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Mechanisms for the Reactions of Nitrones with Aroyl Chlorides

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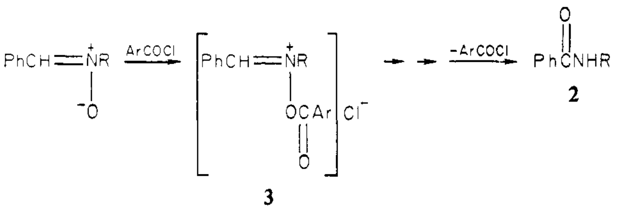
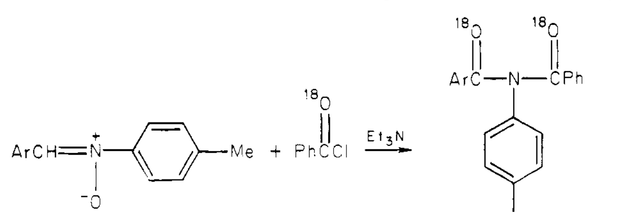
Abstract: Reaction of nitrones with equivalent quantities of aroyl chlorides in anhydrous dioxane containing triethylamine yields *N*,*N*-diacylamines. Treatment of nitrones with ^{18}O-labeled benzoyl chloride under these conditions forms *N*,*N*-diacylamines with the ^{18}O label distributed equally between the two acyl groups. Reaction of nitrones with equivalent or excess amounts of aroyl chlorides in anhydrous dioxane in the absence of triethylamine brings about isomerization of the nitrones to amides. α-Phenyl-*N*-(*p*-tolyl)nitrone treated with [^{18}O]benzoyl chloride leads to an amide containing 50% of the ^{18}O label. Both reactions can be rationalized on the basis of the formation of aroyloxy(benzylidene)ammonium chlorides which undergo elimination of acid anions or acids to yield nitrilium ions. Addition of the acid anions or acids to the nitrilium ions affords the precursors to the *N*,*N*-diacylamines or isomerized amides, respectively.

Aldonitrones (**1**) are easily isomerized to amides (**2**) by catalytic amounts of acylating agents in anhydrous solvents. Many examples of the isomerization have been reported since Beckmann's observation in 1890 that α-phenyl-*N*-benzylnitrone is converted into *N*-benzylbenzamide by benzoyl chloride.[1-5] Recently evidence was presented indicating that the first step in the isomerization is the formation of an aroyloxy(benzylidene)ammonium chloride **3**.[6,7] What follows is an account of the reactions of nitrones when they are treated with an equivalent or more of aroyl chlorides in dry dioxane in the presence and in the absence of triethylamine. Formation of the products, *N*,*N*-diacylamines and amides, respectively, is explicable on the basis of the intermediacy of nitrilium ions—a species heretofore not implicated in these reactions.



Results and Discussion

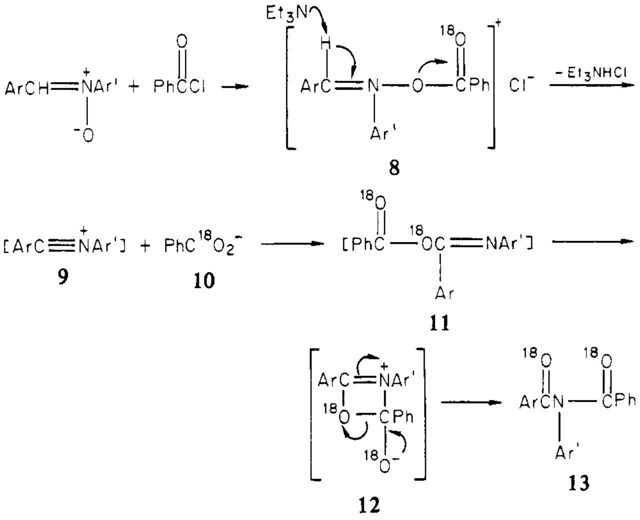
Aroyl chlorides added to mixtures of nitrones and triethylamine in dry dioxane at ambient temperature form *N*,*N*-diacylamines (Table I). An insight into the pathway of the reaction was provided by reacting nitrone **4** with an equivalent of [^{18}O]benzoyl chloride (92% ^{18}O enriched). Each of the carbonyl moieties of the diacylamine **5** contained 50% of the ^{18}O label (as shown by the ^{18}O content of the ions PhCO$^+$ and ArCO$^+$ in the mass spectrum of **5**). Nitrone **6** reacted analogously with [^{18}O]benzoyl chloride to give **7**. Control runs involving the addition of benzoyl chloride to a mixture of either *N*-(*p*-tolyl)benzamide or *N*-*tert*-butyl]benzamide and triethylamine in dry dioxane resulted in the recovery (>95%) of the starting amide.



