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of Reliable Methods  
for the Preparation  
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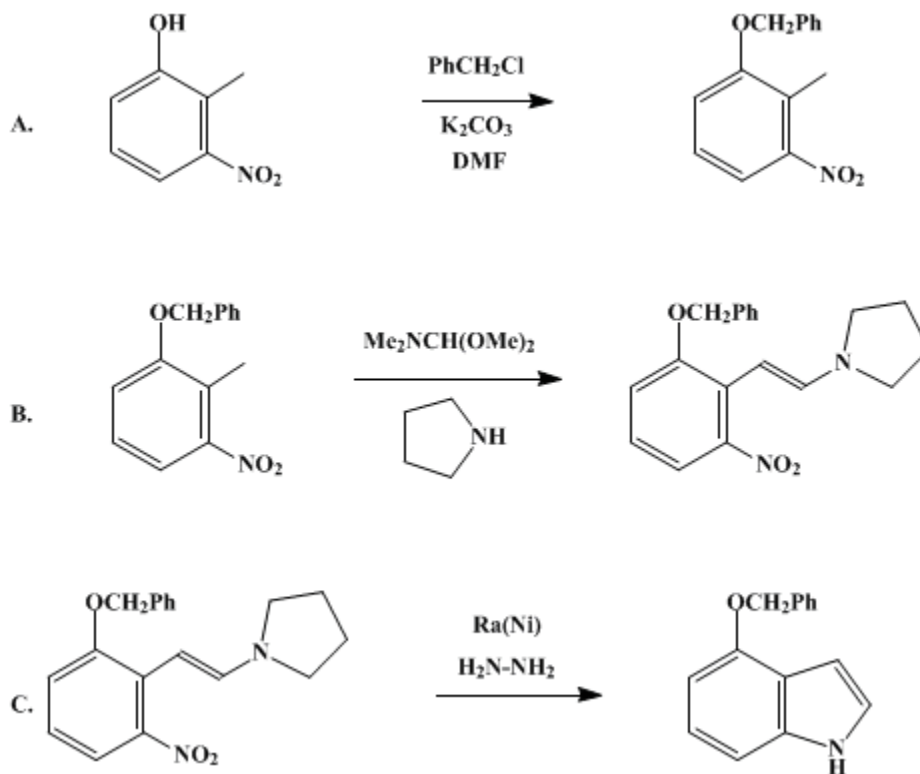
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*September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

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## INDOLES FROM 2-METHYLNITROBENZENES BY CONDENSATION WITH FORMAMIDE ACETALS FOLLOWED BY REDUCTION: 4-BENZYLOXYINDOLE

[1*H*-Indole, 4-(phenylmethoxy)-]



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### 1. Procedure

A. *6-Benzyloxy-2-nitrotoluene*. A stirred mixture of 124.7 g (0.81 mol) of 2-methyl-3-nitrophenol (Note 1), 113.2 g (0.90 mol) of benzyl chloride, 112.2 g (0.81 mol) of anhydrous potassium carbonate, and 800 mL of dimethylformamide (DMF) is heated at 90°C for 3 hr. Most of the DMF is removed on a rotary evaporator (20 mm) and the oily residue is poured into 400 mL of 1 *N* sodium hydroxide and extracted with ether (3 × 800 mL). The combined extracts are dried (Na<sub>2</sub>SO<sub>4</sub>), filtered, and evaporated to give 203.5 g of yellowish solid. Recrystallization from 1 L of methanol cooled to 0°C affords 177.6 (90%) of 6-benzyloxy-2-nitrotoluene as pale-yellow crystals, mp 61–63°C<sup>3</sup> (Note 2).

B. *(E)-6-Benzyloxy-2-nitro-β-pyrrolidinostyrene*. To a solution of 175.4 g (0.72 mol) of 6-benzyloxy-2-nitrotoluene in 400 mL of DMF are added 102.5 g (0.84 mol) of *N,N*-dimethylformamide dimethyl acetal (Note 3) and 59.8 g (0.84 mol) of pyrrolidine. The solution is heated at reflux (110°C) for 3 hr (Note 4) under nitrogen and allowed to cool to room temperature. The volatile components are removed on a rotary evaporator, and the red residue (Note 5) is dissolved in 200 mL of methylene chloride and 1.60 L of methanol. The solution is concentrated to a volume of about 1.40 L on the steam bath and then is cooled to 5°C. Filtration and washing of the filtercake with 200 mL of cold methanol affords 209.8 g of red crystals, mp 87–89°C (Note 6). The mother liquors are evaporated, and the residue is recrystallized from 50 mL of methanol (5°C) to give an additional 12.30 g of red solid, mp 81–83°C (Note 7). Thus the total yield is 222.1 g (95%) of a 15 : 1 mixture of (*E*)-6-benzyloxy-2-nitro-

$\beta$ -pyrrolidinostyrene (Note 8) and (*E*)-6-benzyloxy- $\beta$ -dimethylamino-2-nitrostyrene.

