CYCLIZATION REACTIONS OF KETENIMINES WITH SOME NITROGEN CONTAINING COMPOUNDS

Jan SVĚTLÍK[a]* and Augustín MARTVOŇ[b]

[a] *Department of Organic Chemistry, Slovak Institute of Technology, 880 37 Bratislava, and*
[b] *Drug Research Institute, 801 00 Bratislava*

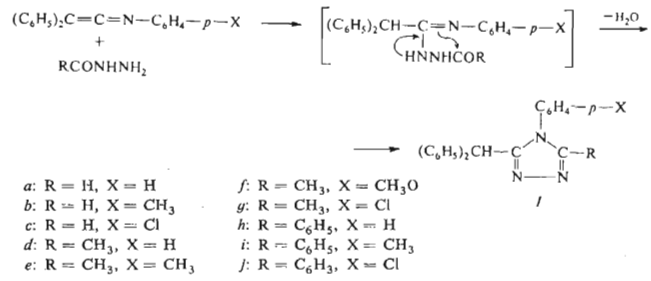
Received May 15th, 1980

3,4-Disubstituted, 3,4,5-trisubstituted 4*H*-1,2,4-triazoles and 2,3-disubstituted 4(3*H*)-quinazolinones were prepared in one step by cyclization reactions of ketenimines with formohydrazide, acetohydrazide, benzohydrazide, carbodihydrazide, thiosemicarbazide or anthranilic acid in xylene or dimethylformamide. Reaction of diphenylketene-N-phenylimine with aminoacetaldehyde dimethyl acetal gave an adduct at the C=C bond, which afforded 2-diphenylmethyl-1-phenylimidazole with dilute HCl. ^{13}C-NMR spectra of triazoles *Id*, *IIa* and *IIb* are commented.

The synthetic application of ketenimines in heterocyclic chemistry is first of all based on cycloaddition reactions[1], nevertheless the number of the realized cyclization reactions is, when compared with other heterocumulenes, poor[1-3]. As known, ketenimines readily react with nucleophilic reagents at the C=C bond of the cumulene system of multiple bonds. Addition of primary amines led to N,N'-disubstituted amidines, whereas phenylhydrazine gave the corresponding amidrazones[1]. Species possessing further appropriate functional group react with the title heterocumulenes to furnish some nitrogen containing heterocycles *via* the addition-cyclization mechanism.

Thus, in xylene triarylketenimines and carboxylic acid hydrazides give substituted 1,2,4-triazoles *Ia–Ij* in good yields directly in one step. The ring closure of the formed adduct occurred immediately by the loss of water (Scheme 1). The proposed reaction pathway is in line with the described methods of preparation of 1,2,4-triazoles by a base or thermal cyclization of amidrazones[4]. The effort to isolate the anticipated intermediate failed. Under milder conditions (dichloromethane, 41°C, 5 h) the yield of *Id* dropped from 75% to 62%. With benzoic hydrazide also diphenylacetamides, originating from the respective ketenimide and reaction water, were isolated in a small amount. An analogous interaction of ketenimine with carbodihydrazide afforded 3-diphenylmethyl-4-phenyl-4*H*-1,2,4-triazol-5-one (*IIa*) in accordance with the proposed reaction pathway. The same compound was prepared from ethoxycarbonyl-

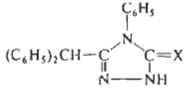
* Present address: Drug Research Institute, 801 00 Bratislava.



a: R = H, X = H
b: R = H, X = CH_3
c: R = H, X = Cl
d: R = CH_3, X = H
e: R = CH_3, X = CH_3
f: R = CH_3, X = CH_3O
g: R = CH_3, X = Cl
h: R = C_6H_5, X = H
i: R = C_6H_5, X = CH_3
j: R = C_6H_5, X = Cl

SCHEME 1

hydrazine and diphenylketene-N-phenylimine by heating in benzene. Thiosemicarbazide in dimethylformamide gave *IIb* in a 52% yield.



