166. *The Oxidation of Derivatives of* o-*Phenylenediamine. Part V.*[1] N^3-*Substituted Derivatives of Anilino*apo*safranine (Rimino-compounds) and Related Compounds.*

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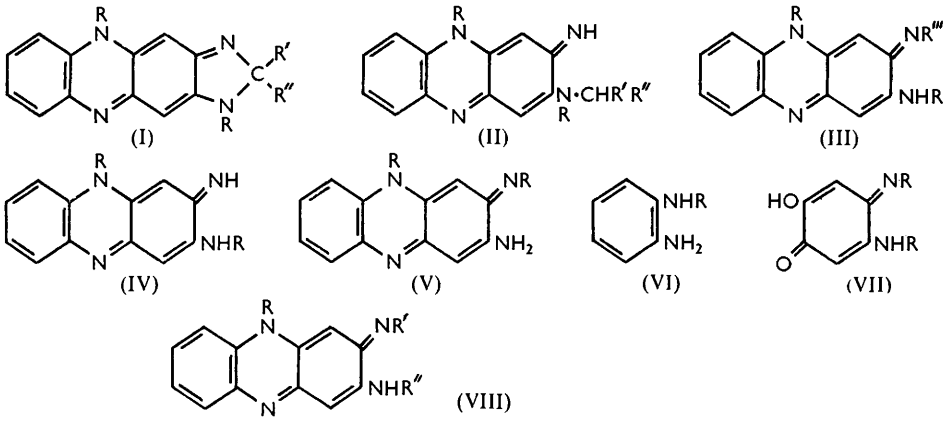
Catalytic hydrogenation of glyoxalinophenazines [1] yields N^3-substituted derivatives of anilino*apo*safranines (rimino-compounds) and not N^2-substituted derivatives as stated earlier.[1] Some of these compounds, and others not accessible by the glyoxalinophenazine route, have been prepared by reaction of primary amines with anilino*apo*safranines. Similar compounds have also been obtained by condensation of isomers [2] of anilino*apo*safranines with some primary amines and by condensation of 4 : 5-diarylamino-*o*-quinones with *o*-phenylenediamines.[3]

We have described in Part IV [1] the hydrogenation of dihydrogloxalinophenazines (I) which resulted in the opening of the glyoxaline ring and formation of derivatives to which we assigned structure (II). The presence of =NH in these compounds was suggested by their relative instability in boiling acetic anhydride and the appearance of a sharp band at 3300 cm.$^{-1}$ in the infrared region. Further examination of the infrared spectra of these and related compounds has shown that this band is due to an $-NH-$ group. Again, deductions based on the results of bromination of these compounds are now known to be of doubtful validity, as a continuing investigation of the bromination of a variety of related phenazines has shown the situation to be very complex. It is now clear that formula (II) does not represent the structure of these "rimino-compounds" and that hydrogenation results in the opening of the 1′ : 2′-bond of the glyoxalino-ring giving



compounds of structure (III). We have also shown that compound (III; R = Ph, R‴ = Pr^i) may be recyclised to the glyoxalino-compound (I; R = Ph, R′ = R″ = Me) by heating it in nitrobenzene under reflux. Identical compounds (III) have been obtained by treating anilino*apo*safranine with appropriate primary alkyl- and *cyclo*alkylamines. For example, compound (III; R = Ph, R‴ = Pr^i) has been obtained from the glyoxalino-compound (I; R = Ph, R′ = R″ = Me) and also by heating anilino*apo*safranine with *iso*propylamine.

Further, heating anilino*apo*safranine (IV; R = Ph) with aniline yields the compound

[1] Part IV, Barry, Belton, O'Sullivan, and Twomey, *J.*, 1956, 3347.
[2] *Idem*, *J.*, 1956, 896.
[3] Kehrmann and Cordone, *Ber.*, 1913, **46**, 3009.

(III; R = R‴ = Ph) which has been synthesised unambiguously by condensation of the amine (VI; R = Ph) with the quinone imine (VII; R = Ph). By analogy, therefore, it may be concluded that primary amines react with anilino*apo*safranines replacing the =NH by =NR. The absence of the =NH group from the substituted compounds (III) is also supported by their failure to react with 1-fluoro-2 : 4-dinitrobenzene.