C. *4-Benzyloxyindole*. To a stirred solution of 162.2 g (0.50 mol) of (*E*)-6-benzyloxy-2-nitro- $\beta$ -pyrrolidinostyrene (Note 9) in 1 L of THF and 1 L of methanol at 30°C under nitrogen is added 10 mL of Raney nickel (Note 10) followed by 44 mL (0.75 mol) of 85% hydrazine hydrate. Vigorous gas evolution is observed. The red color turns to dark brown within 10 min, and the reaction temperature rises to 46°C. An additional 44 mL of 85% hydrazine hydrate is added after 30 min and again 1 hr later. The temperature is maintained between 45 and 50°C with a water bath during the reaction and for 2 hr after the last addition. The mixture is cooled to room temperature and the catalyst is removed by filtration through a bed of Celite (Note 11) and is washed several times with methylene chloride. The filtrate is evaporated and the residue dried by evaporating with 500 mL of toluene. The reddish residue (118.5 g), dissolved in ca. 1 L of toluene-cyclohexane (1 : 1), is applied to a column of 500 g of silica gel (70–230-mesh, Merck) prepared in the same solvent. Elution with 6.0 L of toluene-cyclohexane (1 : 1) followed by 3 L of toluene-cyclohexane (1 : 2) affords 108.3 g of white solid, which is crystallized from 150 mL of toluene and 480 mL of cyclohexane (Note 12). A total of 107.3 g (96% yield) of 4-benzyloxyindole (Note 13) is obtained in three crops as white prisms, mp 60–62°C (Note 14).

## 2. Notes

1. 2-Methyl-3-nitrophenol was obtained from Aldrich Chemical Company, Inc.
2. The <sup>1</sup>H NMR spectrum is as follows  $\delta$ : 2.42 (s, 3 H), 5.10 (s, 2 H), 7.13 (m, 3 H), 7.35 (m, 5 H).
3. *N,N*-Dimethylformamide dimethyl acetal was prepared according to a procedure of Brederick.<sup>4</sup> *N,N*-Dimethylformamide diethyl acetal can also be used. Both the dimethyl and the diethyl acetal are commercially available from Aldrich Chemical Company, Inc.
4. The reaction was followed by TLC on silica gel plates developed with ether-petroleum ether (1 : 1).
5. Since it contained nonvolatile *N*-formylpyrrolidine, direct reduction of the crude material was not attempted.
6. This crop contained 5% 6-benzyloxy- $\beta$ -dimethylamino-2-nitrostyrene (by NMR). Pure 6-benzyloxy-2-nitro- $\beta$ -pyrrolidinostyrene melts at 91.5–92.5°C.
7. This crop contained 15% 6-benzyloxy- $\beta$ -dimethylamino-2-nitrostyrene (by NMR).
8. The <sup>1</sup>H NMR spectrum is as follows:  $\delta$ : 1.8 (m, 4 H), 3.08 (m, 4 H), 5.03 (s, 2 H), 5.20 (d, 1 H, *J* = 12.2), 6.91 (dd, 2 H, *J* = 9), 7.25 (m, 6 H), 7.75 (d, 1 H, *J* = 12.2).
9. This compound may contain varying amounts of 6-benzyloxy- $\beta$ -dimethylamino-2-nitrostyrene.
10. Raney nickel is commercially available as type #28 from the Davison Chemical Division of W. R. Grace and Co.
11. The catalyst is pyrophoric and should not be sucked dry.
12. The material tenaciously holds hydrocarbons, such as pentane, hexane, and petroleum ether, which cannot be removed even under high vacuum. The solvated crystals show hydrocarbon protons in NMR and exhibit a broad melting point. However, we found that cyclohexane is not retained in the crystals.
13. The <sup>1</sup>H NMR spectrum is as follows:  $\delta$ : 6.65 (m, 2 H), 6.95 (m, 3 H), 7.9 (br s, 1 H), 7.32 (m, 5 H).
14. We could not reproduce the reported<sup>5</sup> melting point of 72–74°C (toluene). The material has the proper microanalysis and is pure by NMR and thin-layer chromatography (TLC).