IIa, X = O
IIb, X = S

Aminoacetaldehyde dimethyl acetal was chosen for this reaction as a bifunctional amino compound. It is worth noting that in this case the product of addition *III* was isolated; it undergoes cyclization with 25% HCl to furnish an imidazole derivative

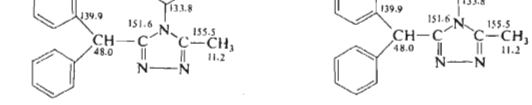
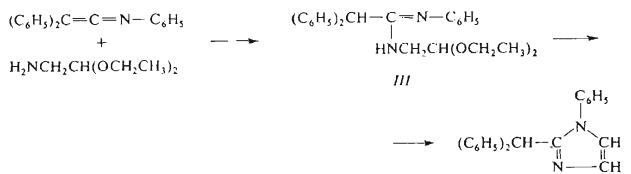


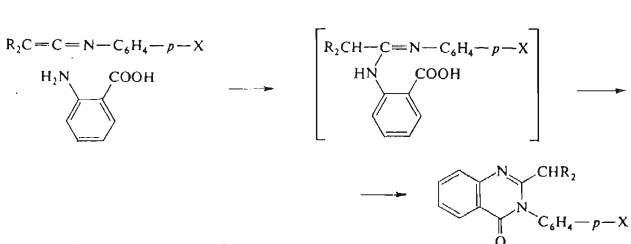
FIG. 1
Carbon-13 NMR Data of *Id* ($CDCl_3$ solvent, tetramethylsilane internal standard)

IV (Scheme 2). Of the synthesized compounds the ^{13}C-NMR spectrum of 3-diphenylmethyl-5-methyl-4-phenyl-4*H*-1,2,4-triazole (*Id*) was measured. The respective carbons of the benzhydryl group were ascribed on the basis of relative peak heights and of the comparison with known values of chemical shifts of diphenylacetic acid[5]. Also the downfield shift of *para*- and *meta*-carbons of the N-phenyl group in relation to the *ortho*-carbons was in line with that reported[6]. The inspection of spectra of compounds *Id*, *IIa*, *IIb* made it possible to distinguish both heteroring carbons (Fig. 1). Following resonation frequencies were assigned to the last two triazole rings (relative to tetramethylsilane by correcting the internal $(CD_3)_2SO$ reference): 151·7 (C—3) and 158·4 (C=O) in *IIa* and 153·1 (C—3) and 168·1 ppm (C=S) in *IIb*, respectively. In accordance with[7,8] our results also favour the tautomeric oxoform of *IIa*, or the thiono form of *IIb*.



SCHEME 2

The addition-cyclization mechanism was involved in the formation of disubstituted 4(3*H*)-quinazolinones *Va*—*Vf*, as well. These heterocycles were obtained in a fair yield by a 3 h boiling of the equimolar mixture of anthranilic acid and ketenimine



a; R = C_6H_5, X = H *d*; R = C_6H_5, X = Br
b; R = C_5H_5, X = CH_3O *e*; R = C_6H_5, X = Cl
c; R = C_6H_5, X = CH_3 *f*; R = CH_3, X = CH_3

SCHEME 3

in xylene. Cyclization occurred as a result of an intramolecular nucleophilic attack of the imino-nitrogen to an electron defficient carbon of the carboxylic group in the intermediate which has primarily been formed (Scheme 3).

The constitution of the prepared quinazolinones was proved by mass spectrometry. The peak due to a diphenylmethyl ion at m/z 167 dominated in addition to the molecular ion peak; the loss of a molecule of hydrogen and the concurrent skeletal rearrangement of aromatic rings led to a fluorenyl cation of m/z 165 (ref.[9]). ^{1}H-NMR spectrum showed the *peri*-proton towards carbonyl as a doublet with a fine triplet structure characteristic of a *meta* spin-spin coupling. Of diagnostic value was also the downfield shift of this proton by 0·5 ppm in relation to the remaining protons of the ring due to an anisotropy of the C=O group. Moreover, the IR spectra of the isolated compounds displayed very strong bands at 1680 and 1 609 cm^{-1} associated with the 4-quinazolinone system[10].