Compound (III; R = R‴ = Ph) has also been obtained by treating glyoxalino-compounds (I; R = Ph, R′ = R″ = Me; and R = Ph, CR′R″ = *cyclo*hexane) with aniline under reflux, and under similar conditions from the substituted imines (III; R‴ = alkyl or *cyclo*alkyl). It has also been obtained by refluxing the isomer (V; R = Ph) of anilino*apo*safranine with aniline, and thus this compound has now been made by five methods.

Certain substituted imines, *e.g.*, (III; R = Ph, R‴ = Me), not accessible by the glyoxaline route, have been prepared by reaction of anilino*apo*safranines (IV) with primary aliphatic amines. The compound (III; R = Ph, R‴ = Me) was also obtained by direct alkylation of compound (IV; R = Ph). Phenethylamine reacted as expected with anilino*apo*safranine. On the other hand, benzylamine gave a product which did not have the properties expected for compound (III; R = Ph, R‴ = CH_2Ph): this product was unstable, showed a bright green fluorescence, had unique colour reactions, and failed to yield a crystalline material on hydrogenation; the analytical data fit the requirements of either the substituted imine or the glyoxalino-compound.

Compound (III; R = Ph, R‴ = CH_2·OH) is readily obtained by warming gently an ethanolic solution of anilino*apo*safranine (IV; R = Ph) with formaldehyde; it is unstable and decomposes in warm ethanol.

The reaction of anilino*apo*safranines (IV) with primary aromatic amines yielded some unexpected results. Thus, while heating compound (IV; R = Ph) with aniline or *p*-chloroaniline yielded the substituted imines (III; R = Ph, R‴ = Ph or *p*-C_6H_4Cl), the compound (IV; R = *p*-C_6H_4Cl) on treatment with aniline gave a product which, although melting sharply and behaving on an alumina column as a single substance, is shown by analytical data to be a mixture of mono- and di-chlorinated derivatives of structure

TABLE 1. 5 : 1′ : 2′ : 2′-*Substituted* 5 : 2′-*dihydroglyoxalino*(5′ : 4′-2 : 3)*phenazines* (I).

		Found (%)					Required (%)			
CR′R″	M. p.*	C	H	N	Cl	Formula	C	H	N	Cl
Compounds where R = Ph.										
CMe·CH_2·CO·NEt_2	168—170°	76·1	6·2	14·0	—	$C_{32}H_{31}ON_5$	76·6	6·4	14·0	—
CMe·CH_2·COMe	174—175	78·4	5·5	12·3	—	$C_{29}H_{24}ON_4$	78·4	5·4	12·6	—
*cyclo*Heptane	296—297	81·6	6·2	12·3	—	$C_{31}H_{28}N_4$	81·6	6·1	12·3	—
4-Methyl*cyclo*hexane ...	317—318	81·5	6·4	12·2	—	$C_{31}H_{28}N_4$	—	—	—	—
3 : 5-Dimethyl*cyclo*-hexane	288—289	81·7	6·5	11·9	—	$C_{32}H_{30}N_4$	81·7	6·4	11·9	—
Compounds where R = *p*-C_6H_4Cl.										
CMeEt	174—176	69·5	4·7	11·7	14·5	$C_{28}H_{22}N_4Cl_2$	69·3	4·5	11·5	14·6
CEt_2	179—181	69·9	4·9	11·4	14·4	$C_{29}H_{24}N_4Cl_2$	69·7	4·8	11·2	14·2
*cyclo*Heptane	290—292	71·0	5·0	10·4	13·7	$C_{31}H_{26}N_4Cl_2$	70·9	5·0	10·7	13·5
4-Methyl*cyclo*hexane ...	277—279	70·8	4·7	10·7	13·6	$C_{31}H_{26}N_4Cl_2$	—	—	—	—
3 : 5-Dimethyl*cyclo*-hexane	267—269	71·0	5·2	10·6	13·5	$C_{32}H_{28}N_4Cl_2$	71·2	5·2	10·4	13·2
Compound where R = *o*-C_6H_4Cl.										
CMe_2	217—218	68·6	4·2	11·9	14·8	$C_{27}H_{20}N_4Cl_2$	68·8	4·2	11·9	15·1

* All the compounds in this Table were recrystallised from benzene.

(VIII; R = R″ = *p*-C_6H_4Cl, R′ = Ph; and R = *p*-C_6H_4Cl, R′ = R″ = Ph). This indicates that the *p*-chloroaniline residue in the 2-position is to some extent replaceable by aniline under these conditions.

A similar result was obtained with the isomers (V) of anilino*apo*safranines. Compound (V; R = Ph) on treatment with aniline gave compound (III; R = R‴ = Ph) in high

TABLE 2. N³-*Substituted anilino*apo*safranine derivatives* (III).

