# Hydroxyiminoisoquinolin-3(2*H*)-ones. 9 [1]. Synthesis of some Oxazolo[5,4-*c*]-, Thiazolo[5,4-*c*]- and 2,3-Dihydro-1*H*-[1,4]oxazino[2,3-*c*]isoquinolines



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4-Acetylamino-1-phenyl-1,4-dihydro-3(2*H*)-isoquinolinones and 4-amino-1-phenylisoquinolin-3-ol, prepared from the corresponding 4-hydroxyimino compounds, were converted into new isoquinolines containing fused oxazole, thiazole and 1,4-oxazine rings.

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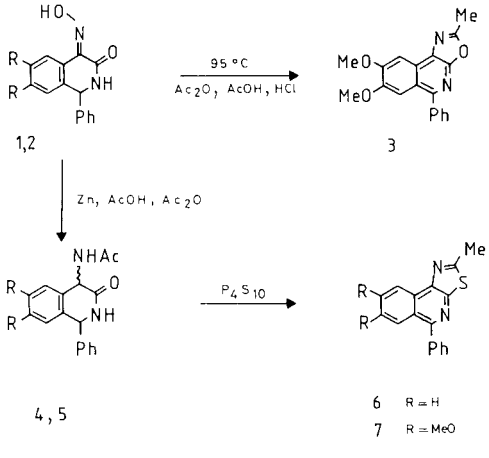
In earlier work [2] reactions of 4-hydroxyimino-1-phenyl-1,4-dihydro-3-(2*H*)-isoquinolinones with Beckmann's mixture at 95° gave the ring system **3**. The 4-acetylamino-1-phenyl-1,4-dihydro-3(2*H*)-isoquinolinones **4** and **5** prepared from the above oximes by reductive acetylation [3], reacted with phosphorus pentasulfide to yield, instead of the expected thiones, 2-methyl-5-phenylthiazolo[5,4-*c*]isoquinolines **6** and **7** involving water elimination leading to ring formation (Scheme 1). This is evidenced in the ir spectra (*cf.* Table 1) by the absence of the $\nu$ NH thioamide bands; also, in the $^1$H nmr spectra the *C*-methyl singlets appear in the chemical shift range characteristic of methyl groups attached to heteroaromatic rings (at about 2.95 ppm), while in the $^{13}$C nmr spectrum the C-1 signal (see Table 2) is also found in the range characteristic of heteroaromatics, near the downfield limit (158.0 and 156.0 ppm).

Scheme 1




As reported in the previous part of this series [1], reaction of 4-hydroxyimino-1-phenyl-1,4-dihydro-3(2*H*)-isoquinolinones with formic acid gave 4-formamido-1-phenylisoquinolin-3-ol; acid hydrolysis of the latter resulted in 4-amino-1-phenylisoquinolin-3-ol, and the amino and hydroxyl groups in this compound allowed the formation of new ring systems. Accordingly, when 4-amino-1-phenylisoquinolin-3-ol hydrochloride (**8**) was heated in acetic acid with an excess of acetic anhydride, 2-methyl-5-phenyloxazolo[5,4-*c*]isoquinoline (**9**) was obtained [1] (Scheme 2).

