Iminyls. Part 2.[1] Intramolecular Aromatic Substitution by Iminyls. A New Route to Phenanthridines and Quinolines †

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Biphenyl-2-yl- and triarylvinyl-iminyls, generated by oxidation of the corresponding imino-oxyacetic acids with persulphate and by thermolysis of the t-butyl peresters of these acids, readily cyclise in high yield to phenanthridines and quinolines, respectively.

IMINYLS do not react with benzene or other aromatic solvents. Diphenylmethyleneaniline is not formed when the t-butyl perester of diphenylmethyleneamino-oxyacetic acid is decomposed in benzene solution,[1] and the reported [2] iminylation of aromatic solvents on photolysis of solutions of aromatic ketone *O*-aroyloximes was subsequently found to be an indirect process.[3] The key step in the latter reaction is substitution of the arene by the aroyloxyl radicals which are produced with the iminyls on initial O–N bond scission [equation (i)].

$$Ar_2C{=}NOCOAr' \longrightarrow Ar_2C{=}N\cdot + \cdot OCOAr' \qquad (i)$$

In contrast, intramolecular aromatic substitution proceeds with ease given favourable geometry. Thus, biphenyl-2-yliminyls (4), produced by (a) persulphate oxidation of the corresponding amino-oxy-acetic and -2-methylpropanoic acids (1; R′ = H and Me), (b) thermolysis of the t-butyl peresters of these acids (2), and (c) oxidation of the oxime methyl ethers (3) with t-butoxyl radicals, cyclise to phenanthridines in almost all cases, in good yield (Table 1). Hunsdiecker oxidation of the acid (1; R = Ph, R′ = H) was less successful giving only 9% yield of phenanthridine (5; R = Ph) and only traces of phenanthridine (5; R = Ph) were produced when phenyl radicals (from benzoyl peroxide) were generated in a solution containing 2-cyanobiphenyl (7).

TABLE 1

Yields (%) of phenanthridines (5) from iminyl precursors

Iminyl precursor	Method of production	Phenanthridine (5)	Other products
(1; R = R′ = H)	*a*	71 (80*)	1* (7)
(1; R = Me, R′ = H)	*a*	60	
(1; R = Ph, R′ = H)	*a*	66	
(1; R = H, R′ = Me)	*a*	78	2 (7)
(1; R = R′ = Me)	*a*	71	
(1; R = Ph, R′ = Me)	*a*	75	
(2; R = H)	*b*	60	13 (6; R = H)
(2; R = Me)	*b*	52	20 (6; R = Me)
(3; R = Ph)	*c*	22	

* By g.l.c. [a] Persulphate oxidation of imino-oxy acid. [b] Thermolysis of t-butyl perester of imino-oxy acid in benzene. [c] Oxidation of methyl ether of oxime with Bu^tO.

The triarylvinyl iminyl (8; Ar = Ph), produced from the corresponding oxyacetic acid or t-butyl perester, also cyclises readily to give the quinoline (9; Ar = Ph)

TABLE 2

Yields (%) of quinolines (9) obtained from vinyliminyls (8)

Iminyl	Method of production	Quinoline (9)	Acetal
(8; Ar = Ph)	*a*	91	
(8; Ar = *o*-MeC_6H_4)	*a*	75	
(8; Ar = Ph)	*b*	35	45

[a,b] See footnotes to Table 1.

(Table 2). This cyclisation is apparently energetically more favourable than intramolecular abstraction of

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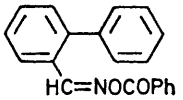
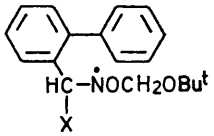
benzylic hydrogen since the *o*-tolyl(triphenylvinyl)-iminyl (8; Ar = *o*-MeC_6H_4) gave the quinoline (9; Ar = *p*-MeC_6H_4, R = Ph) in 75% yield in preference to products derived from the benzylic radical (10) (see Part 4).

