HETEROCYCLIC ANALOGS OF PLEIADIENE

XXVIII.* REACTION OF 1,8-NAPHTHALENEDIAMINE WITH AROMATIC AND HETEROAROMATIC ALDEHYDES. SYNTHESIS OF 2-SUBSTITUTED PERIMIDINES AND 2-R-2,3-DIHYDROPERIMIDINES

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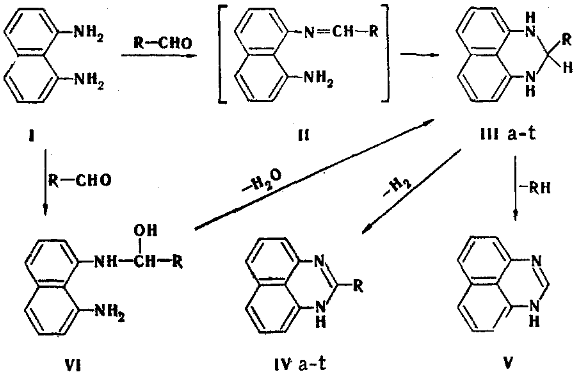
1,8-Naphthalenediamine reacts with aromatic and heteroaromatic aldehydes under mild conditions to give 2-R-2,3-dihydroperimidines in high yields. In contrast to aromatic o-diamines, Schiff bases cannot be isolated in these reactions. Some of the 2-R-2,3-dihydroperimidines are stabilized to give 2-R-perimidines (in the case of oxidation) or perimidine itself (in the case of splitting out of RH) when the reaction is carried out under more severe conditions (by refluxing in xylene). Catalytic dehydrogenation makes it possible to convert the 2-R-2,3-dihydroperimidines to various 2-R-perimidines.

A number of studies have been devoted to the reaction of 1,8-naphthalenediamine (I) with aromatic and heteroaromatic aldehydes [2-6]. A relatively limited number of aldehydes have been used in these studies, and the corresponding 2-substituted 2,3-dihydroperimidines (III) were obtained in all cases:



The formation of aromatized perimidines has been noted only in [5] in the case of the reaction of I with ortho- and peri-substituted dialdehydes (for example, VII in the case of phthalaldehyde). This reaction pathway can be explained by the possibility of hydride migration in the intermediately formed 2,3-dihydroperimidine, which is accompanied by reduction of the second aldehyde group to an alcohol with subsequent splitting out of a molecule of water and intramolecular cyclization.

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* See [1] for communication XXVII.

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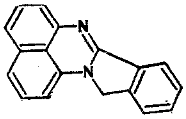

VII

The aim of the present research was to study the reaction of I with the largest possible number of aromatic and heteroaromatic aldehydes and the effect of variation of the reaction conditions. It was assumed that this may lead to the detection of new reaction pathways and the isolation of heretofore undescribed Schiff bases from aromatic peri-diamines (II).

We found that I reacts readily with most aromatic and heteroaromatic aldehydes in alcohol solution even at room temperature to give the corresponding 2-substituted 2,3-dihydroperimidines (IIIa-t) in almost quantitative yields. The R groups are presented in Table 2. The reaction proceeds at an appreciable rate when the reaction mixture is heated and also leads exclusively to III. In this case the use of cyclization catalysts of any kind and removal of the water formed in the reaction are not necessary (see [7]).

The absence of the color characteristic for Schiff bases or for the perimidines themselves in the products indicates structure III. Only the compounds with a nitro group (IIIe,s,t) are orange, but the IR and PMR spectral data also make it possible to exclude a Schiff base structure for them. The IR spectra of dilute solutions of III in $CHCl_3$ or CCl_4 contain one ν_{NH} band at 3380-3395 cm^{-1}. In the case of crystalline samples, in which there is no possibility for intramolecular hydrogen bonding (as in the case of IIIa-f,h-j,l-r,t), the ν_{NH} band is split into a doublet, but the distance between the peaks of the doublet does not correspond to a formula linking the bands of the symmetrical and asymmetrical stretching vibrations of the amino group [8]. The latter makes it possible to exclude an azomethine structure for crystalline samples of III.