		Found (%)					Required (%)			
R‴	M. p.*	C	H	N	Cl	Formula	C	H	N	Cl
Compounds where R = Ph.										
Me	180—181°	79·4	5·5	14·6	—	$C_{25}H_{20}N_4$	79·8	5·3	14·9	—
Et	182—183	79·8	5·8	13·9	—	$C_{26}H_{22}N_4$	80·0	5·6	14·4	—
Pr^n	173—175	80·2	5·9	13·9		$C_{27}H_{24}N_4$	80·2	5·9	13·9	—
Bu^n	147—149	80·2	6·5	13·5	—	$C_{28}H_{26}N_4$	80·4	6·2	13·4	—
Bu^i	160—161	80·6	6·4	13·4	—	$C_{28}H_{26}N_4$				
Bu^t	189—190	80·3	6·0	13·5	—	$C_{28}H_{26}N_4$				
n-C_5H_{11}	143—145	80·4	6·5	13·3	—	$C_{29}H_{28}N_4$	80·6	6·5	13·0	—
n-C_7H_{15}	130—131	80·8	6·7	12·2	—	$C_{31}H_{32}N_4$	80·9	7·0	12·2	—
3 : 5 : 5-Trimethylhexyl	131—133	81·2	7·5	—	—	$C_{33}H_{36}N_4$	81·2	7·4	—	—
n-$C_{10}H_{21}$	92—94	81·1	7·8	11·2	—	$C_{34}H_{38}N_4$	81·3	7·6	11·2	—
n-$C_{12}H_{25}$	95—96	81·1	8·0	10·3	—	$C_{36}H_{42}N_4$	81·5	7·9	10·6	—
3 : 5-Dimethyl*cyclo*hexyl	202—204	81·0	6·7	11·8	—	$C_{32}H_{32}N_4$	81·3	6·8	11·9	—
4-Methyl*cyclo*hexyl	175—177	81·0	6·5	12·1	—	$C_{31}H_{30}N_4$	81·2	6·6	12·2	—
*cyclo*Heptyl	191—192	81·1	6·6	12·0	—	$C_{31}H_{30}N_4$				
CH_2·OH	154—156	76·5	5·3	13·9	—	$C_{25}H_{20}ON_4$	76·5	5·1	14·3	—
CH_2·CH_2·OH	179—180	76·6	5·3	14·0	—	$C_{26}H_{22}ON_4$	76·8	5·4	13·8	—
CH_2·CH_2·NEt_2	137—139	77·0	6·7	14·4	—	$C_{30}H_{31}N_5$ †	77·0	6·9	14·3	—
CH_2·CH_2Ph	166—168	82·2	5·6	12·1	—	$C_{32}H_{26}N_4$	82·4	5·6	12·0	—
p-C_6H_4Cl	235—237	76·0	4·4	11·6	7·8	$C_{30}H_{21}N_4Cl$	76·2	4·4	11·9	7·5
Compounds where R = *p*-C_6H_4Cl.										
Me	224—225	66·8	4·1	12·1	15·8	$C_{25}H_{18}N_4Cl_2$	67·4	4·0	12·6	16·0
Et	193—195	68·0	4·5	12·0	15·4	$C_{26}H_{20}N_4Cl_2$	68·0	4·4	12·2	15·5
Pr^n	200—201	68·4	4·7	11·9	15·0	$C_{27}H_{22}N_4Cl_2$	68·5	4·7	11·8	15·0
Bu^n	182—184	68·3	4·8	11·4	14·5	$C_{28}H_{24}N_4Cl_2$	69·0	4·9	11·5	14·6
Bu^s	199—201	69·1	5·1	11·7	—	$C_{28}H_{24}N_4Cl_2$				
Bu^i	183—185	68·9	5·1	11·6	14·7	$C_{28}H_{24}N_4Cl_2$				
n-C_5H_{11}	146—148	69·6	5·1	11·2	14·1	$C_{29}H_{26}N_4Cl_2$	69·5	5·2	11·2	14·2
$CHEt_2$	215—217	69·6	5·4	11·3	13·9	$C_{29}H_{26}N_4Cl_2$				
n-C_6H_{13}	173—175	69·7	5·5	11·0	13·9	$C_{30}H_{28}N_4Cl_2$	69·9	5·4	10·9	13·8
n-$C_{10}H_{21}$	121—123	71·0	6·4	—	12·3	$C_{34}H_{36}N_4Cl_2$	71·5	6·3	9·8	12·4
n-$C_{12}H_{25}$	98—100	71·9	6·7	9·5	11·9	$C_{36}H_{40}N_4Cl_2$	72·1	6·7	9·3	11·9
4-Methyl*cyclo*hexyl	237—239	70·6	5·5	10·6	13·2	$C_{31}H_{28}N_4Cl_2$	70·6	5·3	10·6	13·5
*cyclo*Heptyl	234	70·8	5·1	10·5	13·4	$C_{31}H_{28}N_4Cl_2$	—	—	—	—
3 : 5-Dimethyl*cyclo*hexyl	218—220	70·4	5·8	10·5	13·2	$C_{32}H_{30}N_4Cl_2$	71·0	5·5	10·4	13·1
CH_2·CH_2·OH	207—208	65·6	4·4	12·0	14·8	$C_{26}H_{20}ON_4Cl_2$	65·7	4·2	11·8	14·9
CH_2·CH_2·NEt_2	158—160	67·4	5·3	13·2	13·4	$C_{30}H_{29}N_5Cl_2$	67·9	5·5	13·2	13·4
p-C_6H_4Cl	275—277	66·5	3·3	9·9	20·0	$C_{30}H_{19}N_4Cl_3$	66·5	3·5	10·3	19·7
Compound where R = *o*-C_6H_4Cl.										
Pr^i	193—195	68·0	4·5	11·8	15·2	$C_{27}H_{22}N_4Cl_2$	68·5	4·7	11·8	15·0
Compound where R = *p*-C_6H_4·OMe.										
p-C_6H_4·OMe	219—221	75·3	5·4	10·7	—	$C_{33}H_{28}O_3N_4$	75·0	5·3	10·6	—