E.s.r. Spectra.—Unlike the diaryliminyls described in Part 1, which dimerised to azines, the iminyls (4) and (8) which readily undergo intramolecular cyclisation could not be detected by e.s.r. when solutions of the t-butyl peresters of the corresponding amino-oxyacetic acids in benzene were heated at 75° in the spectrometer. However, a spectrum with a_N 14.35, a_H 20.0 (1 H), a_H 1.75 (2 H), and *g* 2.004 9 was slowly produced when the perester (2; R = H) was heated. Attempts to generate this spectrum in other ways such as by reaction of phenanthridine with t-butoxyl radicals (from di-t-butyl peroxide and peroxalate) and irradiation of the benzoate (11) were not successful. Hence, we attribute this spectrum to the alkoxylaminyl (12) (*cf.* $PhCH_2\dot{N}OCH_2Ph$ which



(11)



(12)

has a_N 14.41, $a_H^{CH_2}$ 23.79, $a_H^{OCH_2}$ 2.62, and *g* 2.004 64 [4] formed by addition of a radical (X·) to the acetal (6; R = H). Similar spectra have been detected from other peresters in this work (see later papers). The identity of the radical X· has not been established although it does not appear to be t-butoxyl since alkoxyaminyls (12) cannot be detected when the corresponding acetals (6) are irradiated with di-t-butyl peroxide.

EXPERIMENTAL

I.r. spectra were measured as KBr discs and n.m.r. spectra using deuteriochloroform as solvent, unless stated otherwise. Petrol refers to light petroleum, b.p. 60—80°. Merck silica gel GF_{254} was used for chromatographic separations.

Preparation of Ketones, Imines, and Oximes.—*o*-Phenylbenzophenone,[5] *o*-phenylacetophenone,[6] and *o*-phenylbenzaldehyde [7] were prepared by literature methods.

αβ-Diphenylchalcone Imine.—To a solution of triphenylvinylmagnesium bromide [prepared from triphenylvinyl bromide (40 g, 0.12 mol) and magnesium (3.1 g, 0.13 mol) in ether (800 ml)] benzonitrile (62 g, 0.6 mol) was added dropwise with stirring. The mixture was heated under reflux overnight before the cream solid which had separated was collected, and washed with ether. The solid was suspended in ether, anhydrous methanol (25 ml) was added, and the mixture was heated under reflux for 2 h. The inorganic salts were collected, and the filtrate was evaporated to give *αβ-diphenylchalcone imine* as plates, m.p. 151—155° (from acetone) (Found: C, 90.2; H, 5.9; N, 4.0. $C_{27}H_{21}N$ requires C, 90.2; H, 5.9; N, 3.9%), $\nu_{max.}$ 3 238 cm^{-1}.

When the imine–magnesium complex was decomposed by passage of dry hydrogen chloride through an ethereal suspension, the *imine hydrochloride* was formed. This gave yellow rhombs, m.p. 210—218° (from methanol) (Found: C, 81.6; H, 5.7; Cl, 8.7; N, 3.7. $C_{27}H_{22}ClN$ requires C, 81.9; H, 5.6; Cl, 9.0; N, 3.55%), $\nu_{max.}$ 2 710br and 1 649 cm^{-1}, δ 4.68 (2 H, s, NH_2).

αβ-Diphenyl-2′-methylchalcone imine was similarly prepared from *o*-toluonitrile and triphenylvinylmagnesium bromide. It formed plates, m.p. 151—153° (from aqueous ethanol) (Found: C, 90.1; H, 6.2; N, 4.0. $C_{28}H_{23}N$ requires C, 90.05; H, 6.2; N, 3.75%), $\nu_{max.}$ 3 235 cm^{-1}. The *hydrochloride* formed yellow needles, m.p. 218° (from petrol–chloroform) (Found: C, 82.05; H, 5.8; Cl, 8.6; N, 3.4. $C_{28}H_{24}ClN$ requires C, 82.05; H, 5.9; Cl, 8.65; N, 3.4%), $\nu_{max.}$ 2 680br and 1 636 cm^{-1}, δ 2.53 (3 H, s, Me).

o-*Phenylacetophenone oxime* was prepared from the ketone and hydroxylamine hydrochloride in pyridine–ethanol. It formed needles, m.p. 103—108° (from methanol–water) (Found: C, 79.3; H, 6.3; N, 6.6. $C_{14}H_{13}NO$ requires C, 79.6; H, 6.2; N, 6.6%). *o*-Phenylbenzophenone [8] and *o*-phenylbenzaldehyde oximes,[9a] were similarly prepared.

