# Heterocycles. IX. Synthesis of New Substituted Benzo[*h*]quinolones



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2-Arylidine-1-tetralones I were condensed with phenyl-, *p*-chlorophenyl-, and *p*-methoxyphenylacetamides **A** in the presence of a base to yield the corresponding benzo[*h*]quinolones II-IV. Dehydrogenation of compounds II gave the benzoquinolones V which upon acetylation yielded the corresponding 2-acetyl derivatives VI. The structure of all products was substantiated by chemical and spectral methods.

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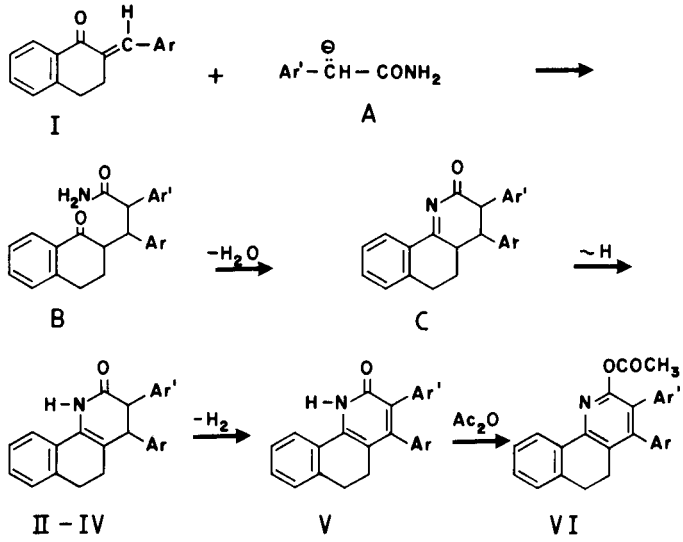
In a previous investigation the reaction of 1,3-diaryl-2-propen-1-ones with arylacetamides was reported to give the corresponding 2-pyridones [1]. The present work was intended to introduce a new route for the synthesis of benzo-2(1*H*)-quinolones [2,3] and to establish the structure of the products by chemical and spectral methods. Thus different exocyclic $\alpha,\beta$-unsaturated cyclic ketones Ia-k were prepared as previously reported [4]. These were then condensed with phenyl-, *p*-chlorophenyl-, and *p*-methoxyphenylacetamides in the presence of sodium ethoxide to give the corresponding 3,4-diaryl-3,4,5,6-tetrahydrobenzo-[*h*]quinolin-2(1*H*)-ones II, III and IV respectively (*cf.* Scheme 1).

The structure of these benzoquinolone derivatives II-IV was established by spectroscopic and chemical evidence (*cf.* Table 1 and 2). Thus, the infrared spectra of compounds II-IV show absorptions in the 3410-3060 $cm^{-1}$ (broad), 1690-1660 $cm^{-1}$ and 1660-1600 $cm^{-1}$ regions which are correlated to the $-NH$, $C=O$ and $C=C$ stretching frequencies of these compounds [1,5]. The uv spectra of the above compounds lend further support to the proposed structure. They show two absorption bands which can be assigned to $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transitions of their chromophores [6]. The nmr spectra agree well with the proposed structure and show two multiplets in the regions $\delta$ 2.07-3.20 ppm and $\delta$ 6.20-8.94 which correspond to the




| Compound | Ar | Compound | Ar' |
|---|---|---|---|
| I-VI a | $C_6H_5$ | I-VI h | $C_4H_3S$ (2-thienyl) |
| b | p-$CH_3$-$C_6H_4$ | i | $C_4H_3O$ (2-furyl) |
| c | p-$OCH_3$-$C_6H_4$ | j | 3-$C_5H_4N$ (3-pyridyl) |
| d | p-Cl-$C_6H_4$ | k | 4-$C_5H_3N$ (4-pyridyl) |
| e | m-Cl-$C_6H_4$ | Compound | Ar' |
| f | 1-$C_{10}H_7$ | II, V, VI | $C_6H_5$ |
| g | 2-$C_{10}H_7$ | III | p-Cl-$C_6H_4$ |
| | | IV | p-$OCH_3$-$C_6H_4$ |