The signal of an azomethine proton that usually appears at 8-9 ppm is absent in the PMR spectra of the synthesized compounds (Table 1). The spectrum also contains a singlet at ~5.5 ppm, which, as in the case of other 2,3-dihydroperimidines, corresponds to the proton attached to the μ-carbon atom. The H_4 and H_9 protons in the naphthalene ring show up in the form of the quartet (δ 6.3-6.4 ppm, $J_o = 4.7, J_m = 3.5$ Hz) that is characteristic for structures of this type [8] and are chemically equivalent; this could not occur in the case of the azomethine.

Thus it can be concluded that either the azomethines of the II types, in contrast to azomethines based on aromatic o-diamines [10, 11], are unstable because of peri interaction [12], which leads to rapid cyclization of II to III, or that the formation of III takes place with bypassing of the step involving the formation of a Schiff base altogether. For example, one may imagine that the initially formed carbinol VI undergoes immediate cyclization to dihydroperimidine III with splitting out of a molecule of water. In fact, we were unable to obtain an azomethine when we heated 1-amino-8-dimethylaminonaphthalene with benzaldehyde under a broad range of conditions (we will report the results of the reaction of N-substituted 1,8-naphthalenediamines with aldehydes separately). According to the data obtained in our laboratory, 1-formyl-8-hydroxynaphthalene also does not form azomethines when it is heated with aromatic amines.

During a study of the reaction of I with aldehydes under more severe conditions (in refluxing xylene) we observed two other types of transformations. In the first case perimidine V and the corresponding deformylated heterocycle are unexpectedly formed instead of IIIi,f,g,k when 1,8-naphthalenediamine is refluxed in xylene with aldehydes of π-surplus heteroaromatic systems (indole, pyrrole, N-methylimidazole, and N-methylbenzimidazole). We assumed that under these conditions the intermediately formed IIIr,g,i-k are stabilized by loss of an RH molecule, as in the case of other dihydroquinoxalines [13, 14], rather than by splitting out of a hydrogen molecule. This assumption was confirmed. It was found that when genuine samples of IIIf,g,i-k are heated in xylene with a π-surplus heteryl system, they undergo dissociation at different rates (for example, the 3-indolyl derivative reacts faster than the 2-pyrrolyl derivative) to give the perimidine. This reaction proceeds considerably more rapidly and gives the product in quantitative yield in glacial acetic acid or when the mixture is heated. On the other hand, IIIq,r, which have hydroxyphenyl substituents, can be aromatized only when they are fused. The other 2,3-dihydroperimidines obtained in this research (IIIa-e, h,l,m-p,s,t) were completely stable with respect to thermolysis.

We observed a second anomaly during a study of the reaction of I with 2-formylquinoline. We found that if this reaction is carried out in refluxing xylene, an aromatized 2-α-quinolylperimidine (IVl), which differs from the dihydro derivative with respect to its bright-red color, is formed in up to 70% yield. This transformation also occurs in an inert atmosphere, i.e., dehydrogenation occurs without the aid of atmospheric oxygen. 2-α-Quinolyl-2,3-dihydroperimidine (IIIl) also undergoes aromatization under these conditions. It might be

TABLE 1. Chemical Shifts of Some Protons in the PMR Spectra of IIIa,n,s (on the δ scale with respect to hexamethyldisiloxane)

Compound	Solvent	H_2	$H_{4,9}$	NH
IIIa	$CDCl_3$	5,48 s	6,43 q	**4,35**
IIIn	$CDCl_3$	5,32 s	6,33 q	**4,35**
IIIs	CF_3COOH	5,73 s	6,80 q	—

TABLE 2. 2-R-2,3-Dihydroperimidines (IIIa-t)