EXPERIMENTAL

The reaction course and purity of compounds were monitored by thin-layer chromatography (TLC) on Silufol (Kavalier, Votice) plates (visualization with iodine vapours). Melting points were measured with a micro hot-stage Boëtius. The IR spectra were recorded with a UR-20 (Zeiss, Jena) spectrometer as saturated chloroform solutions (0·6 mm cell width) or in KBr discs in the 700—3 800 cm^{-1} range. The ^{1}H-NMR spectra were run with a BS 487 C (Tesla, Brno) instrument operating at 80 MHz, tetramethylsilane being the internal standard. The ^{13}C-NMR spectra were recorded with a Jeol FX-100 NMR spectrometer (Fourier transform) with a proton noise decoupling. The saturated solution of the sample was measured in a 10-mm tube at 22°C, 2 000 FID was accumulated with 5000 Hz spectral width using an 8 K data points. The mass spectra were recorded with an MS 902 S apparatus (AEI, Manchester) at 70 eV, trap current 100 μA, and ionization chamber temperature 115°C (*Ia*), 130°C (*Id*) and 175°C (*IIb*).

Cyclization of Ketenimines with Hydrazides

A solution of ketenimine (2 mmol) and an equimolar amount of hydrazide in xylene (15 ml) were refluxed for 3 h, the solvent was removed under reduced pressure and ether (5 ml) was added to the remaining oil. The precipitate was filtered off, washed with light petroleum and crystallized from ethanol. Derivatives *Ih—Ij* were chromatographed on a silica gel column (eluent chloroform) and the products were crystallized from ethanol. In addition to the unreacted ketenimine (1st fraction, detection on TLC) also anilides of diphenylacetic acid (2nd fraction, identification by mass spectrometry) were isolated in a minute amount.

3-*Diphenylmethyl*-4-*phenyl*-4*H*-1,2,4-*triazole* (Ia): Yield 47%, m.p. 137—139°C. IR spectrum, cm^{-1} ($CHCl_3$): 3 000, 1 600, 1 560, 1 466, 1 169. ^{1}H-NMR spectrum ($CDCl_3$): 8·20 (1 H, s, H—5), 7·55—6·98 (5 H, m, C_6H_5), 7·23 (10 H, s, C_6H_5), 5·30 (1 H, s, CH). Mass spectrum, m/z, (rel. intensity, %): 312 (18), 311, M^+ (84), 310 (10), 296 (7), 242 (6), 241 (12), 234 (8), 233 (12), 232 (6), 220 (11), 206 (7), 205 (8), 193 (4), 190 (5), 180 (5), 178 (7), 167 (7), 166 (13), 165 (53), 152 (11), 104 (6), 91 (11), 77 (30). For $C_{21}H_{17}N_3$ (311·4) calculated: 81·00% C, 5·50% H, 13·49% N; found: 81·12% C, 5·45% H, 13·39% N.

3-*Diphenylmethyl*-4-(4-*tolyl*)-4*H*-1,2,4-*triazole* (Ib): Yield 51%; m.p. 185—186°C. IR spectrum cm^{-1} ($CHCl_3$): 3000, 2924 sh, 1601, 1504, 1166. ^{1}H-NMR spectrum ($CDCl_3$): 8·17 (1 H, s, 5-H), 7·24 (11 H, s, C_6H_5 + 1 H of *p*-tolyl), 7·18, 6·98, 6·88 (3 H portion of the AA′BB′ system, *p*-tolyl), 5·15 (1 H, s, CH), 2·40 (3 H, s, CH_3). For $C_{22}H_{19}N_3$ (325·4) calculated: 81·20% C, 5·88% H, 12·91% N; found: 81·10% C, 5·79% H, 13·01% N.

4-(4-*Chlorophenyl*)-3-*diphenylmethyl*-4*H*-1,2,4-*triazole* (Ic): Yield 56%; m.p. 204—206°C. IR spectrum cm^{-1} ($CHCl_3$): 3000, 1601, 1511, 1171, 1096, 1014, 836. ^{1}H-NMR spectrum ($CDCl_3$): 8·13 (1 H, s, 5-H), 7·23 (10 H, s, C_6H_5), 7·43, 7·33, 7·03, 6·92 (4 H, AA′BB′ system, *p*-Cl-C_6H_4). For $C_{21}H_{16}ClN_3$ (345·8) calculated: 72·93% C, 4·66% H, 12·15% N, 10·25% Cl, found: 73·00% C, 4·72% H, 12·01% N, 10·19% Cl.