* All the compounds in this Table were recrystallised from ethanol. † Solvate, $+\frac{1}{2}C_2H_5$·OH. The following ten compounds (III) have already been characterised (Table 2, Part IV [1]) but were formulated incorrectly: (III; R = Ph, and R‴ = Pr^i, Bu^s, $CHEt_2$, $CHMeBu^i$, *cyclo*hexyl or, CHMe·$[CH_2]_2$·OH); (III; R = *p*-C_6H_4Cl, and R‴ = Pr^i or *cyclo*hexyl); (III; R = *p*-C_6H_4Me, R‴ = Pr^i); (III; R = *p*-C_6H_4·OPr^i, R‴ = *cyclo*hexyl).

TABLE 3.

		Found (%)					Required (%)			
R′ = R″	M. p.*	C	H	N	Cl	Formula	C	H	N	Cl
Compounds (VIII; R = Me).										
Ph	205—206°	79·5	5·6	15·0	—	$C_{25}H_{20}N_4$	79·8	5·3	14·9	—
p-C_6H_4Cl	245—246	67·3	4·1	12·6	16·0	$C_{25}H_{18}N_4Cl_2$	67·4	4·0	12·6	16·0
Compounds (VIII; R = CH_2·CH_2·NEt_2).										
Ph	146—147	78·2	6·9	15·3	—	$C_{30}H_{31}N_4$	78·1	6·7	15·2	—
p-C_6H_4Cl	168—169	67·8	5·5	13·4	13·6	$C_{30}H_{29}N_4Cl_2$	67·9	5·5	13·2	13·4
Compounds (VIII; R = *cyclo*hexyl).										
Ph	185—186	80·9	6·3	12·7	—	$C_{30}H_{28}N_4$	81·1	6·3	12·6	—
p-C_6H_4Cl	198	70·2	5·4	10·7	13·9	$C_{30}H_{26}N_4Cl_2$	70·1	5·1	10·9	13·8
Compound (VIII; R = *p*-C_6H_4Cl).										
Ph	205—206	75·8	4·5	12·3	7·8	$C_{30}H_{21}N_4Cl$	76·2	4·4	11·9	7·5

* All compounds recrystallised from ethanol.

yield, but with *p*-chloroaniline a mixture similar to that described above was obtained. Again, treatment of compound (V; R = *p*-C_6H_4Cl) with aniline gave a mixture of mono- and di-chlorinated derivatives of sharp m. p., and not separable on alumina. When *p*-chloroaniline was used instead of aniline in this reaction, a single trichlorinated compound (III; R = R''' = *p*-C_6H_4Cl) resulted. It is clear, therefore, that compounds of structure (III) may be readily obtained in a pure state from structures (IV and V) when the reacting arylamine is the same as that already substituting in the 2- or 3-position of the phenazine nucleus.