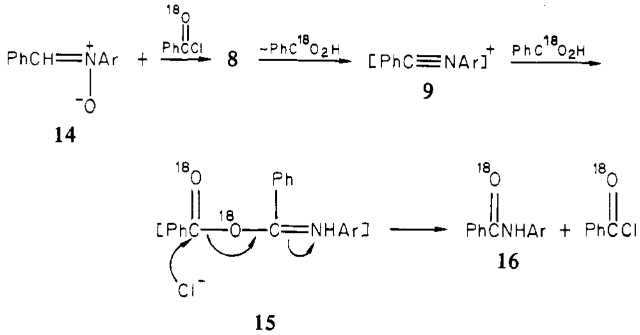
4, A = *p*-$MeOC_6H_4$
6, Ar = *o*-$O_2NC_6H_4$

5, Ar = *p*-$MeOC_6H_4$
7, Ar = *o*-$O_2NC_6H_4$

Scheme I



Scheme II



A mechanism in harmony with these results has the nitrone interacting with the aroyl chloride to form **8**. Intermediate **8** undergoes an elimination reaction to yield the nitrilium ion **9** and the anion **10** in which the ^{18}O label has become scrambled.

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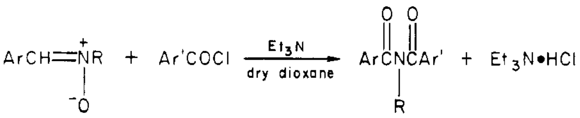
Table I. *N,N*-Diacylamines[a] from the Reaction of Nitrones with Acid Chlorides in Dry Dioxane in the Presence of Triethylamine

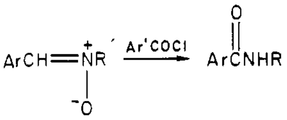


$$\mathrm{ArCH{=}\overset{+}{N}(O^-)R} + \mathrm{Ar'COCl} \xrightarrow[\text{dry dioxane}]{\mathrm{Et_3N}} \mathrm{ArC(O)N(R)C(O)Ar'} + \mathrm{Et_3N{\cdot}HCl}$$

	Ar	R	Ar′	crude % yield	recry[a] mp, °C
1	Ph	Me	Ph	72.4	92–95[11]
2[f]	Ph	*t*-Bu	Ph	81.8	79–80.5[b]
3	Ph	Ph	Ph	82.3	160–162[10]
4	Ph	Ph	*p*-ClC_6H_4	67.4	130–133[10]
5	Ph	Ph	*p*-MeC_6H_4	69.5	160–162[10]
6	Ph	*p*-ClC_6H_4	Ph	45.0	156–157[10]
7	Ph	*p*-MeC_6H_4	Ph	71.4	144–145.5[10]
8[g]	Ph	*p*-MeC_6H_4	*p*-$MeOC_6H_4$	36.2	111–113[b]
9[h]	Ph	*p*-MeC_6H_4	*p*-$O_2NC_6H_4$	87.5	129–131[b,c]
10[i]	Ph	2,6-$(Me)_2C_6H_3$	Ph	90.7	174.5–175.5[b]
11[j]	*p*-ClC_6H_4	*p*-MeC_6H_4	Ph	42.1	118–120.5[b,d]
12[k]	*o*-$O_2NC_6H_4$	*p*-MeC_6H_4	Ph	81.9	181–182[b,e]
13	*p*-$MeOC_6H_4$	*p*-MeC_6H_4	Ph	67.4	111–113[b]