Compounds III	R	mp, °C (from alcohol)	ν_{NH}, cm^{-1}		Found, %			Empirical formula	Calc., %			Yield, %
			in $CHCl_3$	in mineral oil	C	H	N		C	H	N	
a	2-Furyl	100—101	3380	—	76,3	5,1	11,9	$C_{15}H_{12}N_2O$	76,2	5,2	11,8	90
b	2-Methyl-5-furyl	123	3385	3270 3330	76,7	5,3	11,3	$C_{16}H_{14}N_2O$	76,7	5,6	11,2	96
c*	2-Chloro-5-furyl	134	—	3360 3410	—	—	—	$C_{15}H_{11}ClN_2O$	—	—	—	92
d*	2-Iodo-5-furyl	109	—	3360 3400	—	—	—	$C_{15}H_{11}IN_2O$	—	—	—	90
e*	2-Nitro-5-furyl	165—166	—	3260 3355	—	—	—	$C_{15}H_{11}N_3O_3$	—	—	—	90
f	2-Pyrrolyl	190—191	—	3315 3325	76,6	5,5	17,8	$C_{15}H_{13}N_3$	76,6	5,6	17,9	85
g	1-Methyl-2-imidazolyl	185—186	3390	3260	71,9	5,6	22,4	$C_{15}H_{14}N_4$	72,0	5,6	22,4	86
h	2-Thienyl	120—121	3390	3330 3350	71,5	4,8	11,1	$C_{15}H_{12}N_2S$[d]	71,4	4,8	11,1	95
i	3-Indolyl	68—69[b]	—	3285 3325	80,1	5,2	14,8	$C_{19}H_{15}N_3$	80,0	5,3	14,7	80
j	1-Methyl-3-indolyl	194—195[b]	—	3375 3395	80,4	5,7	14,2	$C_{20}H_{17}N_3$	80,1	5,7	14,1	95
k	1-Methyl-2-benzimidazo.	203—204	3390	3300	75,8	5,5	18,5	$C_{19}H_{16}N_4$	76,0	5,4	18,6	60
l	2-Quinolyl	138—139	3380	3295 3340	80,7	5,2	14,3	$C_{20}H_{15}N_3$	80,8	5,1	14,1	83
m	3-Pyridyl	158—159	—	3173 3245	77,4	5,2	17,0	$C_{16}H_{13}N_3$	77,7	5,3	17,0	90
n	C_6H_5	102[c]	3395	3350 3370	82,8	5.6	11,3	$C_{17}H_{14}N_2$	82,9	5,7	11,4	95
o	p-BrC_6H_4	139	—	3350 3380	62,7	4,1	8,7	$C_{17}H_{13}BrN_2$	62,8	4,0	8.6	95
p	2,4-$Cl_2C_6H_3$	134	—	3350 3395	65,0	4,1	8,5	$C_{17}H_{12}Cl_2N_2$	64,8	3,8	8,9	90
q	p-HOC_6H_4	160—161	—	3285 3385	77,9	5,5	10,7	$C_{17}H_{14}N_2O$	77,9	5,4	10,7	93
r	2,4-$C_6H_3(OH)_2$	148—149	—	3305 3370	73,8	5,3	10,1	$C_{17}H_{14}N_2O_2$	73,4	5,1	10,1	85
s	p-$NO_2C_6H_4$	200 (dec.)	—	3355	70,2	4,4	14,5	$C_{17}H_{13}N_3O_2$	70,1	4,5	14.4	95
t	m-$NO_2C_6H_4$	182—183	3380	3222 3335	70,3	4,5	14,6	$C_{17}H_{13}N_3O_2$	70,1	4,5	14,4	95

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*See [6] for the synthesis of IIIc-e. [b] From butanol. [c] According to the data in [4], this compound has mp 128°C. We obtained a compound with the same melting point by refluxing the compound with mp 102° in alcohol with activated charcoal. The product was slightly yellowish, and its IR and PMR spectra were identical to those of the low-melting compound. On the basis of this it can be concluded that the two substances are crystalline modifications of 2-phenyl-2,3-dihydroperimidine. [d] Found: S 12.5%. Calculated: S 12.7%.

assumed that the dehydrogenation of the perimidine ring proceeds with the assistance of the quinoline ring, which, according to polarographic data, [15], has a considerably lower reduction potential than the perimidine ring. We will study this interesting problem in greater detail in the case of other III that contain substituents with lower reduction potentials.