Scheme 1

Table 1

The Infrared, Electronic and Nuclear Magnetic Resonance Spectral Data of Compounds II-VI

| Compound | Infrared Spectra (potassium bromide) | | Electronic Spectra (Ethanol) | | NMR (Deuteriochloroform) | |
|---|---|---|---|---|---|---|
| | $Cm^{-1}$ | $\nu$ | $\lambda$ max (nm) | $\epsilon$ | $\delta$ | Assignment (no. of protons) |
| IIa | 1645 (s) | C=C | 225 | 12300 | 2.07-2.77 (m) | (4) $CH_2$-$CH_2$ |
| | 1665 (s) | C=O | 295 | 3075 | 3.7-3.93 (d, d) | (2) CH-CH |
| | 3220 (br) | NH | | | 7.13-7.40 (m) | (14) Ar-H |
| | | | | | 7.93 (br) | (1) NH |
| b | 1643 (m) | C=C | 227 | 14110 | 2.09-2.69 (m) | (4) $CH_2$-$CH_2$ |
| | 1665 (s) | C=O | 265 | 3290 | 3.68-3.88 (d, d) | (2) CH-CH |
| | 3220 (br) | NH | 314 | 3840 | 3.0 (s) | (3)$CH_3$ |
| | | | | | 7.27-7.80 (m) | (13) Ar-H |
| | | | | | 8.07 (br) | (1) NH |
| c | 1660 (s) | C=C | 228 | 27180 | 2.07-2.80 (m) | (4) $CH_2$-$CH_2$ |
| | 1675 (s) | C=O | 251 (sh) | 8345 | 3.70-3.87 (d, d) | (2) CH-CH |
| | 3222 (br) | NH | 292 | 6200 | 3.73 (s) | (3) $OCH_3$ |
| | | | | | 6.70-7.30 (m) | (13) Ar-H |
| | | | | | 8.03 (br) | (1) NH |
| d | 1658 (s) | C=C | 227 | 26050 | 2.20-2.80 (m) | (4) $CH_2$-$CH_2$ |
| | 1672 (s) | C=O | 298 | 4825 | 3.73-3.88 (d, d) | (2) CH-CH |
| | 3217 (br) | NH | | | 6.70-7.80 (m) | (13) Ar-H |
| | | | | | 8.13 (br) | (1) NH |
| e | 1652 (s) | C=C | 226 | 15680 | 2.17-2.73 (m) | (4) $CH_2$-$CH_2$ |
| | 1670 (s) | C=O | 297 | 3860 | 3.68-3.83 (d, d) | (2) CH-CH |
| | 3218 (br) | NH | | | 6.73-7.68 (m) | (13) Ar-H |
| | | | | | 8.07 (br) | (1) NH |
| f | 1640 (w) | C=C | 223 | 25270 | 2.93 (s) | (4) $CH_2$-$CH_2$ |
| | 1660 (s) | C=O | 268 | 21080 | 3.50 (s) | (2) CH-CH |
