# Ring–Chain Tautomerism of Some *o*-Benzoylbenzanilides

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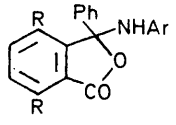
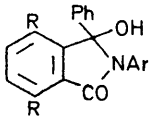
Interconversions of ring–chain tautomers of *o*-benzoylbenzanilide are described, but one of the three structures is observed only in solution. *N*-*o*-Biphenylylphthalimidines have been prepared, some of them by reduction of ring–chain tautomers of *o*-benzamidobiphenyl derivatives. Structural assignments are based mainly on i.r. spectroscopic evidence.

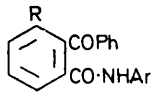
A COMPOUND at first formulated as the 3-hydroxyphthalimidine (1b),[1] then as *o*-carboxybenzophenone anil (on polarographic evidence),[2] and more recently as the 3-anilinophthalide (1a) (from i.r. spectra),[3] is reduced by zinc and acetic acid to 1,2-diphenylphthalimidine (14),[1] which we required in the course of preparation of 1,2,3-triphenylisoindole [4] for studies of its autoxidation.[5]




(a)




(b)




(c)

(1) R = H, Ar = Ph
(2) R = H, Ar = *o* – $PhC_6H_4$
(3) R = Me, Ar = *o* – $PhC_6H_4$

We thus followed other workers [1-3,6-11] into the field of ring–chain tautomerism of derivatives of *o*-benzoylbenzoic acid, and report our observations on some *o*-benzoylbenzanilides.

The ψ-chloride (4) from *o*-benzoylbenzoic acid reacts with aniline to give mixtures of open-chain and cyclic anilides;[1] the lactone (1a) is the major product in chloroform or dimethylformamide as solvent.[3] We also obtained (1a) from the same reaction in benzene, but the open-chain isomer (1c) predominates (56% isolated) if pyridine is used as solvent. In the latter case, when the crude product was recrystallised from ethanol, compound (5) was obtained instead. This structure assignment follows from the elemental analysis, the mass spectrum (in which ethoxy is lost from the molecular ion to give the base peak at *m/e* 284), and the i.r. absorption (see Table) characteristic of a lactam carbonyl group.[10] There is also the precedent of the related ether (6),[2] for which similar spectroscopic evidence was not available.

In the $^1$H n.m.r. spectrum of compound (5) the ethyl group gives rise to an $ABX_3$ pattern. There is an unusually large separation (0.4 p.p.m.) of the resonances due to the prochiral methylene protons, which comprise a quartet of quartet lines ($J_{AB}$ 9; $J_{AX} = J_{BX} = 7$ Hz).[12]

[1] H. Meyer, *Monatsh.*, 1907, **28**, 1211.
[2] S. Wawzonek, H. A. Laitinen, and S. J. Kwiatkowski, *J. Amer. Chem. Soc.*, 1944, **66**, 830.
[3] M. V. Bhatt, K. M. Kamath, and M. Ravindranathan, *J. Chem. Soc.* (*C*), 1971, 1772.
[4] W. Theilacker and W. Schmidt, *Annalen*, 1957, **605**, 43.
[5] M. Ahmed, L. J. Kricka, and J. M. Vernon, *J.C.S. Perkin I*, 1975, 71.
[6] C. Graebe and F. Ullman, *Annalen*, 1896, **291**, 8.
[7] G. Egerer and H. Meyer, *Monatsh.*, 1913, **34**, 69, and earlier references.
[8] J. R. Schaefgen, F. H. Verhoek, and M. S. Newman, *J. Amer. Chem. Soc.*, 1945, **67**, 253.
[9] A. Dunet and A. Willemart, *Bull. Soc. chim. France*, 1948, 887.
[10] W. Graf, E. Girod, E. Schmid, and W. G. Stoll, *Helv. Chim. Acta*, 1959, **42**, 1085, and references therein.
[11] M. V. Bhatt and K. M. Kamath, *J. Chem. Soc.* (*B*), 1968, 1036.
[12] Magnetic non-equivalence of the methylene protons in a series of chiral benzyl ethers has been described by D. M. Whitesides, D. Holtz, and J. D. Roberts, *J. Amer. Chem. Soc.*, 1964, **86**, 2628.