Scheme 2

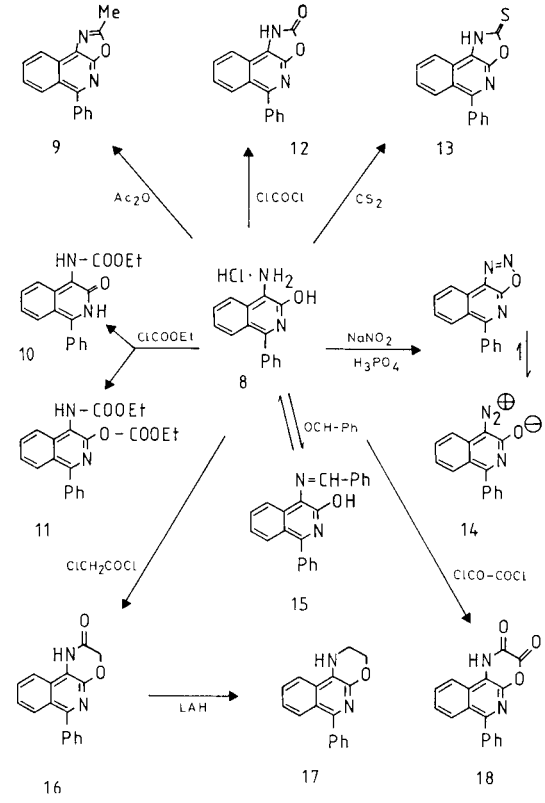


Table 1

Characteristic IR Bands (Potassium Bromide, $cm^{-1}$) and $^1H$ NMR Chemical Shifts [a] ($\delta$ TMS = 0 ppm) of Compounds **6**, **7**, **10-14** and **16-18** at 250 MHz

| Compound | NH Band | C=O/Amide-I Band | | Other IR Bands | $CH_3/NCH_2$ s/t (3/2H) [b] | $OCH_{3/2}$ s/t/qa (3/2H) [c] | | NH s (1H) [d] H-2',6' D (2H) | ArH (H-5-8 of and H-2'-6') [e] H-3'-5' m (3H) | H-6 t (1H) | H-7 t (1H) | H-8 d (1H) | H-5 d (1H) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **6** | – | – | – | – | 2.94 | – | – | 7.75 | 7.45 | | 7.82 | 8.15 | 8.72 |
| **7** | – | – | – | C–O 1252 | 2.95 | 3.88 | 4.16 – | 7.57 | 7.55 | – | – | 7.44 [f] | 8.02 [f] |
| **10** | 3500-2500 | 1701 [g] | 1637 | C–O 1246 | 1.29 | 4.22 | 7.15 | 7.5 [h] | 7.4 | 7.05 | 7.5 [h] | 7.67 | |
| **11** | 3220, 3115 | 1771 | 1709 [g] | C–O 1225 | 1.30 [g], 1.40 | 4.25 [g] | 4.37 6.45 | 7.7 [h] | 7.5 [i] | 7.7 [h] | 7.5 [i] | 8.13 | 8.05 |
| **12** | 3100 | 1767 | 1728 [g] | – | – | – | – | – | 7.65 | – | 7.88 | 8.10 | |
| **13** | – | – | – | – | – | – | – | | 7.65 | – | 7.92 | 8.10 | 8.24 |
| **14** | – | – | – | N=N 2102 | – | – | – | 7.8 | 7.6 [h] | 7.35 | | 7.6 [h] | 7.8 |
| **16** | 3250-3000 | – | 1701 | – | – | 4.89 | 11.3 | – | 7.45 | | 7.78 | 8.00 | 8.35 |
| **17** | 3217 | – | – | – | 3.59 | 4.47 | 4.2 | 7.7 [h] | | 7.45 | – | 7.7 [h] | 8.06 |
| **18** | 3250-3000 | 1709 [l] | 1695 [l] | – | – | – | – | – | 7.65 | – | 7.92 | 8.12 | 8.65 |

[a] In deuteriochloroform (**6**, **7**, **10**, **11**, **17**) or dimethyl-$d_6$ sulfoxide solution (**12-14**, **16**, **18**). [b] s (3H) for **6** and **7**, t (3H) for **10** and **11** and t (2H) **17**, respectively. [c] s (3H) for **7**, qa (2H) for **10** and **11**, s (2H) for **16** and t (2H) for **17**, respectively. [d] Broad, in cases of **12**, **13** and **18** not identifiable. [e] Signals of the condensed benzene ring (H-5,6,7,8) and the 1-phenyl group (H-2',3',4',5',6') of 9H-intensity (in case of **7**, 7H-intensity). [f] s (1H). [g] Urethane group. [h,i] Overlapping signals. [k] Split band pair. [l] The $\nu$ as C=O-$\nu$s C=O band pair of the coupled carbonyl stretching vibrations.