[a] Unless designated otherwise the diacylamines were recrystallized from 95% ethanol. [b] Satisfactory analytical data for C, H, and N were reported for all new compounds listed in the table. [c] Recrystallized from MeOH/$CHCl_3$ (10:1). [d] Recrystallized from MeOH. [e] Recrystallized from *i*-PrOH/$CHCl_3$ (1:1). [f] Anal. Calcd for $C_{18}H_{19}NO_2$: C, 76.85; H, 6.80; N, 4.98. Found: C, 77.15; H, 6.84; N, 4.98. [g] Anal. Calcd for $C_{22}H_{19}NO_3$: C, 76.50; H, 5.56; N, 4.06. Found: C, 76.37; H, 5.57; N, 4.04. [h] Anal. Calcd for $C_{21}H_{16}N_2O_4$: C, 69.99; H, 4.48; N, 7.77. Found: C, 69.85; H, 4.65; N, 7.77. [i] Anal. Calcd for $C_{22}H_{19}NO_2$: C, 80.21; H, 5.81; N, 4.25. Found: C, 80.51; H, 5.58; N, 4.30. [j] Anal. Calcd for $C_{21}H_{16}ClNO_2$: C, 72.10; H, 4.61; N, 4.00. Found: C, 72.25; H, 4.83; N, 4.03. [k] Anal. Calcd for $C_{21}H_{16}N_2O_4$: C, 69.97; H, 4.48; N, 7.77. Found: C, 69.88; H, 4.51; N, 7.79.

Table II. The Isomerization of Nitrones to Amides by Aroyl Chlorides in Anhydrous Solvents



$$\mathrm{ArCH{=}\overset{+}{N}(O^-)R} \xrightarrow{\mathrm{Ar'COCl}} \mathrm{ArC(O)NHR}$$

	Ar	R	Ar′	ratio of nitrone/ ArCOCl	solvent	% yield[a]	mp, °C	
							found	lit.
1	Ph	Me	*p*-$O_2NC_6H_4$	1:1	ether	71	75–77	75–81[13]
2	Ph	*t*-Bu	Ph	1:1	dioxane	86	134–136	135–136[14]
3	Ph	Ph	Ph	1:5	dioxane	71	162–163	163[15]
4	Ph	*p*-ClC_6H_4	*p*-$O_2NC_6H_4$	1:1	ether	91	193–194	192–193[16]
5	Ph	*p*-MeC_6H_4	Ph	1:1	dioxane	82	154–157	156–157[17]
6	Ph	*p*-MeC_6H_4	Ph	1:2	dioxane	86	154–157	156–157[17]
7	Ph	2,6-$(Me)_2C_6H_3$	Ph	1:5	dioxane	87	159–161	161–163[18]
8	*p*-$MeOC_6H_4$	*p*-MeC_6H_4	Ph	1:5	dioxane	60	152–154	155–156[19]
9	*o*-$O_2NC_6H_4$	Ph	Ph	1:5	dioxane	81	152–154	155[20]
10	*o*-$O_2NC_6H_4$	*p*-MeC_6H_4	Ph	1:1	dioxane	72	145–146	147[23]

[a] Crude yield.

Addition of **10** to the carbon of **9** generates **11** which is the recognized precursor for the Mumm rearrangement.[8–12] The Mumm rearrangement of **11** via the transition state **12** affords the diacylamine **13** (Scheme I).

If the triethylamine is omitted from the reactions between the nitrones and aroyl chlorides then all the nitrones listed in Table I isomerize to the corresponding amides (Table II) instead of forming *N,N*-diacylamines. Furthermore, treatment of α-phenyl-*N*-(p-tolyl)nitrone (**14**) with 3, 7, 10, and 14 equiv. of [^{18}O]benzoyl chloride in dry dioxane gave in each instance *N*-(*p*-tolyl)benzamide with 50% ^{18}O enrichment. The [^{18}O]benzoyl chloride was employed in excess to ensure that all of the nitrone was converted to **8** initially. The isomerization then proceeds by elimination of benzoic acid from **8** to give the nitrilium ion **9** Addition of benzoic acid (rather than the benzoate anion as was the case in the reactions with triethylamine) to **9** gives **15** (Scheme II) which cannot undergo the Mumm rearrangement since the nitrogen is protonated. As a consequence the chloride ion attacks the carbonyl carbon of **15** to produce the isomerized amide **16** with 50% ^{18}O enrichment and the aroyl chloride (Scheme II).