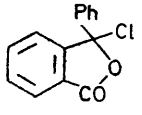
We attribute the formation of compound (5) to catalysis by pyridine hydrochloride remaining in the crude product (1c), since a pure sample of the latter also

I.r. absorption frequencies ($\nu_{max.}$/cm$^{-1}$) [a]

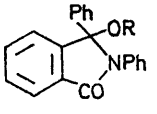
| | C=O | O–H or N–H |
|---|---|---|
| (A) Phthalide derivatives | | |
| (1a) | 1 761 [b] | 3 350 [c] |
| (2a) | 1 756 | 3 420 [c] |
| (3a) | 1 756 | 3 422 [c] |
| (13a) | 1 745 | 3 388 [c] |
| (B) Phthalimidine derivatives | | |
| (1b) | 1 717 [d] | 3 517 (bonded),[e] 3 590 (free O–H) |
| (2b) | 1 716 [d] | 3 562 [d] |
| (5) | 1 700 | |
| (13b) | 1 689 [f] | 3 335 |
| (14) | 1 683 | |
| (15) | 1 688 | |
| (16) | 1 702 | |
| (C) Open chain *o*-benzoylbenzanilides | | |
| (1c) | 1 681, 1 663 [g] | 3 201 |
| | 1 684br [d] | 3 312 [e] |
| (2c) | 1 680br, 1 642 | 3 340 |

[a] In Nujol, except as stated otherwise. [b] In chloroform, but 1 741 cm$^{-1}$ in Nujol, *cf.* 1 760 cm$^{-1}$ (ref. 2). [c] Free N–H absorptions all characteristically sharper than for bonded N–H or O–H. [d] In carbon disulphide. [e] In 1,2-dimethoxyethane. [f] And 1 677w cm$^{-1}$, possibly due to amide C=O of open-chain tautomer. [g] *Cf.* 1 680 cm$^{-1}$ (ref. 2), but 1 720 cm$^{-1}$ in chloroform presumably owing to formation of the lactam tautomer (1b).

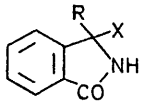
afforded (5) on recrystallisation from ethanol containing mineral acid. Conversely, the ether (5), like (6),[2] was hydrolysed in hot aqueous hydrochloric acid to the open-chain anilide (1c). Analogous interconversions, some



(4)



(5) R = Et
(6) R = Me

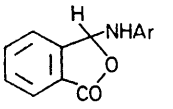


(7) R = H, X = OH
(8) R = H, X = OR′
(9) R = 4-Cl-3-$NH_2$·$SO_2$·$C_6H_3$, X = OH
(10) R = 4-Cl-3-$NH_2$·$SO_2$·$C_6H_3$, X = OR′
(11) R = Ph, X = OH

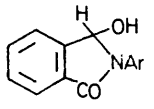
involving also transesterification, of normal open-chain and cyclic $\psi$-esters of *o*-benzoylbenzoic acid catalysed by acids or bases have been recorded.[7,8]

Although the third tautomer (1b) has not been isolated, it is probably an intermediate in the interconversions of (1c) and (5). Other hydroxyphthalimidines (7) and (9) are known, from which the series of ethers (8) and (10), respectively, are obtained with alcohols and hydrochloric acid.[9,10] Whereas reduction of *N*-phenylphthalimide with sodium borohydride is known to give, in part, the hydroxyphthalimidine (12b),[13] its reaction with an equimolar amount of phenyl-lithium gave *o*-benzoylbenzanilide (1c) instead of the cyclic isomer (1b). Of these isolable 3-hydroxyphthalimidines, only (9) contains an aryl substituent at the 3-position: a phenyl group in this position apparently causes a differential stabilisation, *e.g.* by conjugation within the benzoyl group, of the open-chain structure (c) with respect to the lactam (b).

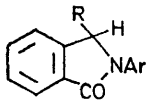
Polarographic evidence [2] and i.r. spectra [10,11] show the existence of *o*-benzoylbenzamide as the cyclic tautomer (11) in solution. Certain features of the i.r. spectra likewise indicate the reversible formation of (1b) from (1c)



(a)



(b)



(12) Ar = Ph
(13) Ar = *o*-$PhC_6H_4$
(14) R = Ar = Ph
(15) R = Ph, Ar = *o*-$PhC_6H_4$
(16) R = H, Ar = *o*-$PhC_6H_4$

in solution. In dry dimethoxyethane three distinct absorptions are recorded in the region 3 300—3 600 cm$^{-1}$, and the intensity of the highest frequency band [$\nu_{max.}$ 3 590 cm$^{-1}$, attributable to free O–H of structure (1b)] increases relative to those of the other two [$\nu_{max.}$ 3 517 and 3 312 cm$^{-1}$, due to hydrogen bonded O–H and N–H, respectively, the latter of structure (1c)] with increasing dilution. In the same solvent the absorption in the carbonyl region is broad and unresolved, but in carbon disulphide separate bands are seen ($\nu_{max.}$ 1 684 and 1 717 cm$^{-1}$), which are due to structures (1c) and (1b), respectively. Both maxima are shifted characteristically to lower frequency by hydrogen bonding on addition of ethanol. Similar i.r. absorptions for C=O and O–H (see Table) show that the analogous *N*-*o*-biphenylylamide (2c) exists in solution mainly in the cyclic form (2b).