EXPERIMENTAL

The IR spectra of IIIa-t and IVa-t were obtained with a UR-20 spectrometer. The PMR spectra were recorded with a Tesla spectrometer to 80 MHz with hexamethyldisiloxane as the internal standard.

2-R-2,3-Dihydroperimidines (IIIa-h,k-t). A saturated alcohol solution of the appropriate aldehyde (0.1 mole) was added to a solution of 15.8 g (0.1 mole) of 1,8-naphthalenediamine in 100 ml of alcohol, and the resulting solution was allowed to stand at room temperature for 1-3 h (the completeness of the reaction was monitored by chromatography), after which the precipitated crystals of the dihydroperimidine were removed by filtration, washed with a small amount of alcohol, and crystallized. The characteristics of IIIa-h,k-t are given in Table 2.

2-(3-Indolyl)-2,3-dihydroperimidine (IIIi) and 2-(N-Methyl-3-indolyl)-2,3-dihydroperimidine (IIIj). A mixture of 0.1 mole of I, 0.1 mole of 3-formylindole or N-methyl-3-formylindole, and 300 mole of butyl alcohol was refluxed for 5 h, after which it was cooled, and the precipitated IIIi,j were removed by filtration and washed with a small amount of butanol. The reaction of I with 3-formylindole and N-methyl-3-formylindole did not take place in ethanol even when the mixtures were refluxed, and a slow reaction was observed in propanol.

Reaction of 1,8-Naphthalenediamine with Aldehydes of π-Surplus Heteroaromatic Systems in Refluxing Xylene. A mixture of 15.8 g (0.1 mole) of 1.8-naphthalenediamine, 14.5 g (0.1 mole) of 3-formylindole, and 200 ml of xylene was refluxed for 15 h, the resulting solution was cooled, and the precipitated crystals of perimidine V were removed by filtration, washed, and dried. The yield of V was close to quantitative. The melting point and IR spectrum were close to the melting point and IR spectrum of an authentic sample [17].

The mother liquor from the separation of V was evaporated, and the residue was dissolved in chloroform. The indole was isolated by chromatography of the solution of aluminum oxide.

The reactions of I with 2-formylpyrrole, N-methyl-2-formylimidazole, and N-methyl-2-formylbenzimidazole in refluxing xylene were carried out similarly to give perimidine V and, respectively, pyrrole, N-methylimidazole, and N-methylbenzimidazole.

Reaction of 2-R-2,3-Dihydroperimidines (IIIf,g,i-k) with Glacial Acetic Acid. A solution of 0.1 mole of 2-R-2,3-dihydroperimidine (IIIf,g,i-k) in 100 ml of glacial acetic acid was refluxed for 15 min, during which the colorless solution turned yellow-green. The mixture was then cooled and diluted with a twofold volume of water, and the aqueous mixture was neutralized with ammonia. The precipitated crystals of V were removed by filtration and washed with water to give the product in quantitative yield.

2-(5-Nitro-2-furyl)perimidine (IVe). A 0.01-mole sample of dihydroperimidine IIIe, 0.1 g of Pd/C, and 50 ml of nitrobenzene were placed in a three-necked flask equipped with a mechanical stirrer, a thermometer, and a reflux condenser, and the contents of the flask were stirred at 115° for 24 h, after which the mixture was allowed to stand at room temperature overnight. The precipitated crystals of IVe were removed along with the catalyst by filtration, and the mixture of solids was extracted with acetone. The extract was refluxed with activated charcoal and filtered, and the filtrate was diluted with water. The precipitated violet crystals of IVe were removed by filtration and dried to give the product in 15-30% yield. Resinification was observed when the temperature was raised, and the reaction did not take place at lower temperatures.

2-(5-Methyl-2-furyl)perimidine (IVb). A mixture of 2.5 g (0.01 mole) of dihydroperimidine IIb, 50 ml of xylene, and 0.1 g of Pd/C was refluxed for 2 h, and the resulting hot solution was filtered. The filtrate was cooled, and the precipitated crystals of IVb were removed by filtration and washed with xylene. Compounds IVh,o-r were similarly obtained.

