

Reaction Pathways for Arylcarbamoyl Radicals and the Cyclization of *o*-Substituted Phenylcarbamoyl Radicals

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N-Arylcarbamoyl radicals generated from *N*-arylformamides with di-*t*-butyl peroxide (Bu^tOOBu^t) in chlorobenzene at 110 °C give rise to a series of reactions depending on the nature and the position of the substituent in the aromatic ring. When the aryl group is phenyl or *p*-chlorophenyl the following reactions on carbamoyl radicals occur: (i) loss of hydrogen with formation of aryl isocyanates, whereas the loss of carbon monoxide leads to arylamines *via* arylaminyl radicals; (ii) dimerization to *NN'*-diaryloxamides; (iii) aromatic substitution on chlorobenzene leading to *N*-arylbenzamides (*ipso*-substitution) and *N*-arylchlorobenzamides. The isocyanates trapped by *t*-butyl alcohol and arylamines give *t*-butyl *N*-arylcarbamates and *NN'*-diarylureas. With *o*-substituted *N*-phenylformamides intramolecular cyclization is observed as well; in fact, the *N*-(*o*-cyano-phenyl)formamide affords isatin *via* addition of the carbamoyl radical to the cyano-group while the *N*-[(*o*-phenylthio)phenyl]formamide gives 2-benzothiazolone and phenyl radicals through an $S_{\text{H}}i$ reaction on the sulphur atom; finally 2-formamidobiphenyls furnish phenanthridones in very high yields.

CARBAMOYL radicals generated by hydrogen abstraction from formamides have been used in some interesting organic syntheses, *i.e.* the amidation of olefins¹ and hetero-aromatic bases.² E.s.r. studies have been carried out on various carbamoyl radicals; in all cases σ -type radicals have been detected.³

The results we report in this paper throw further light on their reactivity and show carbamoyl radicals to offer a convenient approach to the synthesis of phenanthridones as well. Carbamoyl radicals (2), generated by treatment of *N*-arylformamides (1) with di-*t*-butyl peroxide (Bu^tOOBu^t) in chlorobenzene at 110 °C, undergo a series of reactions according to the nature and position of the substituent in the aromatic ring.

RESULTS AND DISCUSSION

If the aryl group is phenyl or *p*-chlorophenyl the following reactions take place: (i) loss of a hydrogen atom with formation of the aryl isocyanates (3) and loss of carbon monoxide leading to the arylaminyl radicals (4) [which give rise to the arylamines (5) by hydrogen abstraction]; (ii) dimerization to give the *NN'*-diaryloxamides (6) in low yields; (iii) *ipso*- and normal aromatic substitution on chlorobenzene which lead to the *N*-arylbenzamides (7) and *N*-arylchlorobenzamides (8), respectively (Scheme 1).

We have not been able to isolate the isocyanates (3) or detect their presence during the reaction by i.r. spectral analysis of the reaction mixtures; probably isocyanates are immediately trapped both by *t*-butyl alcohol, arising from Bu^tOOBu^t , and arylamines (5) giving the *t*-butyl *N*-arylcarbamates (9) and *NN'*-diarylureas (10). When the reaction was carried out in an excess of *n*-butanol, *n*-butyl *N*-arylcarbamates were obtained, together with trace amounts of the other compounds mentioned above, and no trace of (9) and (10) was observed. In the cases mentioned above, loss of hydrogen, leading to isocyanates, seems to be the most important reaction undergone by *N*-arylcarbamoyl radicals. This trend is supported

by the fact that compounds (9) and (10) were the main reaction products; in addition, *NN*-diphenylformamide under the same conditions led to small amounts of *NN*-diphenylamine (4%) and tetraphenylhydrazine (5%), as well as unreacted material (88%).