Hitherto the isomerization of nitrones to amides by acyl halides was thought to proceed via 3- and 5-membered ring intermediates or transition states, e.g. **17** and/or **18**.[4,5] The isotopic labeling experiments described above for the formation of the diacylamines and the isomerized amides render the formation of **17** and **18**

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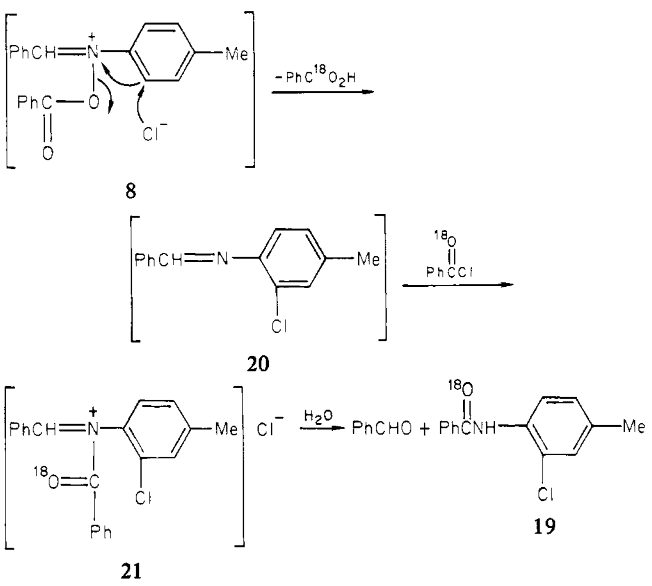
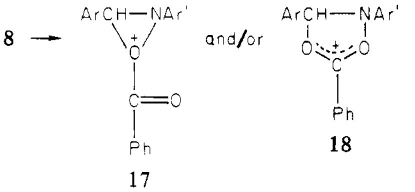
Scheme III



unlikely. The presumption that the isomerization may be occurring simultaneously through both **17** and **18** also appears untenable.[5] Both the isomerization of nitrones into amides and their conversion into diacylamines have mechanistic similarities to the Polonovski and Pummerer reactions.



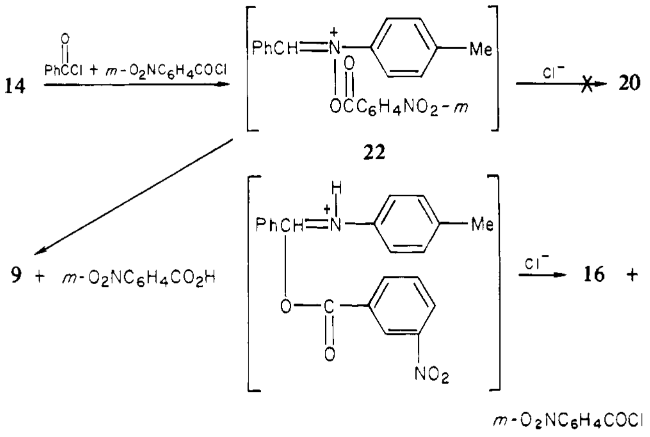
Combined gas chromatography–mass spectrometry demonstrated that the *N*-(*p*-tolyl)benzamide (**16**) isolated from the isomerization of **14** by employing an excess amount of [^{18}O]benzoyl chloride was contaminated with 5–7% of what appeared to be *N*-(2-chloro-4-methylphenyl)benzamide (**19**), the oxygen of which was labeled to the same extent as the [^{18}O]benzoyl chloride. An authentic sample of unlabeled **19** was prepared by the benzoylation of 2-chloro-4-methylaniline. Comparison of the melting points and gas chromatographic, infrared, and mass spectrometric properties of this reference compound with those of the labeled **19** confirmed the proposed structure of the latter. The formation of **19** can be explained by an attack of chloride ion at the ortho position of the *N*-*p*-tolyl group of **8** (Ar′ = *p*-MeC_6H_4) to give the anil **20**. Reaction of **20** with the excess [^{18}O]benzoyl chloride produces **21** which upon intervention of atmospheric moisture during the workup yields **19** (Scheme III). The process leading to **19** competes with that of **8** forming a nitrilium ion.

The ratio of **19** to **16** rose dramatically from 1:15 to 1:1 when a mixture of 1 equiv of α-phenyl-*N*-(*p*-tolyl)nitrone and 10 equiv of benzoic acid is treated with 10 equiv of [^{18}O]benzoyl chloride. We attribute this result to an increase in chloride ion concentration that is formed by a reaction of benzoic acid with benzoyl chloride to give hydrogen chloride and benzoic anhydride.