2-(5-Chloro-2-furyl)perimidine (IVc). A mixture of 2.7 g (0.01 mole) of 2-(5-chloro-2-furyl)-2,3-dihydroperimidine (IIIc), 50 ml of xylene, and 0.1 g of Pd/C was refluxed for 24 h, and the resulting hot solution was filtered. The xylene was removed from the filtrate by distillation at reduced pressure [10 mm (mercury column)], the residue was dissolved in chloroform, and the solution was passed through a column filled with aluminum oxide (elution with chloroform). The first fraction was collected. Compounds IVd,f were similarly obtained. In the preparation of IVf the mixture was refluxed for 1 h.

TABLE 3. 2-R-Perimidines (IVa-t)

Compounds IV	mp, °C[a]	Color	Found, %			Empirical formula[b]	Calc., %			Yield, %
			C	H	N		C	H	N	
a[c]	—	—	—	—	—	$C_{15}H_{10}N_2O$	—	—	—	—
b	230 (dec.)	Orange	77,7	5,0	11,2	$C_{16}H_{12}N_2O$	77,4	4,9	11,3	90
c	224—225	Yellow-orange	67,2	3,4	10,3	$C_{15}H_9ClN_2O$	67,1	3,4	10,4	40
d	217—218	Yellow-orange	50,1	2,6	8,1	$C_{15}H_9IN_2O$	50,0	2,5	7,8	40
e	350 (dec.)	Violet	64,2	3,3	15,1	$C_{15}H_9N_3O_3$	64,5	3,2	15,0	15—30
f	106—107	Light yellow	77,3	4,7	18,2	$C_{15}H_{11}N_3$	77,2	4,8	18,0	60
g	182—183	Canary-yellow	72,4	5,0	22,5	$C_{15}H_{12}N_4$	72,6	4,9	22,6	70
h	165—166	Yellow-red	72,1	4,1	11,3	$C_{15}H_{10}N_2S$	72,0	4,0	11,2	85
i	148—149	Yellow-green	80,5	4,8	14,9	$C_{19}H_{13}N_3$	80,6	4,6	14,8	60
j	209—210	Yellow	80.5	5,2	14,2	$C_{20}H_{15}N_3$	80,7	5,1	14,2	80
k	202—203	Orange	76,4	4,6	18,7	$C_{19}H_{14}N_4$	76,5	4,7	18,8	40
l	290—300 (dec.)	Red	81,1	4,8	14,3	$C_{20}H_{13}N_3$	81,3	4,4	14,2	50
m[c]	—	—	—	—	—	$C_{16}H_{11}N_3$	—	—	—	—
n[c]	—	—	—	—	—	$C_{17}H_{12}N_2$	—	—	—	—
o	169—170	Orange	63.2	3,3	8,8	$C_{17}H_{11}BrN_2$	63,2	3,4	8,7	60
p	193—194 (dec.)	Orange	65,1	3,5	8,7	$C_{17}H_{10}Cl_2N_2$	65,2	3,2	8,9	60
q	200 (dec.)	Lemon-yel.	78,2	4,7	10,9	$C_{17}H_{12}N_2O$	78,4	4,7	10,8	80
r	188—189	Yellow	74,2	4,3	9,9	$C_{17}H_{12}N_2O_2$	73,9	4,4	10,1	70
s[d]	—	—	—	—	—	$C_{17}H_{11}N_3O_2$	—	—	—	—
t[d]	—	—	—	—	—	$C_{17}H_{11}N_3O_2$	—	—	—	—

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[a] The compounds were crystallized: IVb-d,f,h from aqueous alcohol, IVe,k from aqueous acetone, IVg,f from ethyl acetate, IVi from benzene—cyclohexane, IVl,j from alcohol, and IVo,p,r from toluene. [b] The type of group in IV corresponds to that in the analogous III. [c] See [3] for the synthesis of IVa,m,n from IIIa,m,n. [d] See [16] for the synthesis of IVs,t.