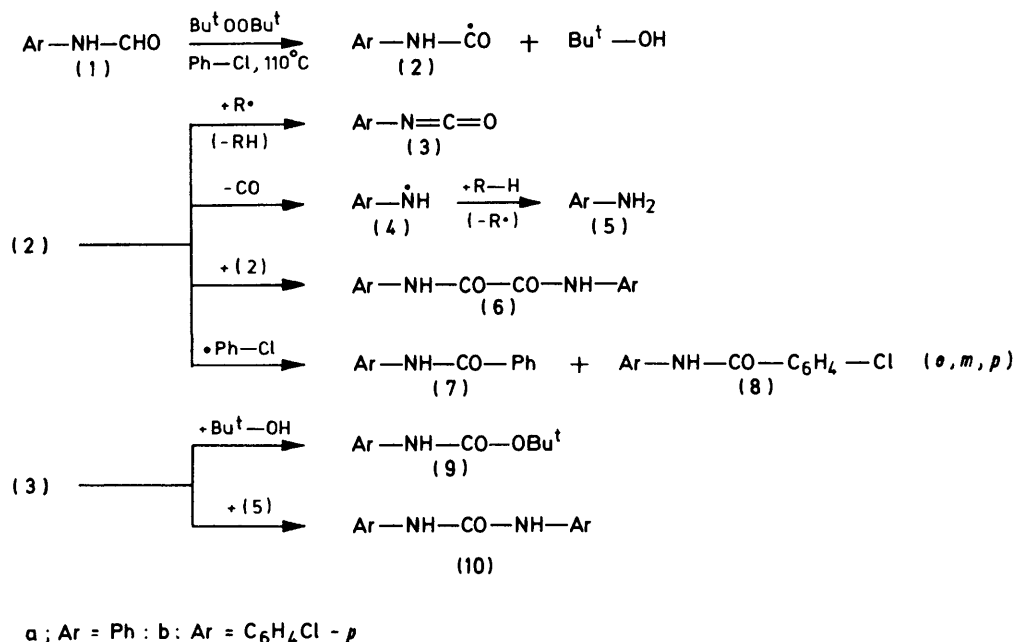
The same reaction carried out with the *N*-(*o*-cyano-phenyl)formamide furnished isatin, probably through intramolecular addition of the intermediate carbamoyl radical to the carbon atom of the cyano-group, leading to isatin- β -imide followed by hydrolysis during separation. No evidence of any product from intramolecular attack on the nitrogen atom was observed. Since our primary aim was to investigate the possibility of carbamoyl addition to the cyano-group, no systematic attempts were made to identify the other reaction products. When the *N*-[(*o*-phenylthio)phenyl]formamide (11) was allowed to react with Bu^tOOBu^t in chlorobenzene or anisole at 110 °C, 2-benzothiazolone was obtained together with the isomeric chloro- and methoxybiphenyls, respectively; in both cases g.l.c. analysis showed the isomer ratio to be similar to that obtained in the phenylation of chlorobenzene or anisole with dibenzoyl peroxide⁴ (Table 1).

TABLE 1

Ph. source	Chlorobiphenyls			Methoxybiphenyls		
	<i>o</i> (%)	<i>m</i> (%)	<i>p</i> (%)	<i>o</i> (%)	<i>m</i> (%)	<i>p</i> (%)
(11) + Bu^tOOBu^t	54	29	17	67	17	16
(PhCO_2) ₂	50	32	18	69.8	14.7	15.6

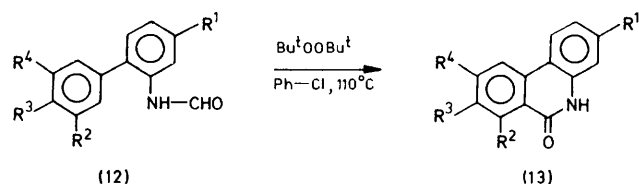
The formation of 2-benzothiazolone might be accounted for in terms of intramolecular homolytic substitution of the carbamoyl radical on the sulphur atom of the phenylthio-group, with expulsion of a phenyl radical which reacts with the solvent to furnish the biphenyl derivatives. This behaviour represents one of very few examples of $S_{\text{H}}i$ reaction on the sulphur atom of organic sulphides.⁵

In the same conditions the 2-formamidobiphenyls (12)



SCHEME 1

furnished the phenanthridones (13) in high yields (Scheme 2, Table 2).



SCHEME 2

With respect to previously reported syntheses of phenanthridones,⁶ we think that this reaction might offer a simple and convenient approach to phenanthridones, particularly when 2-formamidobiphenyls are

TABLE 2

Starting material	R ¹	R ²	R ³	R ⁴	Yield of (13) (%)
(12a)	H	H	H	H	83
(12b)	Cl	H	H	H	85
(12c)	MeO	H	H	H	90
(12d)	H	H	NO ₂	H	55
(12e)	H	Me	H	Me	95

easily accessible. The 2'-X-2-formamidobiphenyls (X = I or NO₂) gave mixtures of (13a) and 10-X-phenanthridone. The formation of (13a) may be reasonably explained in terms of intramolecular *ipso*-substitution of the corresponding carbamoyl radical, whereas a normal intramolecular substitution gives rise to the substituted phenanthridone. When X is iodine *ipso*-substitution is the main reaction; however, when X is a nitro-group normal substitution prevails over the *ipso*-reaction.