| | 3180 (br) | NH | 310 (sh) | 8030 | 7.20-8.23 (m) | (16) Ar-H |
| | 3360 (br) | | | | 8.33 (s) | (1) NH |
| g | 1630 (w) | C=C | 227 | 18570 | 2.77-3.20 (m) | (4) $CH_2$-$CH_2$ |
| | 1665 (s) | C=O | 272 | 6525 | 3.70-3.97 (d, d) | (2) CH-CH |
| | 3240 (br) | NH | 325 | 5020 | 7.27-8.03 (m) | (16) Ar-H |
| | | | | | 8.25 (br) | (1) NH |
| h | 1646 (m) | C=C | 230 | 24575 | 2.33-2.80 (m) | (4) $CH_2$-$CH_2$ |
| | 1666 (s) | C=O | 296 | 4915 | 4.0 (s) | (2) CH-CH |
| | 3220 (br) | NH | | | 6.93-7.30 (m) | (12) Ar-H |
| | | | | | 7.87 (br) | (1) NH |
| i | 1650 (s) | C=C | 224 | 17090 | 2.23-3.05 (m) | (4) $CH_2$-$CH_2$ |
| | 1670 (s) | C=O | 292 | 4120 | 3.90-4.13 (d, d) | (2) CH-CH |
| | 3225 (br) | NH | 370 | 1030 | 6.20-7.70 (m) | (12) Ar-H |
| | | | | | 8.15 (br) | (1) NH |
| j | 1660 (w) | C=C | 226 | 5020 | 2.20-2.80 (m) | (4) $CH_2$-$CH_2$ |
| | 1678 (s) | C=O | 251 (sh) | 2815 | 3.60-3.93 (d, d) | (2) CH-CH |
| | 3210 (br) | NH | 290 | 1585 | 7.46-8.60 (m) | (14) Ar-H, NH |
| | | | 298 | 1585 | | |
| k | 1640 (w) | C=C | 237 (sh) | 6600 | 2.27-2.80 (m) | (4) $CH_2$-$CH_2$ |
| | 1660 (s) | C=O | 300 | 1760 | 3.47-3.67 (d, d) | (2) CH-CH |
| | 3170 (br) | | | | 6.85-7.70 (m) | (13) Ar-H |
| | 3360 (br) | NH | | | 7.86 (br) | (1) NH |
| IIId | 1620 (s) | C=C | 227 | 11940 | 2.33-2.77 (m) | (4) $CH_2$-$CH_2$ |
| | 1670 (s) | C=O | 295 | 1706 | 3.50-3.77 (d, d) | (2) CH-CH |
| | 3100 (br) | NH | | | 7.17-7.76 (m) | (12) Ar-H |
| | 3210 (br) | | | | 8.17 (br) | (1) NH |
| e | 1592 (m) | C=C | 222 | 7875 | 2.13-2.77 (m) | (4) $CH_2$-$CH_2$ |
| | 1630 (m) | | | | | |
| | 1668 (s) | C=O | 297 | 4515 | 3.70-3.90 (d, d,) | (2) CH-CH |
| | 3230 (br) | NH | | | 7.20-7.73 (m) | (12) Ar-H |
| | | | | | 8.10 (br) | (1) NH |

