscous oil, which was kept in a refrigerator. IR $\nu_{max}$ $cm^{-1}$: 3050, 1270.

**Decomposition of 1-(1-Naphthyl)-3-diphenoxyphosphinyltriazene (5)**——The triazene **5** (224 mg, 0.56 mmol) in $Et_2O$ (15 ml) was added to $LiAlH_4$ (75 mg, 2 mmol) in $Et_2O$ (10 ml) at room temperature. The mixture was stirred at room temperature overnight and cooled to 0 °C. Cold water was added, and the insoluble material was filtered off and washed with water, benzene, and $Et_2O$. The organic layer was washed with water, dried over $Na_2SO_4$, and concentrated *in vacuo*. The residue was purified by PLC with hexane–$Et_2O$ (1 : 1) to give 1-naphthylamine (51 mg, 64%).

**General Procedure for the Preparation of Grignard Reagents**——1,2-Dibromoethane (3 drops) was added to magnesium (1.07 g, 44 mg-atom) in $Et_2O$ (4 ml). An aromatic or a heteroaromatic bromide (40 mmol) in $Et_2O$ (20 ml) was added at a rate sufficient to maintain gentle reflux. After the addition, the mixture was refluxed for 0.5 h, cooled, and diluted with $Et_2O$ (24 ml) (or 24 ml of benzene in run 6 of Table V).[13]

**General Procedure for the Preparation of Ortho Lithiated Compounds**——*n*-Butyllithium in hexane (10% (w/v), 0.71 ml, 1.1 mmol) was added to an arene or heteroarene (1.1 mmol), as shown in Table V, in THF (2.5—5 ml) at 0 °C. The mixture was stirred at room temperature for 20 min to give a solution of the ortho lithiated compound.

**Amination of Aromatic and Heteroaromatic Organometallics Using DPPA and Sodium Bis(2-methoxyethoxy)aluminum Hydride (Table V)**——General Procedure. A Grignard or lithium compound (1.1 mmol) in $Et_2O$ (runs 1—3, 5, 6) or THF (runs 4, 7—11) was added to DPPA (275 mg, 1 mmol) in $Et_2O$ or THF (10 ml) at −72 °C. The mixture was stirred under shielding from light at −72 °C for 2 h, then warmed to −20 °C during 40 min. The mixture was again cooled to −70 °C, and sodium bis(2-methoxyethoxy)aluminum hydride in toluene (70% (w/w), 1.23 ml, 4.4 mmol) was added at −70 °C. The mixture was stirred at 0 °C for 1 h, then at room temperature for 0.5 h. Ice-water was carefully added to the mixture at 0 °C. The insoluble material was filtered off, and washed with water, benzene, and $Et_2O$. The combined filtrate was salted out by the addition of NaCl. The separated organic layer was washed successively with 1% NaOH and satd. aq. NaCl, and dried over $Na_2SO_4$. Free amine was obtained by removal of the solvent followed by purification of the concentrated residue.

Alternatively, 5% methanolic HCl (*ca.* 3 ml) was added to the dried solution, and the mixture was concentrated *in vacuo* to give the solid residue, which was washed with $Et_2O$ to give the hydrochloride of the amine.

In runs 1—6, each product was identified as the free amine by spectral comparisons with the corresponding authentic sample.[14,15]

The other products in runs 7—11 were identified by comparisons of their melting points with the literature values as well as from the IR and NMR spectra, as follows.

2-Aminodibenzofuran (run 7): mp 126.5—128 °C (lit.[16] mp 127—128 °C); IR $\nu_{\max}$ cm$^{-1}$: 3420, 3340.

2,6-Dimethoxyaniline (run 8): mp 74—76 °C (lit.[6b] mp 75—76 °C); IR $\nu_{\max}$ cm$^{-1}$: 3460, 3370; NMR $\delta$: 3.80 (8H, s, 2H, disappeared on addition of $D_2O$), 6.37—6.86 (3H, m).

2,5-Bis(methoxymethyloxy)aniline (run 9): IR $\nu_{\max}$ cm$^{-1}$: 3440, 3360; NMR $\delta$: 3.44 (3H, s), 3.47 (3H, s), 3.75 (2H, s, disappeared on addition of $D_2O$), 5.06 (2H, s), 5.09 (2H, s), 6.23—6.55 (2H, m), 6.93 (1H, d, $J$=8.5 Hz).

4-Aminodibenzofuran (run 10): mp 85.5—86 °C (lit.[16] mp 84.5—85.5 °C); IR $\nu_{\max}$ cm$^{-1}$: 3400, 3310.

4-Aminodibenzothiophene (run 11): mp 108.5—110 °C (lit.[17] mp 110 °C); IR $\nu_{\max}$ cm$^{-1}$: 3310, 3200.

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