EXPERIMENTAL

N-Phenylformamide⁷ (1a), *N*-(*p*-chlorophenyl)formamide⁸ (1b), *NN*-diphenylformamide,⁹ *N*-[(*o*-phenylthio)-

phenyl]formamide¹⁰ (11), 2-formamidobiphenyl¹¹ (12a), 4'-nitro-2-formamidobiphenyl¹² (12d) and 2'-nitro-2-formamidobiphenyl¹³ (14b) were prepared according to the literature.

The reaction products *t*-butyl *N*-phenylcarbamate¹⁴ (9a), *t*-butyl *N*-(*p*-chlorophenyl)carbamate¹⁵ (9b), *n*-butyl *N*-(*p*-chlorophenyl)carbamate,¹⁶ *NN'*-diphenylurea¹⁷ (10a), *NN'*-(4,4'-dichlorodiphenyl)urea¹⁸ (10b), *NN'*-diphenyloxamide¹⁹ (6a), *NN'*-(4,4'-dichlorodiphenyl)oxamide²⁰ (6b), *N*-phenylbenzamide²¹ (7a), *N*-(*p*-chlorophenyl)benzamide²² (7b), *N*-phenyl-*o*-chlorobenzamide²³ (8a), *N*-phenyl-*m*-chlorobenzamide²⁴ (8a), *N*-phenyl-*p*-chlorobenzamide²⁵ (8a), *N*-(*p*-chlorophenyl)-*o*-chlorobenzamide²⁶ (8b), *N*-(*p*-chlorophenyl)-*m*-chlorobenzamide²⁶ (8b), *N*-(*p*-chlorophenyl)-*p*-chlorobenzamide²⁶ (8b), tetraphenylhydrazine,²⁷ 2-benzothiazolone,²⁸ *o*-, *m*-, and *p*-chlorobiphenyls, *o*-, *m*-, and *p*-methoxybiphenyls, phenanthridone²⁹ (13a), 3-chlorophenanthridone³⁰ (13b), 3-methoxyphenanthridone³⁰ (13c), 8-nitrophenanthridone³¹ (13d), and 10-nitrophenanthridone³¹ (15b) were identified by mixed m.p. determination and spectral data comparison (i.r. and mass spectroscopy) with authentic specimens which were prepared as described in the literature or were commercially available.

All melting points were uncorrected. G.l.c. analyses were performed on a Varian 1400 instrument. I.r. and mass spectra were recorded on a Perkin-Elmer 257 instrument and a JEOL JMS-D 100 mass spectrometer respectively.

N-(*o*-Cyanophenyl)formamide.—To *N*-(*o*-bromophenyl)formamide³² (5 g, 25 mmol) in DMF (50 ml), copper(I) cyanide (4.9 g, 32 mmol) was added; the mixture was refluxed for 1 h and then poured into water. After extraction with chloroform, the organic layer was washed with water and dried. The solvent was removed under vacuum and the residue crystallized from light petroleum (b.p. 70–120 °C) to give the title compound (2.9 g), m.p. 131–132 °C (lit.,³³ m.p. 130.5–131.5 °C).

General Method for the Preparation of 2-Formamidobiphenyls.—The corresponding amine (50 mmol) was refluxed for 2 h in 99% formic acid (50 ml); evaporation of the excess

of acid under reduced pressure left a residue which was crystallized from light petroleum (b.p. 70–120 °C) affording the formamido-derivative in almost quantitative yields (95–98%). The following formamidobiphenyls were prepared.

2-Formamido-4-chlorobiphenyl (12b) from 2-amino-4-chlorobiphenyl,³⁰ m.p. 93–94 °C; *m/e* 231 (M^+), 203, 202, and 167; ν_{\max} (CHCl₃) 3 410 (NH stretch) and 1 700 cm⁻¹ (CO) (Found: C, 67.4; H, 4.3; Cl, 15.2; N, 6.15. C₁₃H₁₀ClNO requires C, 67.39; H, 4.35; Cl, 15.30; N, 6.04%).

2-Formamido-4-methoxybiphenyl (12c) from 2-amino-4-methoxybiphenyl,³⁰ m.p. 68–69 °C; *m/e* 227 (M^+), 199, 198, 168, and 167; ν_{\max} (CHCl₃) 3 410 (NH stretch) and 1 700 cm⁻¹ (CO) (Found: C, 74.0; H, 5.75; N, 6.25. C₁₄H₁₃NO₂ requires C, 73.98; H, 5.76; N, 6.16%).