A control run of *p*-$O_2NC_6H_4CH{=}NC_6H_4Me$-*p* with benzoyl chloride in dry dioxane followed by the evaporation of the solvent gave **16** (84%) and *p*-nitrobenzaldehyde which indicated that interaction of **20** with benzoyl chloride as shown in Scheme III could produce **19**.

The side reaction leading to **19** during the isomerization of α-phenyl-*N*-(*p*-tolyl)nitrone (**14**) to *N*-(*p*-tolyl)benzamide was also observed when α,*N*-diphenylnitrone (197 mg, 1 mmol) was treated with benzoyl chloride (560 mg, 4 mmol) in dry dioxane (6 mL). The crude material (155 mg) isolated was shown by gas chromatography–mass spectrometry to be a mixture of benzanilide (97%), *N*-(2-chlorophenyl)benzamide (1%), and *N*-(4-chlorophenyl)benzamide (1.3%).

Scheme IV



Interestingly, when the isomerization of α-phenyl-*N*-(*p*-tolyl)nitrone was carried out in the presence of [^{18}O]benzoyl chloride and *m*-nitrobenzoyl chloride in a ratio of 1:2:2 in dry dioxane the isolated *N*-(*p*-tolyl)benzamide showed no enrichment with ^{18}O. This result suggests that the nitrone reacts exclusively with the more electrophilic *m*-nitrobenzoyl chloride to form **22** (Scheme IV). Further, the *N*-(*p*-tolyl)benzamide isolated was not contaminated with any **19** as was the case when benzoyl chloride was the sole isomerizing agent. This means that **22** bears such a good nucleofuge (e.g., the *m*-nitrobenzoyloxy group) that the elimination reaction to give the nitrilium ion **9** is much faster than the attack on **22** by chloride ion to give the imine **20** (Scheme IV).

Experimental Section

Diacylamines (Table I). To a stirred solution of 1 mmol of a nitrone in 8 mL of dry dioxane containing 1 mmol of triethylamine was added 1 mmol of an aroyl chloride. Triethylamine hydrochloride began to precipitate within minutes. The reaction mixture was stirred for 8 h, the triethylamine hydrochloride was removed by filtration, and the solvent was evaporated. The crude diacylamine was slurried with 3 mL of cold Et_2O and was filtered. In the cases of *N*,*N*-dibenzoylmethylamine and *N*,*N*-dibenzoyl-*tert*-butylamine (entries 1 and 2 in Table I) the crude diacylamines were triturated with ether, the ether was evaporated, and the residues were slurried with 2 mL of cold methanol and filtered.

Reactions of α-(*p*-Anisyl)- and α-(*o*-Nitrophenyl)-*N*-(*p*-tolyl)nitrones with [^{18}O]Benzoyl Chloride. Employing the same procedure described for the preparation of diacylamines 1 mmol of α-(*p*-anisyl)- or α-(*o*-nitrophenyl)-*N*-(*p*-tolyl)nitrone was reacted with 1 mmol of [^{18}O]benzoyl chloride[21] (92% enriched with ^{18}O). The mass spectra of the isolated *N*-anisoyl-*N*-benzoyl-*p*-toluidine and *N*-(*o*-nitrobenzoyl)-*N*-benzoyl-*p*-toluidine showed that both aroyl groups of each diacylamine contained 50% of the ^{18}O label.

Isomerization of Nitrones (Table II). A 1-mmol sample of the nitrone was dissolved in 6 mL of either dry ether or dry dioxane. The solution was stirred, and the aroyl chloride was added all at once in the quantities designated in Table II. The reaction mixture was stirred for 7–8 h, and the solvent was evaporated. The crude amide was slurried with 2 mL of ether and was filtered. *N*-Methylbenzamide and *N*-(*tert*-butyl)benzamide (entries 1 and 2 in Table II) were recrystallized from petroleum ether (bp 65–73 °C) while all the other listed amides were recrystallized from 95% ethanol.