## 3. Discussion

Through the years, widespread interest in the synthesis of natural products and their analogs bearing the oxygenated indole nucleus has led to the development of several routes to protected hydroxylated indoles. However, 4-benzyloxyindole was first prepared relatively recently in modest overall yield by the Reissert method, which involves condensation of 6-benzyloxy-2-nitrotoluene with ethyl oxalate, reductive cyclization to the indole-2-carboxylate, hydrolysis to the acid, and decarboxylation.<sup>5</sup>

Although a variety of synthetic methods have been used to prepare indoles,<sup>6,7</sup> many of these lack generality and are somewhat restrictive since they employ conditions, such as acid or strongly basic cyclizations or thermal decarboxylations, which are too harsh for labile substituents. This efficient, two-step procedure<sup>8,9</sup> illustrates a general, simple, and convenient process for preparing a variety of indoles substituted in the carbocyclic ring, as can be seen in Table I. Since many of these examples served to determine the scope of this method, the yields in most cases have not been optimized. In many cases,

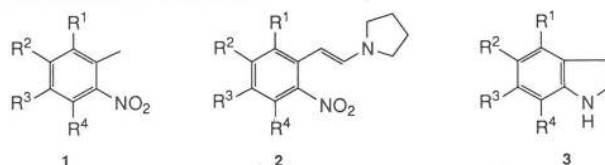
the starting materials are readily available or can be easily prepared.

As can be seen in Table I, variation of the substituent has a profound effect on the rate of reaction of the *o*-nitrotoluene derivative with dimethylformamide acetals but has little effect on the yields, which are often almost quantitative. As can be predicted, electron-withdrawing groups accelerate the reaction. To shorten the somewhat lengthy reaction times that are often necessary when electron-donating substituents are present, more reactive aminomethylenating reagents such as pyrrolidine (or piperidine) acetals,<sup>8</sup> aminals,<sup>10</sup> or trisaminomethanes<sup>11</sup> can be employed. Alternatively, as described above, simply adding pyrrolidine to the reaction mixture also generates in situ a very effective aminomethylenating reagent.<sup>12,13</sup> Thus, for example, in the case of 6-benzyloxy-2-nitrotoluene, the reaction with *N,N*-dimethylformamide dimethyl acetal requires 51 hr versus 3 hr when pyrrolidine is added. Pyrrolidine undergoes exchange reactions with *N,N*-dimethylformamide acetals to produce an equilibrium mixture of formylpyrrolidine acetal and the mixed pyrrolidine dimethylamine aminal (alkoxydimethylaminopyrrolidinomethane) as well as other trisaminomethane species.<sup>14</sup> (In cases where pyrrolidine reacts with the aromatic substrate, addition of the substrate can be delayed until pyrrolidine exchange is complete.) This mixture of reagents gives rise to condensation products—pyrrolidine enamines—that contain 5–10% of the corresponding *N,N*-dimethylenamines.