αβ-Diphenylchalcone oxime was prepared from the imine hydrochloride, hydroxylamine hydrochloride, and sodium acetate.[9b] It gave rhombs, m.p. 192—196° (from methanol) (Found: C, 86.1; H, 5.3; N, 3.7. $C_{27}H_{21}NO$ requires C, 86.35; H, 5.65; N, 3.75%), $\nu_{max.}$ 3 230br cm^{-1}. Similarly prepared was *αβ-diphenyl-2′-methylchalcone oxime* which gave rhombs, m.p. 159—161° (from chloroform–petrol) (Found: C, 86.2; H, 6.0; N, 3.4. $C_{28}H_{23}NO$ requires C, 86.35; H, 5.95; N, 3.6%), $\nu_{max.}$ 3 260br cm^{-1}.

Preparation of Imino-oxyacetic Acids.—These were prepared from the oxime, chloroacetic acid, and sodium hydroxide in aqueous ethanol.[1] The following are new. *Biphenyl-2-yl(phenyl)methyleneamino-oxyacetic acid* (1; R = Ph, R′ = H) had m.p. 123—125° (from petrol–benzene) (Found: C, 76.4; H, 5.3; N, 4.2%; M^+, 331.120 7. $C_{21}H_{17}NO_3$ requires C, 76.1; H, 5.2; N, 4.2%; *M*, 331.120 8), $\nu_{max.}$ (Nujol) 3 400—2 600, 1 730, and 1 705sh cm^{-1}, δ 4.60 and 4.70 (total 2 H, both s, OCH_2) (two isomers), 7.0—7.6 (14 H, m, ArH), and 10.9 (1 H, s, CO_2H); 1-*biphenyl-2-ylethyleneamino-oxyacetic acid* (1; R = Me, R′ = H) was a gum (Found: M^+, 269.105 2). $C_{16}H_{15}NO_3$ requires *M*, 262.1051), $\nu_{max.}$ (Nujol) 3 400—2 600 and 1 730 cm^{-1}, δ 1.74 and 2.08 (total 3 H, both s, Me) (two isomers), 4.57 and 4.75 (total 2 H, both s, OCH_2) (two isomers), and 7.2—7.4 (9 H, m, ArH); *biphenyl-2-ylmethyleneamino-oxyacetic acid* (1; R = R′ = H) had m.p. 142—145° (from petrol–benzene) (Found: C, 70.3; H, 5.0; N, 5.7. $C_{15}H_{13}NO_3$ requires C, 70.6; H, 5.1; N, 5.5%), $\nu_{max.}$ (Nujol) 3 400—2 600 and 1 715 cm^{-1}, δ 4.72 (2 H, s, OCH_2), 7.2—7.6 (8 H, m, ArH), 7.8—8.1 (1 H, m, ArH), 8.20 (1 H, s, CH=N), and 9.45br (1 H, s, CO_2H); *α-triphenylvinylbenzylideneamino-oxyacetic acid* formed needles, m.p. 192—194° (from aqueous alcohol) (Found: C, 80.4; H, 5.2; N, 3.5. $C_{29}H_{23}NO_3$ requires C, 80.35; H, 5.35; N, 3.25%), $\nu_{max.}$ 1 730 cm^{-1}, δ 4.30 (2 H, s, OCH_2); *(2-methyl-α-triphenylvinylbenzylidene)amino-oxyacetic acid* gave needles, m.p. 184—187° (from petrol–benzene) (Found: C, 80.8; H, 5.7; N, 3.3. $C_{30}H_{25}NO_3$ requires C, 80.5; H, 5.65; N, 3.15%), $\nu_{max.}$ 1 724 cm^{-1}, δ 2.11 (3 H, s, Me) and 4.36 (2 H, s, OCH_2).