Table 2

$^{13}C$ NMR Chemical Shifts ($\delta$ TMS = 0 ppm) of Compounds **6**, **7**, **10-13** and **16-18** in Deuteriochloroform or Dimethyl-$d_6$ Sulfoxide Solution [a] at 20.14 MHz

| Compound | C-1 | C-3 | C-4 | C-4a | C-5 | C-6 | C-7 | C-8 | C-8a | C-1' | C-2',6' | C-3',5' | C-4' | $CH_3$ [b] | $OCH_2$ [c] | C=O |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **6** | 158.0 | 150.8 | 140.6 | 125.0 | 122.9 | 130.3 | 127.8 [e] | 126.3 [e] | 131.3 | 139.2 | 129.0 | 128.1 | 128.4 [e] | 20.5 | – | 165.8 |
| **7** | 156.0 | 149.9 [e] | 140.3 | 120.9 | 102.2 | 149.7 [e] | 153.3 | 107.1 | 128.3 [f] | 139.8 | 129.8 | 128.4 | 128.0 [f] | 20.7 | 55.9<br>56.3 | 165.6 |
| **10** | 157.1 [e] | 157.0 [e] | 140.0 [f] | 111.0 | 123.5 [g] | 128.7 | 125.1 [g] | 123.4 [g] | 130.2 [h] | 139.2 [f] | 131.4 | 129.8 | 131.6 [h] | 16.3 | 61.9 | 157.9 |
| **11** | 155.0 [e] | 148.5 | 138.4 [f] | 117.1 | 123.2 [i] | 128.1 | 126.9 | 123.3 [i] | 130.7 [g] | 137.1 [f] | 130.3 | 128.4 | 129.1 [g] | 14.2 | 62.0 | 152.7 |
| **12** | 154.8 [e] | 153.1 [e] | 124.9 [f] | 118.2 | 127.7 | 130.1 | 129.4 | 122.6 | 125.8 [f] | 140.1 | 131.7 | 129.9 | 132.1 | – | – | 148.0 |
| **13** | 155.8 [e] | 153.2 [e] | 126.2 [f] | 119.2 | 128.6 | 130.5 | 129.8 | 123.0 | 126.0 [f] | 139.7 | 131.7 | 130.0 | 133.0 | – | – | 179.7 |
| **16** | 153.6 | 146.9 | 129.2 | 114.7 | 127.1 [e] | 130.1 | 128.9 [e] | 122.0 | 125.0 | 140.2 | 131.5 [i] | 129.9 | 131.5 [i] | – | 68.9 | 166.7 |
| **17** | 148.4 | 145.4 | 128.8 [e] | 119.5 | 124.2 | 128.1 [e,i] | 127.8 | 119.0 | 123.8 | 139.5 | 130.2 | 128.1 [e,i] | 128.6 | 41.1 | 65.4 | – |
| **18** | 155.6 [e] | 143.1 | 129.1 | 115.2 | 128.6 | 130.5 | 128.8 | 122.7 | 126.0 | 139.5 | 131.5 | 130.1 | 132.4 | – | – | 154.0 [e] |

[a] In deuteriochloroform solution for compounds **6**, **7**, **11** and **17** and in dimethyl-$d_6$ sulfoxide solution for compound **10**, **12**, **13**, **16** and **18**, respectively. [b] $NCH_2$ in case of **17**. [c] $OCH_3$ in case of **7**. [d] N=C-S for **6** and **7**, C=S for **13**. [e,f,g,h] Reversed assignments may also be possible. [i] Two overlapping lines.

By reaction with ethyl chloroformate [4] we tried to synthesize fused-ring oxazolones; however, instead of the tricycles expected, different carbethoxy derivatives were obtained depending on the amount of reagent used. One equivalent of the reagent yielded 4-carbethoxyamino-1-phenyl-3(2*H*)-isoquinolinone (**10**), while the reaction of **8** with 3-4 equivalents of ethyl chloroformate gave the dicarbethoxy derivative **11**. The assumed structures of compounds **10** and **11** were confirmed by the $\nu$ NH and urethane carbonyl bands in the ir spectra, and in **11** also by the characteristic high-frequency carbonyl band of carbonic esters [5a]. Further evidence was the methyl triplets and methylene quartets, due to one and two ethoxy groups, respectively, in the $^1$H nmr spectra.