Table 1 (continued)

The Infrared, Electronic and Nuclear Magnetic Resonance Spectral Data of Compounds II-VI

| Compound | Infrared Spectra (potassium bromide) | | Electronic Spectra (Ethanol) | | NMR (Deuteriochloroform) | |
|---|---|---|---|---|---|---|
| | $Cm^{-1}$ | $\nu$ | $\lambda$ max (nm) | $\epsilon$ | $\delta$ | Assignment (no. of protons) |
| f | 1600 (m) | C=C | 223 | 47960 | 2.34-3.0 (m) | (4) $CH_2$-$CH_2$ |
| | 1660 (s) | C=O | 268 | 12535 | 3.66-4.0 (d, d) | (2) CH-CH |
| | 3060 (br) | NH | | | 7.40-8.20 (m) | (16) Ar-H, NH |
| h | 1600 (w) | C=C | 225 | 7350 | 2.80-3.26 (m) | (4) $CH_2$-$CH_2$ |
| | | | 295 | 1765 | 3.50-3.97 (d, d) | (2) CH-CH |
| | 1670 (s) | C=O | | | 7.08-8.17 (m) | (12) Ar-H, NH |
| | 3210 | | | | | |
| | 3410 (br) | NH | | | | |
| i | 1620 (s) | C=C | 224 | 10465 | 2.27-2.60 (m) | (4) $CH_2$-$CH_2$ |
| | 1670 (s) | C=O | 295 | 1590 | 3.70-4.0 (d, d) | (2) CH-CH |
| | 3230 (br) | NH | | | 6.10-7.33 (m) | (11) Ar-H |
| | | | | | 7.60 (s) | (1) NH |
| j | 1600 (w) | C=C | 224 | 14510 | 2.20-2.80 (m) | (4) $CH_2$-$CH_2$ |
| | | | 252 (sh) | 5320 | 3.60-3.93 (d, d) | (2) CH-CH |
| | 1675 (s) | C=O | 295 | 2900 | 7.40-8.80 (m) | (12) Ar-H |
| | 3210 (br) | NH | | | 9.93 (s) | (1) NH |
| k | 1600 (s) | C=C | 225 | 21770 | 2.20-2.80 (m) | (4) $CH_2$-$CH_2$ |
| | | | 295 | 4355 | 3.74-4.0 (d, d) | (2) CH-CH |
| | 1690 (s) | C=O | | | 7.54-8.94 (m) | (12) ArH |
| | 3210 (br) | NH | | | 9.54 (s) | (1) NH |
| IVa | 1630 (s) | C=C | 224 | 27695 | | |
| | 1665 (s) | C=O | 295 | 4870 | | |
| | 3220 (m) | NH | | | | |
| j | 1650 (w) | C=C | 223 | 10505 | 2.66-3.60 (m) | (6) $CH_2$-$CH_2$, |
| | 1670 (s) | C=O | 290 | 15280 | | CH-CH |
| | 3230 (br) | NH | | | 3.90 (s) | (3) O $CH_3$ |
| | | | | | 7.0-8.80 (m) | (12) Ar-H |
| | | | | | 9.40 (s) | (1) NH |
| Vc | 1625 (br) | C=O | 236 | 10420 | 3.07 (s) | (4) $CH_2$-$CH_2$ |
| | 3080 (br) | NH | 260 | 4980 | 4.14 (s) | (3) $OCH_3$ |
| | | | 364 | 3320 | 7.40-8.30 (m) | (13) Ar-H |
| | | | | | 11.86 (s) | (1) NH |
| e | 1615 (s) | | 235 | 7950 | 3.06 (s) | (4) $CH_2$-$CH_2$ |
| | 1640 (s) | C=O | 260 | 4790 | 7.54-8.60 (m) | (13) Ar-H |
| | 2900 (br) | | 366 | 6510 | 12.06 (s) | (1) NH |
| | 3210 (w) | NH | | | | |
| h | 1620 (br) | C=O | 235 | 4440 | 3.14 (s) | (4) $CH_2$-$CH_2$ |
| | 2920 (br) | | 261 | 2660 | 7.42-8.45 (m) | (12) Ar-H |
| | 3110 (w) | NH | 370 | 3460 | 11.95 (s) | (1) NH |
| i | 1620 (br) | C=O | 235 | 7720 | 3.20 (s) | (4) $CH_2$-$CH_2$ |
| | | | | | 6.40-8.50 (m) | (12) Ar-H |
| | 3060 (br) | NH | 259 | 4540 | 11.86 (s) | (1) NH |
| | | | 368 | 4995 | | |
| VIh | 1765 (s) | C=O | 265 | 14510 | 2.20 (s) | (3) CO $CH_3$ |
| | | | 292 | 8355 | 2.97 (s) | (4) $CH_2$-$CH_2$ |
| | | | 319 | 14100 | 6.87-7.0 (m) | (12) Ar-H |
| | | | 370 | 1935 | | |
| i | 1770 (s) | C=O | 267 | 17950 | 2.0 (s) | (3) $COCH_3$ |
| | | | 293 | 9790 | 2.90 (s) | (4) $CH_2$-$CH_2$ |
| | | | 321 | 15500 | 5.92-8.25 (m) | (12) Ar-H |
| | | | 372 | 1960 | | |