3-*Diphenylmethyl*-5-*methyl*-4-*phenyl*-4*H*-1,2,4-*triazole* (Id): Yield 75%; m.p. 186—187°C. IR spectrum cm^{-1} ($CHCl_3$): 2995, 1600, 1511, 1456, 1431. ^{1}H-NMR spectrum ($CDCl_3$): 7·17 (10 H, s, C_6H_5), 7·50—6·87 (5 H, m, C_6H_5), 5·11 (1 H, s, CH), 2·33 (3 H, s, CH_3). Mass spectrum, (*m*/*z*, ref. intensity, %): 326 (15), 325, M^+ (64), 324 (100), 310 (6), 248 (6), 247 (8), 242 (4), 241 (8), 234 (8), 206 (7), 205 (8), 180 (4), 178 (5), 167 (6), 166 (10), 165 (39), 152 (9), 118 (12), 91 (8), 77 (27). For $C_{22}H_{19}N_3$ (325·4) calculated: 81·20% C, 5·88% H, 12·91% N; found: 81·25% C, 5·90% H, 13·00% N.

3-*Diphenylmethyl*-5-*methyl*-4-(4-*tolyl*)-4*H*-1,2,4-*triazole* (Ie): Yield 67%; m.p. 176—177°C. IR spectrum cm^{-1} ($CHCl_3$): 3004, 2926 sh, 1601, 1500, 1456, 1056, 879. ^{1}H-NMR spectrum ($CDCl_3$): 7·21 (12 H, s, C_6H_5 + 2 H of *p*-tolyl), 6·86, 6·75 (2 H, portion of the AA′BB′ system, *p*-tolyl), 5·10 (1 H, s, CH), 2·40 (3 H, s, 5-CH_3), 2·20 (3 H, s, CH_3 of *p*-tolyl. For $C_{23}H_{21}N_3$ (339·4) calculated: 81·38% C, 6·24% H, 12·38% N; found: 81·22% C, 6·33% H, 12·30% N.

3-*Diphenylmethyl*-4-(4-*methoxyphenyl*)-5-*methyl*-4*H*-1,2,4-*triazole* (If): Yield 49%; m.p. 191 to 193°C. IR spectrum cm^{-1} ($CHCl_3$): 3000, 2936 sh, 1609, 1589, 1521, 1460, 1304, 1261, 1106, 1036. ^{1}H-NMR spectrum ($CDCl_3$): 7·21 (10 H, s, C_6H_5), 6·98, 6·86, 6·74 (4 H, an eclipsed AA′BB′ system, *p*-CH_3O—C_6H_4), 5·10 (1 H, s, CH), 3·80 (3 H, s, CH_3O), 2·40 (3 H, s, 5-CH_3). For $C_{27}H_{21}N_3O$ (355·4) calculated: 77·72% C, 5·96% H, 11·82% N; found: 77·88% C, 5·62% H, 11·71% N.

4-(4-*Chlorophenyl*)-3-*diphenylmethyl*-5-*methyl*-4*H*-1,2,4-*triazole* (Ig): Yield 51%; m.p. 145 to 146°C. IR spectrum cm^{-1} ($CHCl_3$): 3000, 1602, 1505, 1106. ^{1}H-NMR spectrum ($CDCl_3$): 7·21 (12 H, s, C_6H_5 + 2 H from *p*-Cl—C_6H_5), 6·86, 6·75 (2 H, portion of the AA′BB′ system, *p*-Cl—C_6H_5), 5·08 (1 H, s, CH), 2·20 (3 H, s, 5-CH_3). For $C_{22}H_{18}ClN_3$ (359·6) calculated: 73·49% C, 5·05% H, 11·69% N, 9·86% Cl, found: 73·58% C, 4·99% H, 11·81% N, 9·75% Cl.

3-*Diphenylmethyl*-4,5-*diphenyl*-4*H*-1,2,4-*triazole* (Ih): Yield 36%; m.p. 156—158°C. IR spectrum cm^{-1} ($CHCl_3$): 3055, 1607, 1504, 1455. ^{1}H-NMR spectrum ($CDCl_3$): 7·18 (10 H, s, C_6H_5), 7·48—6·81 (10 H, m, C_6H_5), 5·15 (1 H, s, CH). For $C_{27}H_{21}N_3$ (387·5) calculated: 83·69% C, 5·46% H, 10·84% N; found: 83·79% C, 5·29% H, 10·68% N.