The amine (V; R = Ph) does not react with alkyl- or *cyclo*alkyl-amines under reflux. At higher temperatures *cyclo*hexylamine reacts with compound (V; R = Ph), yielding compound (VIII; R = Ph, R' = R'' = *cyclo*hexyl). A similar result was obtained with the analogue (V; R = *p*-C_6H_4Cl).

Condensation of *o*-phenylenediamines and 4 : 5-diarylamino-*o*-quinones yields compounds of structure (VIII; R' = R'' = aryl, R = aryl, alkyl, *cyclo*alkyl, etc.) which, when R differs from R', are not accessible by any of the other routes described. In the preparation of the 4 : 5-diarylamino-*o*-quinones we have found sodium iodate to be a much more effective reagent than silver oxide.[3]

New glyoxalinophenazines are listed in Table 1 and the substituted imines in Tables 2 and 3. A preliminary account of the biological properties of these substituted imines has already been published.[4]

Experimental

2-*Anilino*-3 : 5-*dihydro*-3-*methylimino*-5-*phenylphenazine*.—(a) *With methylamine*. Anilino-*apo*safranine hydrochloride (4·0 g.) and 40% aqueous methylamine (20 c.c.) in ethanol (25 c.c.) were heated in a sealed tube at 130° for 3 hr. The dark red solid, recrystallised from ethanol (2·7 g.), had m. p. 180—181° (Found: C, 79·4; H, 5·5; N, 14·6. $C_{25}H_{20}N_4$ requires C, 79·8; H, 5·3; N, 14·9%). This *compound* has also been obtained, with m. p. 188—190°, from benzene. Its solution in concentrated sulphuric acid is petunia-purple, unchanged on dilution.

(b) *With methyl sulphate*. Anilino*apo*safranine (0·7 g.), methyl sulphate (0·7 c.c.), and 10% aqueous sodium hydroxide (3 c.c.) were heated under reflux in acetone (20 c.c.) for 4 hr. The solvent was removed and the product purified chromatographically in benzene on alumina. The product (0·3 g.) was identical with that obtained by method (a).

2-*Anilino*-3 : 5-*dihydro*-3-*hydroxymethylimino*-5-*phenylphenazine*.—Anilino*apo*safranine (0·6 g.), paraformaldehyde (1·6 g.), and ethanol (80 c.c.) were boiled for 3 min. The filtrate yielded orange yellow crystals (0·3 g.), m. p. 154—156° (from benzene) (Found: C, 76·5; H, 5·3; N, 13·9. $C_{25}H_{20}ON_4$ requires C, 76·5; H, 5·1; N, 14·3%).

2-*Anilino*-3 : 5-*dihydro*-5-*phenyl*-3-*phenyliminophenazine*.—This compound [3] was prepared by heating the hydrochloride of each of the following with aniline under reflux for 10 min.: (*a*) anilino*apo*safranine, (*b*) 2-amino-3 : 5-dihydro-5-phenyl-3-phenyliminophenazine, (*c*) 5 : 2'-dihydro-2' : 2'-dimethyl-5 : 1'-diphenylglyoxalino(5' : 4'-2 : 3)phenazine, (*d*) 2-anilino-3-*cyclo*hexylimino-3 : 5-dihydro-5-phenylphenazine. The cooled solutions were poured into ether and the precipitated salt converted into the base in each case. After chromatographic purification the product had m. p. 235—237° (from benzene). Yields were: from (*a*) and (*b*), 80%; from (*c*) and (*d*), 40%. The same compound was prepared by the condensation of 2-aminodiphenylamine hydrochloride and 4 : 5-dianilino-*o*-quinone.

4 : 5-*Di*-p-*chloroanilino*-o-*quinone*.—Catechol (4·4 g.) and *p*-chloroaniline (10·2 g.) were dissolved in ethanol (150 c.c.), and sodium iodate (8·0 g.) in water (150 c.c.) added. The mixture was stirred for 2 hr., then kept overnight; the dark red *quinone* (14·4 g.) which separated had m. p. 222—224° (from chloroform) (Found: C, 60·5; H, 3·4; N, 8·0; Cl, 20·0. $C_{18}H_{12}O_2N_2Cl_2$ requires C, 60·2; H, 3·3; N, 7·8; Cl, 19·8%).