Some *N*-*o*-biphenylyl analogues of (1) were also prepared in connection with other synthetic goals,[14] and structures assigned to them in accord with the key i.r. absorptions for C=O, O–H, and N–H stretching, which are given together in the Table; other relevant spectroscopic and analytical evidence is in the Experimental section. Thus, two isomeric products were obtained from the reaction of the $\psi$-chloride (4) with *o*-aminobiphenyl: reaction in pyridine afforded the open chain oxo-amide (2c), whereas reaction without solvent gave the lactone (2a). Similarly the $\psi$-chloride from 3,6-dimethyl-2-benzoylbenzoic acid reacted with *o*-aminobiphenyl without solvent to give the lactone (3a). The analogous lactone (13a) was obtained from the $\psi$-chloride of phthalaldehydic acid and *o*-aminobiphenyl in pyridine or from phthalaldehydic acid itself and *o*-aminobiphenyl

[13] Z.-I. Horii, Ch. Iwata, and Y. Tamura, *J. Org. Chem.*, 1961, **26**, 2273.
[14] M. Ahmed and J. M. Vernon, unpublished work.

in acetone. The isomeric hydroxy-lactam (13b) was the product of reduction of *N*-*o*-biphenylylphthalimide with sodium borohydride (*cf.* preceding discussion).

Reduction of either *o*-benzoylbenzanilide (1c) or its cyclic tautomer (1a) with zinc and refluxing acetic acid gave 2,3-diphenylphthalimidine (14), although Meyer [1] obtained 3-phenylphthalide instead from (1a), which he mistakenly regarded as proving structure (1b) for the latter compound. Formation of (14) most likely occurs *via* reduction of (1b), which may exist in solution in equilibrium with (1a) as well as with (1c). Although (1a) crystallised unchanged from hot acetic acid, or from dioxan or ethanol containing hydrochloric acid, it was converted into (1c) by heating with phosphoric trichloride and then pouring into water.

Similar reduction of the *N*-*o*-biphenylylbenzamide (2c) or the lactone (2a) gave 3-phenylphthalide and 2-*o*-biphenylyl-3-phenylphthalimidine (15), respectively, more in accord with Meyer's results [1] with the ring–chain tautomers of (1). I.r. (see Table) and mass spectroscopic evidence, as well as the precedent of (14), support the assignment of structure (15). From reduction of the lactone (13a) with zinc and acetic acid the corresponding phthalimidine (16) was obtained in low yield, again with rearrangement of the heterocyclic system.

These results underline the inherent unreliability of assigning structures to individual tautomers on the basis of particular reaction products. Spectroscopic evidence is altogether preferable, and it sometimes indicates a change of structure between solution and solid state. Which particular tautomers are isolable or not may reflect only small differential stabilisation of one structure *versus* another, including the effects of intermolecular interactions in the solid state.

## EXPERIMENTAL

I.r. spectra were recorded with a Perkin-Elmer 621 or Unicam SP 200 spectrometer and calibrated with polystyrene. $^1$H N.m.r. spectra were recorded at 60 MHz for solutions in deuteriochloroform with tetramethylsilane as internal standard. Mass spectra were recorded with an A.E.I. MS 12 instrument operating at 70 eV.

The ψ-chloride (4) was prepared from *o*-benzoylbenzoic acid with thionyl chloride; b.p. 158° at 0.6 mmHg (lit.,[3] 177—178° at 3 mmHg), m.p. 55—58° (lit.,[15] 59—60°). 3,6-Dimethylphthalic anhydride and 2-benzoyl-3,6-dimethylbenzoic acid were prepared by known methods: [16] the acid was heated under reflux in thionyl chloride for 40 min, the excess of thionyl chloride was evaporated off under reduced pressure, and the acid chloride thereby obtained, m.p. 114—117°, was used without further purification. Its i.r. spectrum, including $\nu_{max.}$ 1 758 cm$^{-1}$ (lactone C=O), was very similar to that of the ψ-chloride (4).