TABLE I  
INDOLES FROM 2-METHYLNITROBENZENES BY CONDENSATION WITH *N,N*-DIMETHYLFORMAMIDE ACETALS AND REDUCTION

Substituents				Intermediates 2 (mp or bp/mm)	Reaction Time	Purified Yield (%) (Procedure) <sup>a</sup>	Indoles 3 (mp or bp/mm)	Yield (%) (Procedure) <sup>b</sup>	Refs.
R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup>	R <sup>4</sup>						
—	—	—	—	125°/0.03	22 hr	97(M,E)	52.5–53.5°	80(A)	8, 9
OCH <sub>2</sub> Ph	—	—	—	67–68°	51 hr	90(M)	60–62°	70(B)	12
—	OCH <sub>2</sub> Ph	—	—	98–99°	29 hr	78(E)	103–105°	45(B)[64] <sup>c</sup>	8, 9, 12
—	—	OCH <sub>2</sub> Ph	—	108.5–110°	41 hr	97(M)	118–120°	75(B) <sup>i</sup>	12
—	OCH <sub>2</sub> Ph	OCH <sub>2</sub> Ph	—	99.5–101°	48 hr	86(M)	112–113°	54(B) <sup>j</sup>	8, 9
—	OCH <sub>2</sub> Ph	CH <sub>3</sub>	—	113–134°	31 hr	87(M)	82–83°	84(B)	12
—	OCH <sub>3</sub>	—	—	68.5–70°	16 hr	92(M)	56.5–57.5°	83(A)	8, 9
—	—	OCH <sub>3</sub>	—	152°/0.06	70 hr	64(E)	88–90°	63(A)[62] <sup>c</sup>	8, 9, 12
—	OCH <sub>3</sub>	OCH <sub>3</sub>	—	125–126°	48 hr	68(M)	154–155°	28(A)	8, 9
—	OCH <sub>3</sub>	—	CH <sub>3</sub>	100–101°	8 hr	54(M)	100–110°/ 0.15	66(A)	22
—	—	OCH <sub>2</sub> O	—	114–116°	18 hr	72(E)	110–111°	50(A)[52] <sup>c</sup>	8, 9, 12
Cl	—	—	—	111°/0.03	6 hr	89(E)	90°/0.04	63(B)	8, 9
—	Cl	—	—	81.5–82.5°	7 hr	88(E)	71–72°	78(B)	8, 9
—	—	Cl	—	44–46°	24 hr	57(M)	86.5–88°	52(B)[75] <sup>c</sup>	8, 9, 12
—	—	NH <sub>2</sub> <sup>d</sup>	—	173–174°	2 hr	82(E) <sup>f</sup>	77.5–78.5°	43(A)	8, 9, 12
CN	—	—	—	66–68°	3 hr	93(M)	116–117°	67(C)	17
—	—	CN	—	134–137.5°	2.5 hr	86(E)	128–129°	65(A)	8, 9
—	F	—	—	57.5–59°	3.5 hr	92(E)	46.5–47°	51(B)	8, 9
—	—	F	—	46–47°	22 hr	63(M)	74–75°	80(B)[80] <sup>c</sup>	8, 12
CH <sub>3</sub>	—	—	—	108°/0.05	24 hr	70(E)	82°/0.4	57(A)	8, 9
—	—	CH <sub>3</sub>	—	41.5–43.5°	37 hr	83(M)	29–30.5°	83(A)	23
—	—	—	CH <sub>3</sub>	76–76.5	46 hr	40(E)	83–84°	48(A)	8, 9
—	—	CH(CH <sub>3</sub> ) <sub>2</sub>	—	138–140°/0.06	42 hr	84(E)	40–41°	51(A)	8, 9
—	—	CH(OCH <sub>3</sub> ) <sub>2</sub>	—	67–68°	8 hr	55(E)	62–63.5°	31(A)	8, 9
COOCH <sub>3</sub>	—	—	—	120–130°/0.2	6 hr	86(M)	63°	82(A)[63] <sup>c</sup>	17, 24
COOC <sub>2</sub> H <sub>5</sub>	—	—	—	(Oil)	5 days	93(E)	67–69°	38(D)	17
—	COOC <sub>2</sub> H <sub>5</sub>	—	—	55–56.5°	4.5 hr	70(E)	95–96°	39(A)	8, 9
—	—	—	COOCH <sub>3</sub>	132–134°	9 hr	88(M)	46–48°	72(A) <sup>g</sup>	12

TABLE I (Continued)  
INDOLES FROM 2-METHYLNITROBENZENES BY CONDENSATION WITH *N,N*-DIMETHYLFORMAMIDE ACETALS AND REDUCTION



Substituents				Intermediates 2 (mp or bp/mm)	Reaction Time	Purified Yield (%) (Procedure) <sup>d</sup>	Indoles 3 (mp or bp/mm)	Yield (%) (Procedure) <sup>h</sup>	Refs.
R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup>	R <sup>4</sup>						
Cl	OCH <sub>3</sub>	—	—	—	Over- night	—(M)	109–111°	(B)[59] <sup>c</sup>	25
—	OCH <sub>3</sub>	Cl	—	140–141°	Over- night	78(M)	126–128°	46(B)[45] <sup>c</sup>	25
—	OCH <sub>3</sub>	F	—	116–117°	Over- night	64(M)	73–74°	54(B)	25
—	—	Br	—	—	31 hr	—(M)	93°	37(B) <sup>h</sup>	26

<sup>a</sup>(M) = *N,N*-Dimethylformamide dimethyl acetal; (E) = *N,N*-dimethylformamide diethyl acetal.