3-*Diphenylmethyl*-5-*phenyl*-4-(4-*tolyl*)-4*H*-1,2,4-*triazole* (Ii): Yield 45%; m.p. 163—164°C. IR spectrum cm^{-1} ($CHCl_3$): 3000, 2975, 1605, 1519, 1500, 1457, 1046. ^{1}H-NMR spectrum ($CDCl_3$): 7·50—7·10 (17 H, m, C_6H_5 + 2 H from *p*-tolyl), 6·86, 6·76 (2 H, portion of the AA′BB′ system, *p*-tolyl), 5·17 (1 H, s, CH), 2·38 (3 H, s, CH_3). For $C_{28}H_{23}N_3$ (401·5) calculated: 83·76% C, 5·77% H, 10·46% N; found: 83·91% C, 5·54% H, 10·41% N.

4-(4-*Chlorophenyl*)-3-*diphenylmethyl*-5-*phenyl*-4*H*-1,2,4-*triazole* (Ij): Yield 51%; m.p. 138 to 141°C. IR spectrum cm^{-1} ($CHCl_3$): 3000, 1604, 1506, 1096. ^{1}H-NMR spectrum ($CDCl_3$): 7·46—7·10 (17 H, m, C_6H_5 + 2 H from *p*-Cl—C_6H_4), 6·89, 6·78 (2 H, portion of the AA′BB′

system, *p*-Cl—C_6H_4), 5·14 (1 H, s, CH). For $C_{27}H_{20}ClN_3$ (421·9) calculated: 76·86% C, 4·78% H, 9·96% N, 8·40% Cl; found: 76·62% C, 4·55% H, 10·06% N, 8·44% Cl.

3-Diphenylmethyl-4-phenyl-4*H*-1,2,4-triazol-5-one (*IIa*)

A) A solution of diphenylketene-N-phenylimine (2·3 g, 8·55 mmol) and carbodihydrazide (0·7 g) in dimethylformamide (15 ml) was refluxed for 2·5 h. The solvent was removed under reduced pressure and the remaining solid was washed with water and crystallized from methanol. Yield 0·4 g (15%), m.p. 226—228°C. IR spectrum, cm^{-1} (KBr): 3360, 3145, 3058, 2988, 1722, 1574, 1502, 1335. ^{1}H-NMR spectrum (hexadeuteriodimethyl sulfoxide): 7·18 (10 H, s, C_6H_5), 7·55—6·93 (5 H, m, C_6H_5), 5·23 (1 H, s, CH). For $C_{21}H_{17}N_3O$ (327·4) calculated: 77·04% C, 5·23% H, 12·84% N; found: 77·15% C, 5·31% H, 12·69% N.

B) Ketenimine (0·8 g, 2·97 mmol) and ethoxycarbonylhydrazine (0·3 g) in benzene (20 ml) were refluxed for 1·5 h. After the solvent had been removed, ether (5 ml) was added and the precipitate suction filtered and crystallized from ethanol. Yield 97 mg (10%).

3-Diphenylmethyl-4-phenyl-4*H*-1,2,4-triazole-5-thione (*IIb*)

A solution of ketenimine (0·9 g, 3·35 mmol) and thiosemicarbazide (0·3 g) in dimethylformamide (15 ml) was refluxed for 1 h, concentrated and the residue crystalized from 90% ethanol. Yield 0·6 g (52%), m.p. 265—268°C. IR spectrum, cm^{-1} (KBr): 3407, 3037, 1593, 1570, 1456, 1412, 1340, 1311, 1292. ^{1}H-NMR spectrum (hexadeuteriodimethyl sulfoxide): 7·49—6·90 (15 H, m, C_6H_5), 5·20 (1 H, s, CH). Mass spectrum, *m/z*, (rel. intensity, %): 344 (26), 343, M^+ (100), 342 (40), 311 (13), 310 (18), 203 (6), 193 (6), 192 (8), 178 (5), 167 (16), 166 (10), 165 (34), 152 (11), 105 (9), 91 (5), 77 (21). For $C_{21}H_{17}N_3S$ (343·5) calculated: 73·44% C, 4·99% H, 12·23% N, 9·34% S; found: 73·65% C, 5·08% H, 12·04% N, 9·51% S.