In the $^{13}$C nmr spectra, the C-3 signal of **10**, at 157.0 ppm, is shifted downfield by 8.5 ppm as compared with that in **11**, showing the predominating role of the amide tautomer in deuteriochloroform solution over the other tautomer.

Formation of an additional ring was achieved with phosgene in chloroform solution to give 5-phenyl-1,2-dihydro-oxazolo[5,4-*c*]isoquinolin-2-one **12**. The thione analogue **13** was prepared using carbon disulfide. The cyclic structures of the oxazolinone **12** and oxazolinethione **13** were verified by the presence of the $\nu$ NH band in the ir spectra, the carbonyl band of enol ester type having a high frequency in **12** [5b], split owing to Fermi resonance [5c], and by the downfield-shifted carbon resonance line (179.7 ppm) characteristic of thioxo groups [7a] in **13**.

Treatment of **8** with sodium nitrite in phosphoric acid gave a very unstable and light-sensitive compound. The structure did not correspond to a 1,2,3-oxadiazolo[5,4-*c*]-isoquinoline as expected, but rather to its open-ring zwitterionic form **14**. The presence of an intense band at 2102 cm$^{-1}$ in the ir spectrum, characteristic of diazonium compounds [5d] supported this conclusion.

The development of new ring systems *via* Schiff bases [8] was also attempted. In the reaction of **8** with benzaldehyde, a new product of vivid red colour was formed; its molecular mass supported the formation of the expected compound **15**, but this Schiff base decomposed on standing in solution, also under the conditions of the tlc test, yielding the starting amino derivative and benzaldehyde. This was shown by two-dimensional tlc and $^1$H nmr examination. Reduction of the C=N double bond with sodium borohydride failed.

Reaction of the *o*-aminoalcohol **8** with chloroacetyl chloride in isobutyl methyl ketone [9] gave 6-phenyl-2,3-dihydro-1*H*-[1,4]oxazino[2,3-*c*]isoquinolin-2-one (**16**).

Reduction of the lactam carbonyl was achieved with lithium aluminum hydride to yield **17**. The structure of **16** is evidenced by the presence of the $\nu$ NH and amide-I ir bands, the isolated methylene singlet adjacent to an oxygen atom and by the signal at 68.9 ppm. The ir spectrum of **17** has the $\nu$ NH band, and the amide-I band is absent; in its $^1$H nmr spectrum the two methylene triplets due to the -NH-$CH_2$-$CH_2$-O- can be recognized, while the $^{13}$C nmr spectrum has the corresponding $NCH_2$ and $OCH_2$ lines at the expected shift values, 41.1 and 65.4 ppm, respectively.

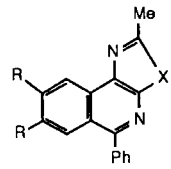
The methylene group in the ring of **16** does not behave like an active methylene group [10], thus it did not undergo nitrosation with butyl nitrite.

The same ring system of higher oxidation level can be formed by the use of oxalyl chloride; with this reagent 6-phenyl-2,3-dihydro-1*H*-[1,4]oxazino[2,3-*c*]isoquinoline-2,3-dione (**18**) was obtained. This is evidenced by the coupling of the neighbouring carbonyl ir valence vibrations; in the $^1$H nmr spectra only the signals of the nine aromatic hydrogens can be identified, and the $^{13}$C nmr spectrum is very similar to that of compound **16**, except that the methylene signal is replaced by a carbonyl line; further, the mutual electron-withdrawing effect of the neighbouring carbonyl groups prevents the positive polarization of the carbonyl carbon atoms; the higher electron density results in an upfield shift of the carbonyl carbon signal (**7b**). This is responsible for the upfield shift of the amide carbonyl line by about 14 ppm in **18** as compared to the usual value for amides and also observed in **16**.