*Ring–Chain Tautomerism of the Anilide* (1c) *and* ψ-*Anilide* (1a).—Freshly distilled aniline (4.65 g) was added to a solution of the ψ-chloride (4) (12.22 g) in dry pyridine. This mixture was stirred for 1 h, then poured into water; the resulting gum became a solid removable by filtration after further vigorous stirring. This solid was collected and recrystallised (chloroform–ethyl acetate) to give *o*-benzoylbenzanilide (1c) (8.42 g, 56%), m.p. 198.5° (lit.,[1] 195°).

Alternatively a solution of phenyl-lithium, freshly prepared from lithium (0.2 g) and bromobenzene (1.88 g) in dry ether and filtered from unchanged lithium, was added dropwise to a suspension of *N*-phenylphthalimide (2.23 g) in dry ether. The mixture was stirred overnight, then heated under reflux for 1 h, cooled, and treated with water. After filtration the ether layer was separated and evaporated; the residue on recrystallisation (methanol) gave the anilide (1c) (0.90 g, 30%), m.p. 196—198°, identical with the material described above.

A solution of the ψ-chloride (4) (4.88 g) and freshly distilled aniline (1.86 g) in dry pyridine was heated under reflux for 1.1 h, and then evaporated under reduced pressure. The residue crystallised from ethanol to give 3-*ethoxy*-2,3-*diphenylphthalimidine* (5) (2.63 g, 40%) as prisms, m.p. 137—139° (Found: C, 80.0; H, 5.9; N, 4.3. $C_{22}H_{19}NO_2$ requires C, 80.2; H, 5.8; N, 4.3%), τ 1.9—2.9 (14 H, m, ArH), 6.70 (2 H, sixteen-line, $CH_2$), and 8.79 (3 H, t, *J* 7 Hz, $CH_3$), *m/e* 329 ($M^+$, 40%), 285 (27), 284 ($M$ − OEt, 100), 224 (14), 209 (19), 105 (10), and 77 (32), *m* * 245 (329 ⟶ 284). The same product (5) was obtained by recrystallisation of (1c) from ethanol containing 1—2 drops of concentrated hydrochloric or sulphuric acid.

The ether (5) was hydrolysed by dissolving in hot dioxan containing aqueous hydrochloric acid and heating under reflux for 3 h. Solvents were removed under reduced pressure, and the residue was washed with water, dried, and recrystallised (ethyl acetate) to give the anilide (1c) (65%), m.p. and mixed m.p. 195—196°. Alternatively the ether (5) was dissolved in concentrated sulphuric acid and this red solution [$\lambda_{max.}$ 382 nm (ε 16 000)] was then added to water. The precipitated solid was collected and recrystallised to give the anilide (1c), identical (i.r. spectrum and mixed m.p.) with an authentic sample.

Freshly distilled aniline (4.09 g) was added with stirring to a solution of the ψ-chloride (4) (9.78 g) in dry benzene. After 0.5 h the solid was collected, washed with dilute hydrochloric acid and with water, dried, and recrystallised (chloroform–methanol) to give 3-anilino-3-phenylphthalide (1a) (8.40 g, 70%), m.p. 221.5—223.5° (lit.,[1,3] 221°). This compound, unlike its isomer (1c), gave no red colour with concentrated sulphuric acid, and it was unchanged after being heated under reflux in ethanol containing hydrochloric acid or in acetic acid containing sulphuric acid. However, (1a) (1.0 g) gave a red solution in phosphoric trichloride, which was heated under reflux for 45 min, then evaporated under reduced pressure. Aqueous sodium carbonate solution was added to the residue, and the mixture was stirred for 30 min before the solid was collected, washed, and dried. Recrystallisation (methanol) gave the anilide (1c) (0.60 g), m.p. and mixed m.p. 196–198°.

N-o-*Biphenylylamides*.—A mixture of the ψ-chloride (4) (2.24 g) and *o*-aminobiphenyl (1.69 g) was heated at 100—110 °C for 5 min, and then dissolved in hot ethanol. A first crop of yellow crystals deposited from this solution as it cooled was anthraquinone (0.22 g), m.p. and mixed m.p. 283—285°, i.r. spectrum identical with that of an authentic sample. [The formation of anthraquinone from the ψ-chloride (4) above 100 °C has been noted previously.[6,17] The mother liquor after concentration under reduced pressure then afforded 3-o-*biphenylylamino*-3-*phenylphthalide*

[15] H. C. Martin, *J. Amer. Chem. Soc.*, 1916, **38**, 1142.
[16] M. S. Newman and B. T. Lord, *J. Amer. Chem. Soc.*, 1944, **66**, 733.
[17] H. Meyer, *Monatsh.*, 1904, **25**, 1177.