Reaction with Aminoacetaldehyde Dimethyl Acetal

A) A solution of ketenimine (0·5 g, 1·86 mmol) and aminoacetaldehyde dimethyl acetal (0·25 g) in benzene (10 ml) was refluxed for 6 h, the solvent removed and the residue chromatographed on a silica gel column (eluent chloroform). Addition of light petroleum to the concentrated fraction resulted in separation of adduct *III* (0·5 g, 67%), m.p. 73—74°C. ^{1}H-NMR spectrum ($CDCl_3$): 7·45—6·52 (15 H, m, C_6H_5), 5·28 (1 H, s, CH from benzhydryl), 4·83 (2 H, m, CH + + NH), 3·53 (6 H, m, CH_2), 2·33 (6 H, t, CH_3). For $C_{26}H_{30}N_2O_2$ (402·5) calculated: 77·58% C, 7·51% H, 6·96% N; found: 77·69% C, 7·70% H, 6·73% N.

B) Dilute HCl (25%, 6 ml) was added to the adduct *III* (0·3 g), the mixture was simmered for 1·5 h, neutralized with 30% KOH and poured onto a crushed ice. The separated precipitate was filtered off, dried and recrystallized from acetone. Yield 0·14 g (61%) of 2-diphenylmethyl--1-phenylimidazole (*IV*); m.p. 131—133°C. IR spectrum, cm^{-1} ($CHCl_3$): 3015, 1601, 1508, 1458, 1308, 1166, 1120, 1100. ^{1}H-NMR spectrum ($CDCl_3$): 7·44—6·96 (17 H, m, C_6H_5 + 2 H from imidazole), 5·32 (1 H, s, CH). For $C_{22}H_{18}N_2$ (310·4) calculated: 85·13% C, 5·85% H, 9·02% N; found: 85·30% C, 5·82% H, 9·01% N.

Reaction with Anthranilic Acid

The cyclization was carried out according to the procedure given with hydrazides. The isolated crude products were crystallized from acetone.

2-*Diphenylmethyl*-3-*phenyl*-4(3*H*)-*quinazolinone* (Va): Yield 32%; m.p. 176—177°C. IR spectrum ($CHCl_3$): 3081, 1680, 1609, 1591, 1498, 1474, 1361, 1336, 1303, 1118, 1088, 1078. ^{1}H-NMR spectrum ($CDCl_3$): 8·25 (1 H, d, H—5), 7·13 (10 H, s, C_6H_5), 7·73—6·93 (8 H, m, C_6H_5—N + + H—6 + H—7 + H—8), 5·18 (1 H, s, CH). For $C_{27}H_{20}N_2O$ (388·5) calculated: 83·48% C, 5·19% H, 7·21% N; found: 83·20% C, 5·11% H, 7·39% N.

2-*Diphenylmethyl*-3-(4-*methoxyphenyl*)-4(3*H*)-*quinazolinone* (Vb): Yield 34%; m.p. 189—190°C. IR spectrum, cm^{-1} ($CHCl_3$): 3002, 2925, 1679, 1610, 1595, 1498, 1458, 1475, 1362, 1338, 1304, 1282, 1111. ^{1}H-NMR spectrum ($CDCl_3$): 8·26 (1 H, d, H—5), 7·75—7·04 (3 H, m, H—6 + + H—7 + H—8), 7·18 (10 H, s, C_6H_5), 6·89 (4 H, s, an eclipsed AA'BB' system, *p*-CH_3O—C_6H_4), 5·24 (1 H, s, CH), 3·84 (3 H, s, CH_3O). For $C_{28}H_{22}N_2O_2$ (418·5) calculated: 80·36% C, 5·30% H, 6·69% N; found: 80·19% C, 5·42% H, 6·74% N.

2-*Diphenylmethyl*-3-(4-*tolyl*)-4(3*H*)-*quinazolinone* (Vc): Yield 31%; m.p. 173—174°C. IR spectrum, cm^{-1} ($CHCl_3$): 3066, 2961, 1677, 1609, 1593, 1571, 1516, 1475, 1299. ^{1}H-NMR spectrum ($CDCl_3$): 8·25 (1 H, d, H—5), 7·77—7·03 (4 H, m, H—6 + H—7 + H—8 + 1 H from *p*-tolyl), 7·28, 6·94, 6·83 (3 H, portion of the AA'BB' system, *p*-tolyl), 5·21 (1 H, s, CH), 2·40 (3 H, s, CH_3). For $C_{28}H_{22}N_2O$ (402·5) calculated: 83·56% C, 5·51% H, 6·96% N; found: 83·59% C, 5·50% H, 6·90% N.