2-Formamido-3',5'-dimethylbiphenyl (12e) from 2-amino-3',5'-dimethylbiphenyl,³⁴ m.p. 100–102 °C; *m/e* 225 (M^+), 197, 196, 182, and 167; ν_{\max} (CHCl₃) 3 410 (NH stretch) and 1 700 cm⁻¹ (CO) (Found: C, 79.95; H, 6.75; N, 6.25. C₁₅H₁₅NO requires C, 79.97; H, 6.71; N, 6.22%).

2-Formamido-2'-iodobiphenyl from 2-amino-2'-iodobiphenyl,³⁵ m.p. 106–108 °C; *m/e* 323 (M^+), 196, 168, and 167; ν_{\max} (CHCl₃) 3 410 (NH stretch) and 1 700 cm⁻¹ (CO) (Found: C, 48.35; H, 3.10; I, 39.15; N, 4.4. C₁₃H₁₀INO requires C, 48.31; H, 3.12; I, 39.27; N, 4.33%).

General Procedure for the Reactions of N-Arylformamides with Bu^tOObu^t.—To the formamido-derivative (10 mmol) in chlorobenzene (60 ml), was added Bu^tOObu^t (50 mmol) and the solution was maintained at 110 °C for 48 h. The solvent was removed under vacuum; after column chromatography on silica gel (Kieselgel 60, 70–230 mesh ASTM, Merck) of the residue, the products were isolated in the reported yields using light petroleum–diethyl ether gradient as eluant. During the reaction, evolution of carbon monoxide was proved by collecting it in a saturated palladium(II) chloride solution, which became black due to the formation of metallic palladium.

Reaction of N-Phenylformamide (1a) with Bu^tOObu^t.—Using the procedure described above, the residue was chromatographed on silica gel (80 g). Elution with light petroleum–diethyl ether (95 : 5 v/v) gave *t*-butyl *N*-phenylcarbamate (390 mg, 20%) and aniline (28 mg, 3%). Further elution with increasing amounts of diethyl ether provided unreacted starting material (220 mg, 18%), *N*-phenylbenzamide (60 mg, 3%), *N*-phenyl-*o*-chlorobenzamide (70 mg, 3%), *N*-phenyl-*m*-chlorobenzamide (70 mg, 3%), *N*-phenyl-*p*-chlorobenzamide (93 mg, 4%), *NN'*-diphenyloxamide (72 mg, 3%) and *NN'*-diphenylurea (530 mg, 25%).

Reaction of N-(*p*-Chlorophenyl)formamide (1b) with Bu^tOObu^t.—Using the procedure described above, the residue was chromatographed on silica gel (100 g). Elution with light petroleum–diethyl ether (95 : 5 v/v) gave *t*-butyl *N*-(*p*-chlorophenyl)carbamate (520 mg, 23%) and *p*-chloroaniline (38 mg, 3%). Further elution with increasing amounts of diethyl ether provided unreacted starting material (220 mg, 14%), *N*-(*p*-chlorophenyl)-benzamide (93 mg, 4%), *N*-(*p*-chlorophenyl)-*o*-chlorobenzamide (110 mg, 4%), *N*-(*p*-chlorophenyl)-*m*-chlorobenzamide (80 mg, 3%), *N*-(*p*-chlorophenyl)-*p*-chlorobenzamide (130 mg, 5%), *NN'*-(4,4'-dichlorodiphenyl)oxamide (62 mg, 2%) and *NN'*-(4,4'-dichlorodiphenyl)urea (760 mg, 27%).

Reaction of N-(*p*-Chlorophenyl)formamide (1b) with Bu^tOObu^t in an Excess of *n*-Butanol.—When the reaction

was carried out in the presence of an excess of *n*-butanol (100 mmol), the main reaction product was *n*-butyl *N*-(*p*-chlorophenyl)carbamate (1.25 g, 55%), with trace amounts of the above compounds.

Reaction of NN-Diphenylformamide with Bu^tOObu^t.—Using the procedure described above, the residue was chromatographed on silica gel (80 g). Elution with light petroleum–diethyl ether (95 : 5 v/v) gave tetraphenylhydrazine (168 mg, 5%). Further elution with increasing amounts of diethyl ether provided diphenylamine (68 mg, 4%) and unreacted starting material (1.74 g, 88%).

Reaction of N-(*o*-Cyanophenyl)formamide with Bu^tOObu^t.—Using the procedure described above, the residue was chromatographed on silica gel (100 g). Elution with light petroleum–diethyl ether (90 : 10 v/v) gave isatin (370 mg, 25%), and other unidentified products. No presence of quinazoline-2,4(1*H*,3*H*)-dione or quinazolin-2(1*H*)-one was observed.