(2a) (1.50 g, 40%), m.p. 205—206° (Found: C, 82.5; H, 5.2; N, 3.6. $C_{26}H_{19}NO_2$ requires C, 82.7; H, 5.1; N, 3.7%), *m/e* 377 ($M^+$, 8%), 210 (15), 209 (*o*-$PhCOC_6H_4CO^+$, 100), 167 (23), 153 (17), 152 (28), 105 (10), and 77 (19), *m** 116 (377 ⟶ 209). The same product (2a) (50%) was prepared from the ψ-chloride (4) and *o*-aminobiphenyl in dry benzene, as described above for the ψ-anilide (1a).

Alternatively, reaction of the ψ-chloride (4) (4.89 g) and *o*-aminobiphenyl (3.38 g) in dry pyridine at room temperature and work-up as described above for the anilide (1c) afforded N-o-*biphenylyl*-o-*benzoylbenzamide* (2c) (4.15 g, 55%), m.p. 182—185° (from methanol) (Found: C, 82.6; H, 5.2; N, 3.7. $C_{26}H_{19}NO_2$ requires C, 82.7; H, 5.1; N, 3.7%), *m/e* 377 ($M^+$, 10%), 210 (15), 209 (*o*-$PhCOC_6H_4CO^+$, 100), 167 (4), 153 (11), 152 (18), 151 (4), 105 (8), and 77 (14), *m** 129, 116 (377 ⟶ 209), and 112.

The ψ-chloride (0.51 g) from 2-benzoyl-3,6-dimethylbenzoic acid was heated with *o*-aminobiphenyl (0.34 g) at 108 °C for 3 min and the resulting sticky solid was recrystallised (ethanol) to give the N-o-*biphenylyl ψ-amide* (3a) (0.35 g, 48%), m.p. 217—219° (Found: C, 82.8; H, 5.8; N, 3.4. $C_{28}H_{23}NO_2$ requires C, 82.9; H, 5.7, N, 3.45%), τ 2.6—3.4 (16 H, m, ArH), 5.05 (1 H, s, NH, exchangeable in $D_2O$), and 7.36 and 7.93 (each 3 H, s, $CH_3$), *m/e* 405 ($M^+$, 14%), 238 (19), and 237 ($M$ − *o*-$PhC_6H_4NH$, 100), *m** 138.5 (405 ⟶ 237).

Phthalaldehydic acid (5.0 g) dissolved in thionyl chloride (60 ml) was heated under reflux for 3 h, and the excess of thionyl chloride was removed under reduced pressure. The remaining oil, the crude ψ-chloride, was dissolved in dry pyridine and added to a solution of *o*-aminobiphenyl (5.66 g) in the same solvent. The mixture was stirred during 3 h; it was then poured into water and stirring was continued until the gummy precipitate became a solid removable by filtration. The solid was collected, washed, dried, and recrystallised (chloroform–methanol) to give the 3-o-*biphenylylaminophthalide* (13a) (6.0 g, 60%), m.p. 188—190° (Found: C, 79.9; H, 5.1; N, 4.4. $C_{20}H_{15}NO_2$ requires C, 79.7; H, 5.0; N, 4.65%), τ 2.1—3.2 (13 H, m, ArH), 3.28 (1 H, d, *J* 10 Hz, collapses to singlet on shaking with $D_2O$, 3-H), and 5.30 (1 H, d, NH, exchangeable in $D_2O$), *m/e* 302 (16%), 301 ($M^+$, 69), 273 (6), 256 (23), 196 (11), 180 (56), 168 (20), 167 (38), 166 (9), 152 (20), 134 (11), 133 ($M$ − *o*-$PhC_6H_4NH$, 100), 105 (55), and 77 (44), *m** 247.5 (301 ⟶ 273), 240 (273 ⟶ 256), 82.8 (133 ⟶ 105), and 56.5 (105 ⟶ 77). The same compound (13a) (90%) was prepared directly from the reaction of phthalaldehydic acid and *o*-aminobiphenyl in acetone at room temperature (*cf.* a procedure for the preparation of 3-anilinophthalide [18]).