It should be noted that the given assignments of the

Table 3

Electron-Collision Mass Spectra of Compounds **3**, **6**, **7** and **9** Containing Oxazole or Thiazole Rings




| Type of ion | R = H, X = O **9** M = 260 | R = H, X = S **6** M = 276 | R = $OCH_3$, X = O **3** M = 320 | R = $OCH_3$, X = S **7** M = 336 |
|---|---|---|---|---|
| M$^+$ | 100 | 94 | 100 | 100 |
| [M-H]$^+$ | 75 | 100 | 13 | 25 |
| [M-$CH_3$]$^+$ | – | – | 11 | 21 |
| [M-CHX]$^+$ | 3 | – | – | – |
| [M-$CH_2O$]$^+$ | – | – | 7 | 16 |
| [M-$CH_3O$]$^+$ | – | – | 4 | 8 |
| [M-H-$CH_3CN$]$^+$ | 1 | 8 | – | – |
| [M-$CH_3CH_2O$]$^+$ | – | – | 2 | 3 |
| [M-$CH_3CH_2CN$]$^+$ | – | – | 2 | 3 |
| [M-$CH_3CN$-CX]$^+$ | 14 | 5 | – | – |
| [M-H-$CH_3CN$-CX]$^+$ | 29 | 13 | – | – |
| [M—$CH_3CH_2CN$-CO]$^+$ | – | – | 12 | 4 |
| [M-$CH_3CN$-$CH_2O$-CO]$^+$ | – | – | 3 | 2 |

Table 4

Analytical Data for Compounds **6**, **7**, **10-18**

| Compound | Formula | Mp, °C (solvent) | Analysis, % Calcd./Found | | | | Yield, % | MS M⁺ (m/e) |
|---|---|---|---|---|---|---|---|---|
| | | | C | H | N | S | | |
| **6** | $C_{17}H_{12}N_2S$ | 140-142 (EtOH) | 73.9<br>74.1 | 4.4<br>4.3 | 10.1<br>10.2 | 11.6<br>11.6 | 36 | 276 (94) |
| **7** | $C_{19}H_{16}N_2O_2S$ | 198-200 (MeOH) | 67.6<br>67.8 | 5.1<br>4.9 | 8.3<br>7.9 | 9.5<br>9.1 | 32 | 336 (100) |
| **10** | $C_{18}H_{16}N_2O_3$ | 186-187 (EtOAc) | 70.1<br>70.2 | 5.2<br>5.4 | 9.1<br>9.0 | | 51 | 308 (20) |
| **11** | $C_{21}H_{20}N_2O_5$ | 158-160 (EtOH) | 66.3<br>66.7 | 5.3<br>5.6 | 7.4<br>7.2 | | 75 | 380 (19) |
| **12** | $C_{16}H_{10}N_2O_2$ | 292-293 ($Me_2CO$) | 73.3<br>73.4 | 3.8<br>4.0 | 10.7<br>10.6 | | 56 | |
| **13** | $C_{16}H_{10}N_2OS$ | 253-254 (EtOAc) | 69.0<br>69.2 | 3.6<br>3.7 | 10.1<br>10.0 | 11.5<br>11.6 | 81 | |
| **14** | $C_{15}H_9N_3O$ | 123-135 ($Et_2O$) | 72.9<br>72.9 | 3.7<br>3.7 | 17.0<br>16.9 | | 70 | |
| **15** | $C_{22}H_{16}N_2O$ | >220 | 81.5<br>81.3 | 5.0<br>5.0 | 8.6<br>8.7 | | 50 | 324 (100) |
| **16** | $C_{17}H_{12}N_2O_2$ | 276-278 (EtOH) | 73.9<br>74.1 | 4.5<br>4.6 | 10.1<br>10.3 | | 40 | |
| **17** | $C_{17}H_{14}N_2O$ | 156-158 (EtOH) | 77.8<br>77.6 | 5.4<br>5.3 | 10.7<br>10.7 | | 42 | |
| **18** | $C_{17}H_{10}N_2O_3$ | 275-276 ($Me_2CO$) | 70.3<br>70.3 | 3.5<br>3.4 | 9.6<br>9.6 | | 83 | |

aromatic proton signals in the $^1H$ nmr spectra and those of the near carbon lines in the $^{13}C$ nmr spectra (*cf.* Tables 1 and 2) are partly of tentative character. Based on trends of the shifts in related compounds and on the intensitites of the carbon lines, the most probable assignments are given, but in a few cases interchanged assignments are also to be considered. We should like to emphasize, however, that such occasional changes do not influence our conclusions concerning the postulated structures.