4 : 5-*Di*-p-*toluidino*-o-*quinone*.—Prepared as above, this *quinone* formed bright red needles, m. p. 190—192°, from benzene (Found: C, 75·7; H, 5·7; N, 9·0. $C_{20}H_{18}O_2N_2$ requires C, 75·5; H, 5·7; N, 8·8%). The yield was theoretical.

2-p-*Chloroanilino*-3-p-*chlorophenylimino*-3 : 5-*dihydro*-5-*phenylphenazine*.—4 : 5-Di-*p*-chloroanilino-*o*-quinone (3·6 g.) and *N*-phenyl-*o*-phenylenediamine hydrochloride (2·2 g.) were heated

[4] Barry, Belton, Conalty, Denneny, Edward, O'Sullivan, Twomey, and Winder, *Nature*, 1957, **179**, 1013.

in ethanol (150 c.c.) under reflux for 1 hr. The mixture was made alkaline with ammonia and diluted with water. The *product* (2·3 g.) after chromatographic purification was obtained as dark red needles (from benzene), m. p. 241—242° (Found: C, 71·3; H, 3·7; N, 10·8; Cl, 14·0. $C_{30}H_{20}N_4Cl_2$ requires C, 71·0; H, 3·9; N, 11·0; Cl, 14·0%).

2-cyclo*Hexylamino*-3-cyclo*hexylimino*-3 : 5-*dihydro*-5-*phenylphenazine*.—2-Amino-3 : 5-dihydro-5-phenyl-3-phenyliminophenazine hydrochloride (3·0 g.) and *cyclo*hexylamine (35 c.c.) were heated in the autoclave at 185° for 1 hr. Unchanged *cyclo*hexylamine was removed by steam-distillation and the residual solid purified chromatographically on alumina. The *compound* (1·8 g.) was an orange powder, m. p. 188—190° (from ethanol) (Found: C, 79·9; H, 7·6; N, 12·8. $C_{30}H_{34}N_4$ requires C, 80·0; H, 7·6; N, 12·4%).

5-p-*Chlorophenyl*-2-cyclo*hexylamino*-3-cyclo*hexylimino*-3 : 5-*dihydrophenazine*.—2-Amino-5-*p*-chlorophenyl-3-*p*-chlorophenylimino-3 : 5-dihydrophenazine was treated with cyclo*hexyl*amine as in the previous experiment. The *product* was a brown-red powder, m. p. 196—198° (from ethanol) (Found: C, 73·8; H, 6·5; N, 11·5. $C_{30}H_{33}N_4Cl$ requires C, 74·3; H, 6·8; N, 11·6%).

2-p-*Chloroanilino*-5-p-*chlorophenyl*-3 : 5-*dihydro*-3-iso*propyliminophenazine*.—2-*p*-Chloroanilino-5-*p*-chlorophenyl-3 : 5-dihydro-3-iminophenazine hydrochloride (2·0 g.), *iso*propylamine (20 c.c.), and ethanol (40 c.c.) were heated for 2 hr. in the autoclave at 80°. The product, purified chromatographically, was identical with that obtained by catalytic hydrogenation of 5 : 1′-di-*p*-chlorophenyl-5 : 2′-dihydro-2′ : 2′-dimethylglyoxalino(5′ : 4′-2 : 3)phenazine.

2-p-*Chloroanilino*-5-p-*chlorophenyl*-3-cyclo*hexylimino*-3 : 5-*dihydrophenazine*.—2-*p*-Chloroanilino-5-*p*-chlorophenyl-3 : 5-dihydro-3-iminophenazine hydrochloride (0·2 g.) was heated under reflux with *cyclo*hexylamine (15 c.c.) for 30 min. The product (0·15 g.), purified chromatographically, was identical with that obtained by catalytic hydrogenation of 5 : 1′-di-*p*-chlorophenyl*cyclo*hexane*spiro*-2′-glyoxalino(5′ : 4′-2 : 3)phenazine.

Recyclisation of Compound (III; R = Ph, R‴ = Pr^i) *to the Glyoxalinophenazine* (I; R = Ph, R′ = R″ = Me).—The compound (III) (0·5 g.), concentrated hydrochloric acid (0·25 c.c.), and nitrobenzene (25 c.c.) were heated under reflux for 20 min. The cooled solution was poured into ether and the precipitated hydrochloride converted into the base which was purified chromatographically; this had m. p. and mixed m. p. 230—232° (yield, 0·07 g.). The compound in concentrated sulphuric acid is rose-red, unchanged on dilution.

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