2-(2-Quinolyl)perimidine (IVl). A) A mixture of 7.9 g (0.05 mole) of I, 7.85 g (0.05 mole) of 2-formylquinoline, and 100 ml of xylene was refluxed for 3 h, after which the solution was cooled, and the precipitated bright-red crystals of IVl were removed by filtration, washed with xylene, and dried to give 10.4 g (70%) of product.

B) A solution of 0.05 mole of dihydroperimidine IIIl in 50 ml of xylene was refluxed for 3 h, after which it was cooled, and the precipitated crystals were removed by filtration, washed with xylene, and dried to give the product in 50% yield.

2-(N-Methyl-2-imidazolyl)perimidine (IVg). A mixture of 5 g (0.02 mole) of dihydroperimidine IIIg, 150 ml of benzene, and 0.5 g of Pd/C was refluxed for 20 h, after which activated charcoal was added, and the mixture was refluxed for 10 min. It was then filtered and cooled. Hydrogen chloride was bubbled into the filtrate, and the precipitated crystals of the hydrochloride were removed by filtration and dried. A suspension of the hydrochloride in water was treated with ammonium hydroxide until the odor of ammonia persisted, after which the crystals of the base were removed by filtration, washed to neutrality with water, and dried.

2-(N-Methyl-2-benzimidazolyl)perimidine (IVk). A mixture of 6 g (0.02 mole) of dihydroperimidine IIIk, 200 ml of toluene, and 0.5 g of Pd/C was refluxed for 10 h, after which the toluene was removed by distillation, and the residue was dissolved in chloroform. The chloroform solution was passed twice through a column filled with aluminum oxide (elution with chloroform). The first fraction was collected.

2-(n-Methyl-3-indolyl)perimidine (IVj). A mixture of 6 g (0.02 mole) of dihydroperimidine IIIj, 100 ml of xylene, and 0.5 g of Pd/C was refluxed for 1 h, and the resulting hot solution was filtered. The filtrate was cooled, and the precipitated crystals were removed by filtration, washed with xylene, and dried.

2-(3-Indolyl)perimidine (IVi). A mixture of 5.7 g (0.02 mole) of dihydroperimidine IIIi, 150 ml of toluene, and 0.5 g of Pd/C was refluxed for 10 h, after which the hot mixture was filtered, and the toluene was removed from the filtrate distillation at reduced pressure [10 mm (mercury column)]. The residue was dissolved in chloroform, and the solution was passed through a column filled with aluminum oxide. The yellow-green portion of the aluminum oxide column was cut out, and perimidine IVi was extracted from it with acetone. The acetone was evaporated, and the residue was crystallized from benzene—cyclohexane.

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HETEROCYCLIC ANALOGS OF PLEIADIENE

XXIX.* CONVENIENT METHOD FOR THE DEHYDROGENATION OF 2-R-2,3-DIHYDROPERIMIDINES

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In most cases 2-R-2,3-dihydroperimidines are smoothly dehydrogenated by sulfur in refluxing xylene to give 2-substituted perimidines in high yields. 2-(5-Halo-2-furyl)-2,3-dihydroperimidines, which form IV and 2-(5-halo-2-furyl)perimidines when they are heated with sulfur, constitute an exception.

The reaction of 1,8-naphthalenediamine (I) with carboxylic acids and their derivatives as a method for the preparation of 2-substituted perimidines is frequently complicated by side processes [2]. Diamine I reacts much more smoothly with aromatic aldehydes [1, 2], but the products of this reaction are extremely stable 2-R-2,3-dihydroperimidines (II). Palladium on carbon [2], chloranil, and manganese dioxide [3] have been proposed for their aromatization to 2-R-perimidines (III). Each of these reagents has disadvantages: high cost, difficulties involved in the isolation of the reaction products, yields that are not always good, etc. We have found a considerably simpler and cheaper method for the dehydrogenation of II, which con-

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* See [1] for communication XXVIII.

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