The electron collision mass spectra (Table 3) of compounds **3**, **6**, **7** and **9** containing oxazole and thiazole rings reveal interesting features. The common characteristic of these compounds is the molecular peak of high intensity, indicating very high stability of these molecular ions with a fused unsaturated skeleton.

In the case of the oxazole **9**, the main fragment is the $[M-H]^+$ ion; its high intensity can be explained by the ready occurrence of the condensation reaction between the phenyl group and the heterocycle. This process is even more favoured by the O → S exchange (charge localization).

In compounds **9** and **6** the splitting of the skeleton starts by the cleavage of the five-membered ring only (elimination of $CH_3CN$ and CX).

The presence of the methoxy groups in **7** causes an unusually marked substituent effect: both the condensation reaction (formation of $[M-H]^+$) and the cleavage of the five-membered ring are suppressed as compared with the fragmentation of the methoxy groups, in accordance with the very high stability of the skeleton of these compounds.

## EXPERIMENTAL

For analytical and spectroscopic data, see Tables 1-4.

The ir spectra were recorded in potassium bromide pellets on an Aspect 2000 computer-controlled Bruker IFS-113v FT spectrometer.

The $^1H$ and $^{13}C$ nmr spectra were determined in deuteriochloroform or dimethyl-$d_6$ sulfoxide solution in 5 or 10 mm cells at room temperature on a Bruker WM-250 ($^1H$) and a WP-80-SY FT spectrometer ($^{13}C$) at 250.13 and 20.14 MHz, respectively, using the $^2H$ signals of the solvent as the lock and TMS as the internal standard. The most important recording parameters of the $^1H$ and $^{13}C$ nmr spectra were as follows: sweep width 5 kHz, pulse width 1 and 3.5 μs (about 20° and 30° flip angle, respectively): acquisition time, 1.64 s, number of scans, 8 and 1-5 K; computer memory 16 K. Complete proton noise decoupling (about 1.5 W) for the $^{13}C$ spectra, and Lorentzian exponential multiplication for signal-to-noise enhancement were used (line width 0.7 and 1.0 Hz). Mass spectra were run on a Varian MAT spectrometer at 70 eV.

5-Phenyl-2-methylthiazolo[5,4-*c*]isoquinoline (**6**).

4-Acetamino-1-phenyl-1,4-dihydro-3(2*H*)-isoquinolinone (**4**) (8.5 g, 30 mmoles) [3] was dissolved in pyridine (180 ml) and phosphorus pentasulfide (11.5 g) was added slowly, with vigorous stirring in a stream of nitrogen. The temperature of the solution increased spontaneously to 50°. It was heated to boiling and stirred for 1 hour. After pouring onto ice, the product was extracted with chloroform. The organic phase was washed with 2 *M* hydrochloric acid. Evaporation of the solvent left an oil

which crystallized on rubbing with acetone. The product (3.0 g) was recrystallized form ethanol (100 ml).

7,8-Dimethoxy-5-phenyl-2-methylthiazolo[5,4-*c*]isoquinoline (**7**).

The above procedure was carried out starting from **5**. The oil obtained after the extraction step was rubbed with ethyl acetate to give crystalline **7**.

4-Carbethoxyamino-1-phenyl-3(2*H*)-isoquinolinone (**10**).

Compound **8** (6.0 g, 22 mmoles) was dissolved in pyridine (200 ml), cooled to 0° and ethyl chloroformate (2.1 ml, 22 mmoles) was added to the stirred solution in several portions. After pouring onto ice and acidifying with hydrochloric acid, the product was extracted with chloroform. The residue obtained on evaporation of the solvent was treated with ether to yield the product (3.4 g) which was purified by recrystallization from ethyl acetate.