<sup>b</sup>A = Catalytic hydrogenation in benzene using palladium on charcoal; B = catalytic hydrogenation in benzene using Raney nickel; C = iron in acetic acid; D = stannous chloride.

<sup>c</sup>Yield in brackets represents overall yield without purification of intermediate 2.

<sup>d</sup>R<sup>3</sup> = NO<sub>2</sub> in compounds 1 and 2.

<sup>e</sup>R<sup>2</sup> = COOH in compound 1.

<sup>f</sup>No solvent was used.

<sup>g</sup>Methanol was the solvent.

<sup>h</sup>Ethanol was the solvent.

<sup>i</sup>(M) + pyrrolidine gave a mixture (10 : 1) of pyrrolidine enamine, mp 108–110° (MeOH), and *N,N*-dimethylenamine (5 hr reflux, 97% yield), which, on reduction (Raney nickel-hydrazine), gave the indole (93% yield).

<sup>j</sup>(M) + pyrrolidine gave a mixture (9 : 1) of pyrrolidine enamine and *N,N*-dimethylenamine (5 hr reflux, 95% yield), which, on reduction (Raney nickel-hydrazine), gave the indole (89% yield).

The enamine intermediates are usually crystalline, red compounds that can be stored at room temperature for reasonable periods. In cases where the enamines are noncrystalline, it is recommended that the crude product be used directly in the next step, since purification is, in such cases, not practical. Although the more volatile derivatives can be distilled under high vacuum, this entails some risk because of their thermal instability. Moreover, the enamines are not stable to silica gel (TLC or column) chromatography.

Conversion of the intermediate nitroenamine into the indole requires selective reduction of the nitro group. Catalytic hydrogenation results in spontaneous formation of the indole and is generally the mildest and most convenient method of reduction. Although selectivity does vary with the substituent on the aromatic ring, it is generally highly in favor of the nitro group. However, scale-up requires access to large autoclaves or special equipment. To avoid hydrogenolysis of benzyl or chloro functions, **Raney nickel** is the catalyst of choice. Excellent yields have been obtained using **hydrazine** and the appropriate catalyst<sup>20</sup> as, in essence, a hydrogenation process which does not require an autoclave or special equipment and can be easily carried out in the laboratory.

Alternative methods of reduction have also been used: **sodium dithionite**,<sup>21</sup> **iron in acetic acid**,<sup>22</sup> **stannous chloride**,<sup>22</sup> and **titanium trichloride**.<sup>23</sup>

This method has been applied to the preparation of polycyclic indoles<sup>12,24</sup> and azaindoles<sup>24,25,26</sup> as well.