Table 2

Yields, Melting Points and Element Analysis of Compounds II-VI

| Compd. No. | Yield (%) | Mp °C | Formula | Calcd. % C | H | N | Cl | S | Found % C | H | N | Cl | S |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| IIa | 75 | 204-205 | $C_{25}H_{21}NO$ | 85.44 | 6.02 | 3.99 | | | 85.32 | 6.0 | 3.81 | | |
| b | 76 | 190-191 | $C_{26}H_{23}NO$ | 85.45 | 6.34 | 3.83 | | | 85.24 | 6.19 | 3.68 | | |
| c | 78 | 201-202 | $C_{26}H_{23}NO_2$ | 81.86 | 6.08 | 3.67 | | | 81.75 | 6.01 | 3.62 | | |
| d | 74 | 226-228 | $C_{25}H_{20}ClNO$ | 77.81 | 5.22 | 3.63 | 9.18 | | 77.76 | 5.11 | 3.59 | 9.0 | |
| e | 80 | 205-206 | $C_{25}H_{20}ClNO$ | 77.81 | 5.22 | 3.63 | 9.18 | | 77.68 | 5.10 | 3.57 | 8.96 | |
| f | 82 | 125 | $C_{29}H_{23}NO$ | 86.75 | 5.77 | 3.49 | | | 86.55 | 5.61 | 3.38 | | |
| g | 81 | 120 | $C_{29}H_{23}NO$ | 86.75 | 5.77 | 3.49 | | | 86.68 | 5.71 | 3.38 | | |
| h | 79 | 189 | $C_{23}H_{19}NOS$ | 77.28 | 5.35 | 3.92 | | 8.96 | 77.16 | 5.21 | 3.73 | | 8.89 |
| i | 83 | 200 | $C_{23}H_{19}NO_2$ | 80.92 | 5.61 | 4.10 | | | 80.87 | 5.52 | 4.08 | | |
| j | 84 | 185 | $C_{24}H_{20}N_2O$ | 81.79 | 5.72 | 7.95 | | | 81.66 | 5.54 | 7.87 | | |
| k | 78 | 165 | $C_{24}H_{20}N_2O$ | 81.79 | 5.72 | 7.95 | | | 81.68 | 5.80 | 7.92 | | |
| IIId | 77 | 222-223 | $C_{25}H_{19}Cl_2NO$ | 71.44 | 4.56 | 3.33 | 16.87 | | 71.35 | 4.50 | 3.23 | 16.79 | |
| e | 75 | 203-204 | $C_{25}H_{19}Cl_2NO$ | 71.43 | 4.55 | 3.33 | 16.86 | | 71.31 | 4.49 | 3.14 | 16.67 | |
| f | 79 | 114 | $C_{29}H_{22}ClNO$ | 79.90 | 5.08 | 3.21 | 8.13 | | 79.84 | 5.01 | 3.18 | 8.02 | |
| h | 80 | 215-216 | $C_{23}H_{18}ClNOS$ | 70.48 | 4.63 | 3.57 | 9.04 | 8.18 | 70.37 | 4.52 | 3.46 | 9.10 | 8.08 |
| i | 74 | 224-225 | $C_{23}H_{18}Cl_2NO_2$ | 73.50 | 4.83 | 3.73 | 9.43 | | 73.42 | 4.73 | 3.68 | 9.31 | |
| j | 78 | 210-211 | $C_{24}H_{19}ClN_2O$ | 74.51 | 4.95 | 7.24 | 9.16 | | 74.37 | 4.84 | 7.15 | 9.02 | |
| k | 76 | 223-224 | $C_{24}H_{19}ClN_2O$ | 74.51 | 4.95 | 7.24 | 9.16 | | 74.42 | 4.61 | 7.15 | 8.97 | |
| IVa | 75 | 256-257 | $C_{26}H_{23}NO_2$ | 81.86 | 6.10 | 3.67 | | | 81.71 | 6.02 | 3.53 | | |
| j | 76 | 206-207 | $C_{25}H_{22}N_2O_2$ | 78.51 | 5.79 | 7.32 | | | 78.36 | 5.62 | 7.28 | | |
| Vc | 85 | 345-347 | $C_{26}H_{21}NO_2$ | 82.30 | 5.58 | 3.69 | | | 82.15 | 5.29 | 3.57 | | |
| e | 87 | 353-354 | $C_{25}H_{18}ClNO$ | 78.22 | 4.72 | 3.65 | 9.23 | | 78.12 | 4.58 | 3.48 | 9.15 | |
| h | 89 | 349-351 | $C_{23}H_{17}NOS$ | 77.72 | 4.82 | 3.94 | | | 77.51 | 4.63 | 3.81 | | |
| i | 86 | 316-317 | $C_{23}H_{17}NO_2$ | 81.39 | 5.05 | 4.13 | | | 81.21 | 4.91 | 4.21 | | |
| VIh | 88 | 275-276 | $C_{25}H_{19}NO_2S$ | 75.54 | 4.82 | 3.52 | | 8.07 | 75.48 | 4.84 | 3.47 | | 8.12 |
| i | 90 | 236-237 | $C_{25}H_{19}NO_3$ | 78.72 | 5.02 | 3.67 | | | 78.61 | 4.90 | 3.52 | | |

protons at carbons 5,6 as well as the aromatic protons. The spectra show also double doublets in the region $\delta$ 3.47-4.13 which stand for the protons at carbons 3 and 4. The broad signals in the region $\delta$ 7.60-9.54 are attributed to the NH protons and disappear after adding deuterium oxide [1,7]. The mass spectra of compounds IIc,hi,j support their structures and show molecular ion peaks at m/e 380 (42.8%), m/e 357 (48.0%) and m/e 352 (53.03%) respectively. They show also base peaks which correspond to the ions $[M\text{-}C_8H_7NO]^+$.