A solution of sodium borohydride (0.50 g) in methanol (25 ml) was added dropwise to a stirred suspension of *N-o*-biphenylylphthalimide [19] (5.0 g) in methanol (25 ml). Stirring was then continued for 3 h. The resulting clear solution was concentrated under reduced pressure; it was then poured into water, and the resulting solid was collected, washed, and dried. Recrystallisation (carbon tetrachloride–ethanol) afforded 2-*o*-*biphenylyl*-3-*hydroxyphthalimidine* (13 b) (3.0 g, 60%), m.p. 169—171° (Found: C, 79.5; H, 5.0; N, 4.5. $C_{20}H_{15}NO_2$ requires C, 79.7; H, 5.0; N, 4.6%), τ 2.4—3.0 (13 H, m, ArH), 4.63 (1 H, d, *J* 10 Hz, collapses to singlet on shaking with $D_2O$, 3-H), and 6.60 (1 H, d, OH exchangeable in $D_2O$), *m/e* 302 (23%), 301 ($M^+$, 100), 300 (10), 284 (11), 282 (26), 273 (4),256 (11), 254 (15), 196 (15), 180 (26), 167 (14), 154 (56), 152 (23), 133 (31), 127 (11), 105 (74), 91 (18), and 77 (42), *m** 265 (300 ⟶ 282), 248 (301 ⟶ 273), 79 (301 ⟶ 154), and 83 (133 ⟶ 105).

*Reductions with Zinc and Acetic Acid.*—Zinc dust, activated according to the method of Tsuda,[20] was added in portions to a solution of the compound to be reduced in acetic acid–ethanol (3 : 1 v/v) which was heated under reflux for 2 h. Most of the solvents were then evaporated off under reduced pressure, and the residue was poured into water and stirred, if necessary, to obtain a solid removable by filtration. This solid was collected, washed, dried, and recrystallised. The following results were obtained by the same general procedure.

Reduction of the ψ-anilide (1a) (24 g) gave 2,3-diphenylphthalimidine (14) (7.5 g, 33%), m.p. 192—193° (from aqueous methanol) (lit.,[1] m.p. 195°). Reduction of the isomeric anilide (1c) also gave (14) (30%) with the same m.p. and i.r. spectrum.

Reduction of compound (2a) (3.8 g) gave 2-o-*biphenylyl*-3-*phenylphthalimidine* (15) (2.33 g, 65%), m.p. 143—145° (from ethanol) (Found: C, 86.2; H, 5.5; N, 3.7. $C_{26}H_{19}NO$ requires C, 86.4; H, 5.3; N, 3.9%), τ 2.5—3.4 (18 H, m, ArH) and 5.07 (1 H, s, CH), *m/e* 362 (28%), 361 ($M^+$, 100), 360 (5), 332 (17), 281 (9), 255 (18), 253 (10), 153 (10), 105 (6), and 77 (8), *m** 306 (360 ⟶ 332). Similar reduction of the open-chain isomer (2c) afforded 3-phenylphthalide, m.p. 112—115° (lit.,[1] 115°), identical (i.r. spectrum and mixed m.p.) with an authentic sample.[21]

Reduction of 3-*o*-biphenylylaminophthalide (13a) (2.0 g) gave a gum, which was chromatographed on a column of silica gel. Evaporation of the benzene eluate again gave a gum, from which was obtained the *phthalimidine* (16) (0.10 g, 5%), m.p. 308—309.5° (from aqueous methanol) (Found: C, 84.5; H, 5.1; N, 4.9. $C_{20}H_{15}NO$ requires C, 84.2; H, 5.3; N, 4.9%), τ ($CCl_4$) 2.3—3.2 (13 H, m, ArH) and 6.06 (2 H, s, $CH_2$), *m/e* 285 (25%), 284 ($M$ − H, 100), 267 (8), 266 (9), 256 (7), 255 (4), 254 (11), 180 (6), 178 (12), 152 (16), 151 (6), 105 (4), and 77 (5). Further elution of the column with benzene–ether afforded *N-o*-biphenylylphthalimide (0.20 g, 10%), m.p. 163—165° (lit.,[19] 165—166°), identical (i.r. spectrum and mixed m.p.) with an authentic sample. Similar reduction of the isomer (13b) also gave a gum, with i.r. spectrum the same as that of the crude product from (13a), but no crystalline material was isolated.

We are indebted to Dr. P. Hanson for discussions and to Mr. R. B. Girling for microanalyses and for assistance in recording some of the i.r. spectra.

[5/780 *Received*, 25*th April*, 1975]

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