Reaction of N-[(*o*-Phenylthio)phenyl]formamide (11) with Bu^tOObu^t.—(i) *In chlorobenzene.* Using the procedure described above the residue was chromatographed on silica gel (100 g). Elution with light petroleum gave the isomeric chlorobiphenyls (470 mg, 25%). Further elution with light petroleum–diethyl ether (90 : 10 v/v) provided 2-benzothiazolone (460 mg, 30%).

(ii) *In anisole.* Using the procedure described above the residue chromatographed as described for the reaction in chlorobenzene gave the isomeric methoxybiphenyls (470 mg, 25%) and 2-benzothiazolone (470 mg, 30%).

In both cases, g.l.c. (5% FFAP on Chromosorb W) of the biphenyl fraction showed the isomer ratio reported in Table 1.

General Procedure for the Preparation of Phenanthridones.—To 2-formamidobiphenyl (10 mmol) in chlorobenzene (60 ml) was added Bu^tOObu^t (50 mmol); the solution was kept at 110 °C for 48 h and then cooled in an ice-bath. The precipitated phenanthridone was collected by filtration on a sintered glass funnel. Recrystallization from Me₂SO gave very pure phenanthridone. From the mother-liquors, the solvent was removed under vacuum; column chromatography on silica gel of the residue recovered only small amounts of phenanthridone, eluting with diethyl ether. The following phenanthridones were prepared and in each case the reported amounts are referred to the product actually isolated by crystallization from the reaction mixture.

Phenanthridone (13a). From 2-formamidobiphenyl (12a) (1.97 g), phenanthridone (1.6 g) was obtained; m.p. 290–292 °C; *m/e* 195 (M^+) and 167; ν_{\max} (CHCl₃) 3 400 (NH stretch) and 1 670 cm⁻¹ (CO).

3-Chlorophenanthridone (13b). From 4-chloro-2-formamidobiphenyl (12b) (2.31 g), 3-chlorophenanthridone (1.95 g) was obtained; m.p. 295–297 °C; *m/e* 229 (M^+), 201, and 167; ν_{\max} (CHCl₃) 3 400 (NH stretch) and 1 670 cm⁻¹ (CO).

3-Methoxyphenanthridone (13c). From 2-formamido-4-methoxybiphenyl (12c) (2.29 g), 3-methoxyphenanthridone (2.0 g) was obtained; m.p. 248–250 °C; *m/e* 225 (M^+) and 182; ν_{\max} (CHCl₃) 3 400 (NH stretch) and 1 670 cm⁻¹ (CO).

8-Nitrophenanthridone (13d). From 2-formamido-4'-nitrobiphenyl (12d) (2.42 g), 8-nitrophenanthridone (1.3 g) was obtained; m.p. 320–322 °C; *m/e* 240 (M^+), 210, 194, 182, and 166; ν_{\max} (CHCl₃) 3 400 (NH stretch) and 1 680 cm⁻¹ (CO).

7,9-Dimethylphenanthridone (13e). From 2-formamido-

3',5'-dimethylbiphenyl (12e) (2.25 g), 7,9-dimethylphenanthridone (1.78 g) was obtained; m.p. 277—278 °C; m/e 223 (M^+), 208, and 178; ν_{\max} (CHCl_3) 3 400 (NH stretch) and 1 660 cm^{-1} (CO stretching) (Found: C, 80.65; H, 5.85; N, 6.35. $\text{C}_{15}\text{H}_{13}\text{NO}$ requires C, 80.69; H, 5.87; N, 6.27%). The residue obtained by removing the solvent from the mother-liquors provided starting material (0.35 g) after column chromatography on silica gel (80 g) by elution with light petroleum-diethyl ether (1 : 1 v/v).

Reaction of 2-formamido-2'-iodobiphenyl with Bu^tOOBu^t .—Under the same conditions described for the general procedure for the preparation of phenanthridones, from 2-formamido-2'-iodobiphenyl (3.23 g) the parent phenanthridone (13a) (2.9 g) was obtained.

Reaction of 2-formamido-2'-nitrobiphenyl with Bu^tOOBu^t .—Under the conditions described above, from 2-formamido-2'-nitrobiphenyl (2.42 g) as well as the parent phenanthridone (13a) (0.15 g), 10-nitrophenanthridone (1.3 g) was obtained; m.p. 315—317 °C; m/e 240 (M^+), 210, 182, and 166; ν_{\max} (CHCl_3) 3 400 (NH stretch) and 1 680 cm^{-1} (CO).

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