The chemical behaviour of compounds II is also in favour of the proposed structure. Thus upon treatment of IIc,e,h,i with *o*-chloranil [8], they afforded the corresponding 3,4-diaryl-5,6-dihydrobenzo[*h*]quinolin-2(1*H*)-ones Vc,e,h,i respectively (*cf.* Scheme 1).

The structure of compounds V was substantiated from their chemical and spectral behaviour (*cf.* Table 1 and 2). Their infrared spectra revealed strong bands in the region 1625-1615 $cm^{-1}$ which can be ascribed to the dihydroquinolone system. The broad bands in the region 3210-2900 $cm^{-1}$ are attributed to the bonded NH [7]. The uv spectra show great resemblance to each other which reflects their structural identity. They reveal also absorptions at longer wavelengths than their precursors, indicating extended conjugation [9]. The nmr spectra were void of the double doublets characteristic for compounds II-IV, indicating the absence of the protons at $C_3$-$C_4$. They show chemical shifts which can be ascribed to NH, aromatic and $C_5$, $C_6$ protons [10]. The mass spectra of the benzo[*h*]-2(1*H*)-quinolones Vc,i revealed molecular ion peaks at m/e 379 (99.01%) and m/e 339 (100%) respectively. The base peak of Vc was represented by the ion $[M\text{-}1]^+$.

The structure of compounds Vh,i was also supported by their reaction with acetic anhydride to yield the corresponding 2-acetylquinolines VIh,i (*cf.* Scheme 1). The occurrence of the *O*-acetylation is infered from their spectra (*cf.* Table 1). The infrared spectra of VIh and i show only one carbonyl absorption at 1765 and 1770 $cm^{-1}$ ($O\text{-}COCH_3$) respectively, and were void of any NH or OH absorptions. The nmr spectra showed chemical shifts which can be ascribed to acetyl, $C_5$, $C_6$ and aromatic protons. The uv and mass spectra of these 2-acetyl derivatives are also in favour of the proposed structure.

It can be assumed that the formation of the substituted 2-quinolones II-IV results from the Michael addition of the arylacetamide carbanions **A** to the 2-arylidene-1-tetralones (*cf.* Scheme 1).

## EXPERIMENTAL

Melting points are uncorrected. Infrared and ultraviolet spectra were measured on Perkin Elmer 520 B and Pye Unicam Sp 8000 spectrophotometers, respectively. The nmr spectra were run on Varian T 60 A, using TMS as the internal standard. The ms were carried out using Varian MAT 311 A. The purity of the analytical samples was checked by the tlc (silica gel). Microanalyses were determined by H. Mallissa, microanalytical laboratory, West Germany.

Reaction of 2-Arylidene-1-tetralones I with Arylacetamides.

The 2-arylidene-1-tetralone I (0.01 mole) was added to a solution of sodium ethoxide (0.01 mole) and arylacetamide (0.01 mole) in absolute ethanol (50 ml). The reaction mixture was heated on a boiling water-bath for three hours with stirring. The solvent was evaporated and the residual product was dissolved in water, then acidified with dilute hydrochloric acid (10%). The solid separated by filtration was crystallized from benzene to give the corresponding 3,4-diaryl-3,4,5,6-tetrahydrobenzo[*h*]-quinolin-2(1*H*)-ones II-IV. (*cf.* Table 2).

Dehydrogenation of the 3,4-Diaryl-3,4,5,6-tetrahydrobenzo[*h*]quinolin-2(1*H*)-one Derivatives II.

*O*-Chloranil (0.2 g) was added portionwise to a solution of 1 g of the quinolone derivative II in tetrahydrofuran (15 ml). The mixture was stirred for 1 hour at room temperature, and the precipitated solid was filtered. Crystallization from acetone gave the corresponding 3,4-diaryl-5,6-dihydrobenzo[*h*]quinolin-2(1*H*)-ones V. The results are reported in Table 2.

Action of Acetic Anhydride on the 2(1*H*)-Quinolones V.

A mixture of the dihydro-2(1*H*)-quinolone (1.0 g) and acetic anhydride (3 ml) was refluxed on a water bath for two hours. The cold reaction mixture was worked up as usual [11]. The solid product was crystallized from ethanol to give the corresponding 2-acetoxy-3,4-diaryl-5,6-dihydrobenzo-[*h*]quinolines VI (*cf.* Table 2).

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