3-(4-*Bromophenyl*)-2-*diphenylmethyl*-4(3*H*)-*quinazolinone* (Vd): Yield 75% m.p. 208—210°C. IR spectrum, cm^{-1} ($CHCl_3$): 3083, 1693, 1608, 1361, 1337, 1109, 1081, 1017. ^{1}H-NMR spectrum ($CDCl_3$): 8·23 (1 H, d, H—5), 7·75—7·02 (3 H, m, H—6 + H—7 + H—8), 7·15 (10 H, s, C_6H_5), 7·58, 7·47, 6·88, 6·78 (4 H, AA'BB' system, *p*-Br-C_6H_4), 5·10 (1 H, s, CH). For $C_{27}H_{19}BrN_2O$ (467·4) calculated: 69·39% C, 4·10% H, 5·99% N, 17·09% Br; found: 69·48% C, 3·98% H, 6·08% N, 17·01% Br.

3-(4-*Chlorophenyl*)-2-*diphenylmethyl*-4(3*H*)-*quinazolinone* (Ve): Yield 30%; m.p. 197—198°C. IR spectrum, cm^{-1} ($CHCl_3$): 3085, 1684, 1606, 1591s h, 1496, 1477, 1095. ^{1}H-NMR spectrum ($CDCl_3$): 8·24 (1 H, d, H—5), 7·75—7·03 (3 H, m, H—6 + H—7 + H—8), 7·18 (10 H, s, C_6H_5), 7·43, 7·31, 6·96, 6·85 (4 H, AA'BB' system, *p*-Cl—C_6H_4), 5·13 (1 H, s, CH). For $C_{27}H_{19}ClN_2O$ (422·9) calculated: 76·68% C, 4·53% H, 6·62% N, 8·38% Cl; found: 76·80% C, 4·45% H, 6·57% N, 8·25% Cl.

2-*Isopropyl*-3-(4-*tolyl*)-3(3*H*)-*quinazolinone* (Vf): Yield 23%; m.p. 146—148°C. IR spectrum, cm^{-1} ($CHCl_3$): 3075, 2969, 1595, 1477, 1386, 1336, 1307, 1110. ^{1}H-NMR spectrum ($CDCl_3$): 8·28 (1 H, d, H—5), 7·81—7·06 (7 H, m, H—6 + H—7 + H—8 + *p*-tolyl), 2·74 (1 H, m, CH), 2·45 (3 H, s, CH_3 of *p*-tolyl), 1·22 (6 H, d, CH_3). For $C_{18}H_{18}N_2O$ (278·4) calculated: 77·67% C, 6·52% H, 10·06% N; found: 77·51% C, 6·40% H, 10·19% N.

REFERENCES

1. Krow G. R.: Angew. Chem. *83*, 455 (1971).
2. Reimlinger H., Vandewalle J. J. M., Lingier W. R. F.: Chem. Ber. *103*, 1960 (1970).
3. Reimlinger H., Lingier W. R. F., Vandewalle J. J. M.: Chem. Ber. *104*, 639 (1971).
4. Watson K. H., Neilson D. G. in the book: *The Chemistry of Amidines and Imidates* (S. Patai, Ed.), p. 491. Wiley, New York 1979.
5. Bremser W., Ernst L., Franke B., Gerhards R., Hardt A.: *Carbon*-13 NMR *Spectral Data*. Verlag Chemie, Weinheim, New York 1979.
6. Stothers J. B.: *Carbon*-13 NMR *Spectroscopy*, p. 197. Academic Press, New York 1972.
7. Lichtman W. M., Hollstein U., Papadopoulos E. P.: Org. Magn. Resonance *11*, 137 (1978).

8. Milcent R., Redeuilh C.: J. Heterocycl. Chem. *16*, 430 (1979).
9. Světlík J., Martvoň A.: This Journal *44*, 2421 (1979).
10. Pakrashi S. C., Bhattacharyya J., Johnson L. F., Budzikiewicz H.: Tetrahedron *19*, 1011 (1963).

Translated by Z. Votický.