Isomerization of α-Phenyl-*N*-(*p*-tolyl)nitrone (14) with [^{18}O]Benzoyl Chloride. A 1-mmol sample of **14** was dissolved in 6 mL of dry dioxane. To this solution were added 3, 7, 10, and 14 mmol of [^{18}O]benzoyl chloride (92% ^{18}O enriched). The mixtures were stirred for 6 h, and the solvent was evaporated. The residues were triturated with 2 mL of ether and filtered. Gas chromatography–mass spectroscopy of the filtered material revealed that the *N*-(*p*-tolyl)benzamide (**16**) contained 50, 52, 50, and 55% of the ^{18}O label, respectively, and that in each case there was present about 7% *N*-(2-chloro-4-methylphenyl)benzamide (**19**) in which 100% of the ^{18}O label had been incorporated.

Isomerization of 14 by [^{18}O]Benzoyl Chloride in the Presence of Benzoic Acid. To a stirred mixture of benzoic acid (1.221 g, 10 mmol) and **14** (211 mg, 1 mmol) in 6 mL of dry dioxane was added [^{18}O]benzoyl chloride (1.42 g, 10 mmol) all at once. After 14 h the solvent was evaporated and the crude residue dissolved in 60 mL of ether. The ethereal solution was washed with four 30-mL portions of saturated sodium bicarbonate solution. The ether was evaporated, and the 237 mg of crude residue was recrystallized once from ethanol. Gas chromatography indicated that the recrystallized material (mp 115–119 °C) was composed of 55% of **19** annd 45% of **16**. The former compound incorporated 100% of the ^{18}O label while **16** incorporated 50% of the ^{18}O label. A sample of the crude residue (140 mg) was dissolved in 2 mL of chloroform, and the solution was eluted on a column of silica gel (60–200 mesh) with a 3:1 solution of petroleum ether (bp 60–110 °C) and ether. Compound **19** 65 mg, 27%) was found in the first 60 mL of eluent. The eluent was gradually changed to ether, and **16** (50 mg, 24%) was obtained after an additional 400 mL of eluent had passed through the column. Compound **16** melted at 154–157 °C (lit.[17] 156–157 °C) and compound **19** melted at 135–136 °C. Recrystallization from 95% ethanol gave **19**, mp 137–139 °C.

Anal. Calcd for $C_{14}H_{12}ClNO$: C, 68.41; H, 4.92; N, 5.70. Found: C, 68.62; H, 5.09 N, 5.70.

Isomerization of 14 by a Mixture of [^{18}O]Benzoyl Chloride and *m*-Nitrobenzoyl Chloride. A mixture of [^{18}O]benzoyl chloride (284 mg, 2 mmol) and *m*-nitrobenzoyl chloride (371 mg, 2 mmol) was added to a solution of α-phenyl-*N*-(*p*-tolyl)nitrone (211 mg) in 6 mL of dry dioxane. The reaction mixture was stirred for 8 h and the solvent evaporated. Trituration of the crude residue with 2 mL of ether gave **16** (160 mg, 76%), mp 151–154 °C. Recrystallization from ethanol gave 122 mg of **16**, mp 154–156 °C (lit.[17] 156–157 °C). Mass spectrometry of **16** showed the absence of any ^{18}O label.

Gas Chromatography-Mass Spectrometry (LKB 9000 Instrument). Gas chromatographic conditions included: 1.7 m × 4 mm glass spiral column packed with 3% OV-1; oven temperature, 140 °C, flow rate, 30 mL of helium/min. Mass spectrometric conditions included: source temperature, 250 °C; electron energy, 70 eV; accelerating potential, 3.5 kV; trap current, 60 μA. The extent of ^{18}O label was established by comparing the intensities of the appropriate ions (i.e., M and M + 2) from the unlabeled reference compounds with those of the labeled compounds. Averages of three to five scans were employed for determination of ion isotope intensities.

Nitrones. The nitrones were prepared by the reaction of N-substituted hydroxylamines with aldehydes according to literature procedures.[22] Melting points and elemental analyses of new nitrones (or of nitrones mentioned in the literature but uncharacterized) are given below. These nitrones were recrystallized from 95% ethanol except for α-phenyl-*N*-(2,6-dimethylphenyl)nitrone which was recrystallized from cyclohexane.

α-(*p*-Methoxyphenyl)-*N*-(*p*-tolyl)nitrone: mp 127–129 °C. Anal. Calcd for $C_{15}H_{15}NO_2$: C, 74.65; H, 6.26; N, 5.80. Found: C, 74.69; H, 6.40; N, 5.90.