4-Ethoxycarbonylamino-3-ethoxycarbonyloxy-1-phenyl-isoquinoline (**11**).

Compound **8** (5.0 g, 18 mmoles) was dissolved in pyridine (100 ml) and ethyl chloroformate (6.5 ml, 68 mmoles) was added to the solution in several portions, with stirring at 10°. Workup as described above gave 5.25 g of the product.

5-Phenyl-1,2-dihydrooxazolo[5,4-*c*]isoquinoline-2-one (**12**).

Compound **8** (11.1 g, 40 mmoles) was dissolved in pyridine (200 ml) and a chloroform solution containing phosgene (0.118 mole) was added with cooling in ice-water. The reaction mixture was allowed to warm to 20°, and the excess phosgene was removed with a stream of nitrogen gas. After pouring onto ice, the mixture was acidified with dilute hydrochloric acid and extracted with chloroform. The solvent was evaporated and the residue was rubbed with ether to yield the product (6.0 g). It was purified by recrystallization from acetone.

5-Phenyl-1,2-dihydrooxazolo[5,4-*c*]isoquinoline-2-thione (**13**).

Compound **8** (10 g, 36 mmoles) was dissolved in a mixture of pyridine (600 ml) and carbon disulfie (500 ml), and the mixture was refluxed for 40 minutes. The two phases formed in the reaction were then separated, the pyridine fraction was acidified, and the product which separated (8.3 g) was filtered off. It was purified by recrystallization from methanol (1500 ml).

4-Diazonium-1-phenylisoquinoline-3-olate (**14**).

Compound **8** (3.0 g, 11 mmoles) was dissolved in 85% phosphoric acid (100 ml) and sodium nitrite (1.2 g, 17 mmoles) was added to the solution in small portions. After 10 minutes, the reaction mixture was poured into distilled water (400 ml) and the product was extracted with chloroform. The solvent was evaporated and the residue rubbed with ether. The product (1.9 g) was recrystallized from ethyl acetate (30 ml).

4-Benzylideneimino-1-phenylisoquinolin-3-ol (**15**).

Compound **8** (1.0 g, 3.6 mmoles) and benzaldehyde (1.0 ml, 10 mmoles) were refluxed in benzene (60 ml) using a water separator. After 3 hours, the red crystals which separated were filtered off (0.6 g) and analyzed. On recrystallization from acetone the Schiff base partly decomposed into the starting compounds.

6-Phenyl-2,3-dihydro-1*H*-[1,4]oxazino[2,3-*c*]isoquinolin-2-one (**16**).

Compound **8** (2.0 g, 7.3 mmoles) was dissolved in a mixture of isobutyl methyl ketone (40 ml) and water (20 ml), then chloroacetyl chloride (1.0 ml, 13 mmoles) was added in small portions, with cooling. Sodium hydrogen carbonate (3.5 g) was then added and the mixture was refluxed for 4 hours. After dilution with water, the product was extracted with ethyl acetate. Evaporation of the solvent left the product (0.8 g) which was purified by recrystallization from ethanol.

6-Phenyl-2,3-dihydro-1*H*-[1,4]oxazino[2,3-*c*]isoquinoline (**17**).

Compound **16** (5.0 g, 18 mmoles) was dissolved in anhydrous tetrahydrofuran (900 ml) and reduced with lithium aluminum hydride (13.0 g) at 45° in nitrogen atmosphere, with vigorous stirring. When reaction of the starting compound was complete, the complex was decomposed by the addition of ethyl acetate and water. The suspension was filtered and the filtrate evaporated to dryness under vaccum. The residue was treated with ether to obtain the product which was purified by recrystallization from ethanol.

6-Phenyl-2,3-dihydro-1*H*-[1,4]oxazino[2,3-*c*]isoquinoline-2,3-dione (**18**).

Compound **8** (6.0 g, 22 mmoles) was allowed to react with oxalyl chloride (75 ml) and refluxed for 30 minutes. The reaction mixture was evaporated to dryness and the product was washed with ether. It was purified by recrystallization from acetone.

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