Preparation of 2-Imino-oxy-2-methylpropanoic Acids (1; R′ = Me).—These were prepared from the oxime and chloretone following Corey *et al.*[10] 2-[*Biphenyl-2-yl(phenyl)-methyleneamino-oxy*]-2-*methylpropanoic acid* (1; R = Ph, R′ = Me) had m.p. 133—150° (two isomers) (from petrol–benzene) (Found: C, 76.6; H, 5.8; N, 3.9%. $C_{23}H_{21}NO_3$

requires C, 76.9; H, 5.9; N, 3.9%), $\nu_{max.}$ (Nujol) 3 400—2 600 and 1 720 cm^{-1}, δ 1.32br and 1.45br (total 6 H, 2 × s, $OCMe_2$) and 7.1—7.6 (14 H, m, ArH). Fractional crystallisation from petrol–benzene gave one isomer pure, m.p. 157—160°, δ 1.33br (6 H, s, $OCMe_2$); 2-(1-*biphenyl*-2-*ylethylideneamino-oxy*)-2-*methylpropanoic acid* (1; R = R′ = Me) had m.p. 106—110° (one isomer obtained by fractional crystallisation from aqueous ethanol) (Found: C, 72.4; H, 6.5; N, 4.9. $C_{18}H_{19}NO_3$ requires C, 72.7; H, 6.5; N, 4.7%), $\nu_{max.}$ (Nujol) 3 400—2 600 and 1 720 cm^{-1}, δ 1.52 (6 H, s, $OCMe_2$), 1.78 (3 H, s, MeC=N), and 7.3—7.5 (9 H, m, ArH); 2-(*biphenyl*-2-*ylmethyleneamino-oxy*)-2-*methylpropanoic acid* (1; R = H, R′ = Me) had m.p. 129—134° (from petrol–benzene) (Found: C, 71.9; H, 6.3; N, 4.9. $C_{17}H_{17}NO_3$ requires C, 72.1; H, 6.05; N, 4.9%), $\nu_{max.}$ (Nujol) 3 400—2 600 and 1 715 cm^{-1}, δ 1.60 (6 H, s, $OCMe_2$), 7.2—7.6 (8 H, m, ArH), 7.9—8.15 (1 H, m, ArH), and 8.15 (1 H, s, CH=N).

Preparation of t-Butyl Peresters of Imino-oxyacetic Acids.—These were prepared either from the acid chloride and t-butyl hydroperoxide [1] or the acid, t-butyl hydroperoxide, and di-imidazolyl ketone.[11] *Biphenyl-2-ylmethyleneamino-oxyacetyl chloride* formed needles, m.p. 85—89° (from pentane–chloroform) (Found: C, 66.1; H, 4.4; Cl, 12.6; N, 5.4. $C_{15}H_{12}ClNO_2$ requires C, 65.85; H, 4.4; Cl, 12.9; N, 5.1%), $\nu_{max.}$ 1 802 cm^{-1}, δ 4.93 (2 H, s, OCH_2) and 8.18 (1 H, s, CH=N). *t-Butyl diphenyl-2-ylmethyleneamino-oxyperacetate* (2; R = H) was an oil (Found: C, 69.6; H, 6.6; N, 4.0. $C_{19}H_{21}NO_4$ requires C, 69.7; H, 6.45; N, 4.3%), $\nu_{max.}$ 1 785 cm^{-1}, δ 1.35 (9 H, s, Bu^t), 4.74 (2 H, s, OCH_2), and 8.19 (1 H, s, CH=N).

1-*Biphenyl-2-ylethylideneamino-oxyacetyl chloride* was a pale yellow oil (Found: C, 67.1; H, 5.0; Cl, 12.5; N, 5.0. $C_{16}H_{14}ClNO_2$ requires C, 66.8; H, 4.9; Cl, 12.3; N, 4.85%), $\nu_{max.}$ 1 812 cm^{-1}, δ 1.70 (3 H, s, Me) and 4.93 (2 H, s, OCH_2). The *t-butyl peracetate* (2; R = Me) was a pale yellow oil (Found: M^+, 341.163 1. $C_{20}H_{23}NO_4$ requires M, 341.162 7), $\nu_{max.}$ 1 785 cm^{-1}, δ 1.35 (9 H, s, Bu^t), 1.70 (3 H, s, Me), and 4.79 (2 H, s, OCH_2).