α-(*p*-Chlorophenyl)-*N*-(*p*-tolyl)nitrone: mp 165–167 °C. Anal. Calcd for $C_{14}H_{12}ClNO$: C, 68.41; H, 4.92; N, 5.70. Found: C, 68.72; H, 4.91; N, 5.81.

α-Phenyl-*N*-(2,6-dimethylphenyl)nitrone: mp 127–128 °C. Anal. Calcd for $C_{15}H_{15}NO$: C, 79.98; H, 6.71; N, 6.21. Found: C, 80.19; H, 6.88; N, 6.29.

Formation of 16 from the Reaction of Benzoyl Chloride and *N*-(*p*-Nitrobenzylidene)-*p*-toluidine (23). To a solution of **23** (240 mg, 1 mmol) in 6 mL of dry dioxane was added benzoyl chloride (140 mg, 1 mmol). The mixture was stirred for 12 h. Evaporation of the solvent and trituration of the residue with ether followed by filtration gave **16** (168, mg, 80%), melting at 152–155 °C (lit.[17] 154–157 °C). The ether filtrate was evaporated to dryness and the residue dissolved in ethanol. Addition of 2,4-dinitrophenylhydrazine gave the 2,4-dinitrophenylhydrazone of *p*-nitrobenzaldehyde.

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Registry No. 1 (R = Me), 3376-23-6; **1** (R = *t*-Bu), 3376-24-7; **1** (R = Ph), 1137-96-8; **1** (R = *p*-ClC_6H_4), 19865-58-8; **1** (R = *p*-MeC_6H_4), 19064-77-8; **1** (R = 2,6-$(Me)_2C_6H_3$), 33008-36-5; **2** (R = Me), 613-93-4; **2** (R = *t*-Bu), 5894-65-5; **2** (R = Ph), 93-98-1; **2** (R = *p*-ClC_6H_4), 2866-82-2; **2** (R = *p*-MeC_6H_4), 582-78-5; **2** (R = 2,6-$(Me)_2C_6H_3$), 18109-39-2; **4** (Ar = *p*-$MeOC_6H_4$), 52764-31-5; **6** (Ar = *o*-$O_2NC_6H_4$), 81724-80-3; α-*p*-chlorophenyl-*N*-(*p*-methylphenyl)nitrone, 37056-74-9; α-(*o*-nitrophenyl)-*N*-phenylnitrone, 53548-19-9; *N*,*N*-dibenzoylmethylamine, 23825-32-3; *N*,*N*-dibenzoyl-*tert*-butylamine, 81724-81-4; *N*,*N*-dibenzoylphenylamine, 3027-01-8; *N*-benzoyl-*N*-(*p*-chlorobenzoyl)phenylamine, 34916-19-3; *N*-benzoyl-*N*-(*p*-methylbenzoyl)phenylamine, 61582-60-3; *N*,*N*-dibenzoyl(*p*-chlorophenyl)amine, 77930-23-5; *N*,*N*-dibenzoyl(*p*-methylphenyl)amine, 77930-24-6; *N*-benzoyl-*N*-(*p*-methoxybenzoyl)-*p*-methylphenylamine, 81724-82-5; *N*-benzoyl-*N*-(*p*-nitrobenzoyl)-*p*-methylphenylamine, 81724-83-6; *N*,*N*-dibenzoyl(2,6-dimethylphenyl)amine, 81724-84-7; *N*-benzoyl-*N*-(*p*-chlorobenzoyl)-*p*-methylphenylamine, 81724-85-8; *N*-benzoyl-*N*-(*o*-nitrobenzoyl)-*p*-methylphenylamine, 81724-86-9; *N*-benzoyl-*N*-(*p*-methoxybenzoyl)-*p*-methylphenylamine, 81724-82-5; *N*-(*p*-methylphenyl)-*p*-methoxybenzamide, 39192-94-4; *N*-phenyl-*o*-nitrobenzamide, 2385-27-5; *N*-(*p*-methylphenyl)-*o*-nitrobenzamide, 50623-00-2; benzoyl chloride, 98-88-4; *p*-chlorobenzoyl chloride, 122-01-0; *p*-methylbenzoyl chloride, 874-60-2; *p*-methoxybenzoyl chloride, 100-07-2; *p*-nitrobenzoyl chloride, 122-04-3.