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## References and Notes

1. Research Division, Hoffmann-La Roche, Inc., Nutley, NJ 07110.
2. Deceased.
3. Beer, R. J. S.; Clarke, K.; Khorana, H. G.; Robertson, A. *J. Chem. Soc.* **1948**, 1605.
4. Bredereck, H.; Simchen, G.; Rebsdats, S.; Kantlehner, W.; Horn, P.; Wahl, R.; Grieshaber, P. *Chem. Ber.* **1968**, *101*, 47.
5. Stoll, A.; Troxler, F.; Peyer, J.; Hofmann, A. *Helv. Chim. Acta* **1955**, *38*, 1452.
6. Sundberg, R. J. "The Chemistry of Indoles," Academic Press: New York, 1970.
7. Brown, R. K. In "Indoles," Houlihan, W. J., Ed.; Wiley-Interscience: New York, 1972, Part 1, Chapter 2.
8. Leimgruber, W.; Batcho, A. D. Third International Congress of Heterocyclic Chemistry, Japan, August 23–27, 1971.
9. Batcho, A. D.; Leimgruber, W. U.S. Patent 3732245, 1973; U.S. Patent 3976639, 1976; *Chem Abstr.* **1977**, *86*, 29624.
10. Bredereck, H.; Simchen, G.; Wahl, R. *Chem. Ber.* **1968**, *101*, 4048.
11. Kruse, L. I. *Heterocycles* **1981**, *16*, 1119.
12. Batcho, A. D.; Leimgruber, W., unpublished results.
13. Maehr, H.; Smallheer, J. M. *J. Org. Chem.* **1981**, *46*, 1752.
14. See: Abdulla, R. F.; Brinkmeyer, R. S. *Tetrahedron* **1979**, *35*, 1675.
15. Benington, F.; Morin, R. D.; Bradley, R. J. *J. Heterocycl. Chem.* **1976**, *13*, 749.
16. Hengartner, U.; Valentine, D., Jr.; Johnson, K. K.; Larscheid, M. E.; Pigott, F.; Scheidl, F.; Scott, J. W.; Sun, R. C.; Townsend, J. M.; Williams, T. H. *J. Org. Chem.* **1979**, *44*, 3741.
17. Kozikowski, A. P.; Ishida, H.; Chen, Y.-Y. *J. Org. Chem.* **1980**, *45*, 3350.
18. Flaugh, M. E.; Crowell, T. A.; Clemens, J. A.; Sawyer, B. D. *J. Med. Chem.* **1979**, *22*, 63.
19. Dellar, G.; Djura, P.; Sargent, M. V. *J. Chem. Soc., Perkin Trans. 1* **1981**, 1679.
20. Furst, A.; Berlo, R. C.; Hooton, S. *Chem. Rev.* **1965**, *65*, 51.
21. Garcia, E. E.; Fryer, R. I. *J. Heterocycl. Chem.* **1974**, *11*, 219.
22. Ponticello, G. S.; Baldwin, J. J. *J. Org. Chem.* **1979**, *44*, 4003.
23. Somei, M.; Inoue, S.; Tokutake, S.; Yamada, F.; Kaneko, C. *Chem. Pharm. Bull.* **1981**, *29*, 726.
24. Gassman, P. G.; Schenk, W. N. *J. Org. Chem.* **1977**, *42*, 3240.
25. Prokopov, A. A.; Yakhontov, L. N. *Khim. Geterotsikl. Soedin* **1977**, 1135; *Chem. Abstr.* **1977**, *87*, 201382w.
26. Prokopov, A. A.; Yakhontov, L. N. *Khim. Geterotsikl. Soedin* **1979**, *86*; *Chem. Abstr.* **1979**, *90*, 15204u.

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## Appendix

### Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

petroleum ether

2-METHYLNITROBENZENES

toluene-cyclohexane

dimethylformamide acetals

pyrrolidine enamines

N,N-dimethylenamines

N,N-dimethylenamine

ethanol (64-17-5)  
potassium carbonate (584-08-7)  
acetic acid (64-19-7)  
Benzene (71-43-2)  
methanol (67-56-1)  
ether (60-29-7)  
sodium hydroxide (1310-73-2)  
iron (7439-89-6)  
 $\text{Na}_2\text{SO}_4$  (7757-82-6)  
nitrogen (7727-37-9)  
sodium dithionite (7775-14-6)  
stannous chloride  
cyclohexane (110-82-7)  
Raney nickel (7440-02-0)  
toluene (108-88-3)  
palladium (7440-05-3)  
benzyl chloride (100-44-7)  
hydrazine hydrate (7803-57-8)  
Ethyl oxalate  
dimethylamine (124-40-3)  
Pentane (109-66-0)  
hydrazine (302-01-2)  
methylene chloride (75-09-2)  
dimethylenamine (9002-98-6)  
THF (109-99-9)

N,N-dimethylformamide,  
dimethylformamide,  
DMF (68-12-2)

pyrrolidine (123-75-1)

hexane (110-54-3)

N,N-Dimethylformamide diethyl acetal (1188-33-6)

pyrrolidine enamine

titanium trichloride (7705-07-9)

4-Benzyloxyindole,  
1H-Indole, 4-(phenylmethoxy)- (20289-26-3)

2-methyl-3-nitrophenol (5460-31-1)

6-Benzyloxy-2-nitrotoluene (20876-37-3)

6-benzyloxy- $\beta$ -dimethylamino-2-nitrostyrene

6-benzyloxy-2-nitro- $\beta$ -pyrrolidinostyrene

indole-2-carboxylate

formylpyrrolidine acetal

trisaminomethane

N,N-dimethylformamide dimethyl acetal (4637-24-5)

(E)-6-Benzyloxy-2-nitro- $\beta$ -pyrrolidinostyrene (99474-12-1)

(E)-6-benzyloxy- $\beta$ -dimethylamino-2-nitrostyrene

N-formylpyrrolidine (3760-54-1)