α-*Triphenylvinylbenzylideneamino-oxyacetyl chloride* formed needles, m.p. 152—155° (from benzene–petrol) (Found: C, 76.9; H, 5.2; Cl, 7.5; N, 3.3. $C_{29}H_{22}ClNO_2$ requires C, 77.0; H, 4.9; Cl, 7.85; N, 3.1%), $\nu_{max.}$ 1 813 cm^{-1}, δ 4.76 (2 H, s, OCH_2). The corresponding *t-butyl peracetate* was a pale yellow oil (Found: C, 78.1; H, 6.2; N, 3.1. $C_{33}H_{31}NO_4$ requires C, 78.4; H, 6.2; N, 2.75%), $\nu_{max.}$ 1 790 cm^{-1}, δ 1.34 (9 H, s, Bu^t) and 4.54 (2 H, s, OCH_2).

o-*Phenylbenzophenone oxime O-methyl ether* (3; R = Ph) was prepared by heating under reflux an equimolar solution of the ketone and methoxyamine hydrochloride in pyridine. The product was purified by chromatography on silica using chloroform as eluant. It formed crystals, m.p. 95—98° (from methanol) (Found: C, 83.3; H, 6.1; N, 5.0. $C_{20}H_{17}NO$ requires C, 83.6; H, 6.0; N, 4.9%), δ(CCl_4) 3.68 and 3.80 (total 3 H, 2s, OMe) (two isomers).

O-*Benzoyl*-o-*phenylbenzaldoxime*.—To a stirred solution of o-phenylbenzaldoxime (1.5 g) in dry pyridine (2 ml), benzoyl chloride (1.1 g) was added dropwise. After 30 min water was added and the resulting precipitate was collected. Crystallisation from methanol gave the *benzoate*, m.p. 122—124° (Found: C, 79.7; H, 5.3; N, 4.9. $C_{20}H_{15}NO_2$ requires C, 79.7; H, 5.0; N, 4.65%).

Persulphate Oxidations of Imino-oxyacetic Acids.—These were carried out by thermolysis as described previously.[1] Biphenyl-2-yl(phenyl)methyleneamino-oxyacetic acid gave 9-phenylphenanthridine (66%) identical with an authentic specimen,[8] o-phenylbenzophenone (trace), and unchanged acid (14%). 2-[*Biphenyl-2-yl(phenyl)methyleneamino-oxy*]-2-*ethylpropanoic* acid gave 9-phenylphenanthridine [8] (75%), a trace of o-phenylbenzophenone, and unchanged acid (20%).

1-Biphenyl-2-ylethylideneamino-oxyacetic acid gave 9-methylphenanthridine,[12] (60%) identical with an authentic sample, and unchanged acid (<3%). 2-(1-Biphenyl-2-ylethylideneamino-oxy)-2-methylpropanoic acid gave 9-methylphenanthridine [12] (71%), o-methylacetophenone (6%), and unchanged acid (20%). This oxidation was carried out on a single isomer, m.p. 106—110°. A sample of the acid on storage for two months at room temperature was found to contain 9-methylphenanthridine [12] (6%). Biphenyl-2-ylmethyleneamino-oxyacetic *acid* gave phenanthridine (71%), o-phenylbenzaldehyde (7%), and unchanged acid (9%). In a second experiment the neutral products were analysed by g.l.c. (after initial purification by t.l.c.) to give phenanthridine (80%), o-phenylbenzaldehyde (9%), and 2-cyanobiphenyl (<1%). Unchanged acid (10%) was recovered. 2-(Biphenyl-2-ylmethyleneamino-oxy)-2-methylpropanoic acid gave phenanthridine (78%), o-phenylbenzaldehyde (9%), 2-cyanobiphenyl (2%), and unchanged acid (21%). α-Triphenyliminylbenzylideneamino-oxyacetic acid (87 mg) gave 2,3,4-triphenylquinoline (65 mg, 91%) as needles, m.p. 195° (from methanol) (lit.,[13] 198—199°) (Found: C, 90.5; H, 5.3; N, 4.1. Calc. for $C_{27}H_{19}N$: C, 90.7; H, 5.35; N, 3.9%). (2-Methyl-α-triphenyliminylbenzylidene)amino-oxyacetic acid (894 mg) gave 2-(o-*tolyl*)-3,4-*diphenylquinoline* (305 mg, 73%) as rhombs, m.p. 160—162° (from aqueous methanol) (Found: C, 90.7; H, 5.8; N, 3.9. $C_{28}H_{21}N$ requires C, 90.55; H, 5.7; N, 3.75%), δ 2.15 (3 H, s, Me), 6.92—7.88 (17 H, m, ArH), and 8.28 (1 H, dd, o-ArH), and unchanged acid (390 mg).

Other Oxidations.—Biphenyl-2-yl(phenyl)methyleneamino-oxyacetic acid (0.3 mol), mercury(II) oxide (0.3 mol), and bromine (0.4 mol) were heated in refluxing carbon tetrachloride for 1 h. Chromatographic separation of the complex product mixture gave 9-phenylphenanthridine (9%).

(ii) o-Phenylbenzophenone oxime O-methyl ether (0.1 mol) and di-t-butyl peroxalate (0.11 mol) were dissolved in benzene and the solution was heated under reflux for 1 h. Chromatographic separation of the product mixture, which contained much starting material, gave 9-phenylphenanthridine (22%).

Decomposition of t-Butyl Peresters.—t-Butyl biphenyl-2-ylmethyleneamino-oxyperacetate (200 mg) in benzene (10 ml) was heated under reflux under nitrogen for 2 h. Evaporation of solvent followed by chromatography of the residue on silica with petrol–chloroform (2 : 3) gave (a) *biphenyl-2-ylmethyleneamino-oxy-t-butoxymethane* (21.5 mg, 13%) as an oil (Found: C, 76.5; H, 7.4; N, 5.0. $C_{18}H_{21}NO_2$ requires C, 76.3; H, 7.45; N, 4.95%), δ 1.27 (9 H, s, Bu^t), 5.34 (2 H, s, OCH_2), 7.38—7.75 (9 H, m, ArH), and 8.09 (1 H, s, CH=N), and (b) phenanthridine (64.5 mg, 60%). t-Butyl 1-biphenyl-2-ylethylideneamino-oxyperacetate (340 mg) gave (a) 1-*biphenyl-2-ylethylideneamino-oxy-t-butoxymethane* (59 mg, 20%) as an oil (Found: C, 76.9; H, 8.1; N, 4.9. $C_{19}H_{23}NO_2$ requires C, 76.75; H, 7.8; N, 4.7%), δ (two isomers 82 : 8) 1.29 and 1.20 (total 18 H, each s, Bu^t), 1.63 and 1.73 (total 6 H, each s, ArMe), 5.20 and 5.37 (total 4 H, each s, OCH_2), 7.38 (18 H, m,

ArH) and (b) 9-methylphenanthridine (100 mg, 52%). t-Butyl α-triphenyliminylbenzylideneamino-oxyperacetate (200 mg) gave (a) *t-butoxy-α-triphenyliminylbenzylideneamino-oxymethane* (81 mg, 45%) as rhombs, m.p. 122—127° (after short path-distillation) (Found: C, 83.5; H, 6.9; N, 2.8. $C_{32}H_{31}NO_2$ requires C, 83.25; H, 6.75; N, 3.05%), δ 1.24 (9 H, s, Bu^t), 5.25 (2 H, s, OCH_2), 7.71 (2 H, m, *o*-H of 2-Ph), and 7.14 (18 H, m, ArH), and (b) 2,3,4-triphenylquinoline (48 mg, 35%).

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