A One-Pot Synthesis of 1,2-Dihydroisoquinoline Derivatives from Isoquinoline *via* a Four-Component Reaction

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A four-component reaction for the synthesis of 1,2-dihydroisoquinoline derivatives is described. The *Huisgen* 1,4-dipolar intermediate, which is produced from isoquinoline and an electron-deficient acetylene compound **1**, reacts with H_2O in the presence of diketene to produce 1,2-dihydroisoquinoline derivatives **2** (*Scheme 1*). In addition, reaction of isoquinoline, dibenzoylacetylene (=1,4-diphenylbut-2-yne-1,4-dione), and diketene in the presence of H_2O leads to pyrroloisoquinoline derivative **7**. The structures of the compounds **2a**–**f** and **7** were corroborated spectroscopically (IR, ^{1}H- and ^{13}C-NMR, EI-MS) and by elemental analyses. A plausible mechanism for the reaction is proposed (*Schemes 2* and *3*).

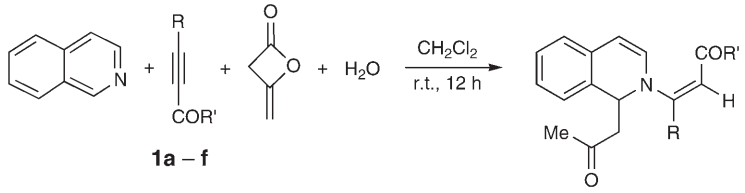
Introduction. – Isoquinoline derivatives refer to isoquinoline itself, substituted congeners (*e.g.*, 6-methoxyisoquinoline), and the various reduced species (1,2- or 3,4-dihydroisoquinolines and 1,2,3,4-tetrahydroisoquinolines), all of which may occur in their neutral or charged quaternary (isoquinolinium ion) form [1]. Not only frequently found in naturally occurring alkaloids, the di- and tetrahydroisoquinoline moieties are also important pharmacophores. Typical examples include indenoisoquinoline (topoisomerase I inhibitor) [2], papaverine (smooth-muscle relaxant) [3], saframycin-B (antitumor agent) [4], and narciclasine (antitumor agent) [5]. They are synthesized *in vivo* by a *Pictet-Spengler* nonenzymatic condensation of biogenic amines (*e.g.*, catecholamines and phenylethylamines) with aldehydes or with α-keto acids followed by decarboxylation [6][7]. Recent studies suggest that some isoquinoline derivatives may also be formed enzymatically in mammalian brain [8].

Huisgen demonstrated that the reaction of isoquinoline and electron-deficient acetylene compounds generates a 1,4-dipolar intermediate [9][10], and he trapped the latter with various dipolarophiles for the formation of five- and six-membered heterocycles [11]. In recent years, these interesting zwitterions have been used in multicomponent reactions to produce a considerable number of heterocyclic compounds [12–19].

In this paper, we report a simple one-pot reaction between isoquinoline, electron-deficient acetylene compounds **1a**–**f**, and H_2O in the presence of diketene, leading to 1,2-dihydroisoquinoline derivatives **2a**–**f** (*Scheme 1*).

Results and Discussion. – The one-pot reaction of isoquinoline, **1**, H_2O, and diketene proceeded in CH_2Cl_2 at room temperature to produce 1,2-dihydroisoquinoline derivatives **2a**–**f** in 85–95% yields (*Scheme 1*). The structures of compounds **2a**–**f**

Scheme 1



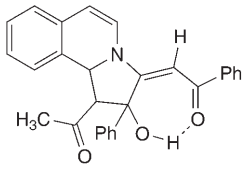
1a – f

	R'	R	Yield [%]
2a	MeO	CO_2Me	95
2b	EtO	CO_2Et	93
2c	^{t}BuO	$CO_2{}^t$Bu	90
2d	MeO	H	88
2e	EtO	H	85
2f	Me	H	85

were deduced from their elemental analysis, IR, and ^{1}H- and ^{13}C-NMR spectra. The MS of **2a** displayed the molecular ion peak at *m*/*z* 329, which is consistent with a 1:1:1:1 adduct of isoquinoline, dimethyl but-2-ynedioate, diketene, and H_2O having lost CO_2.

The ^{1}H-NMR spectrum of **2a** exhibited seven signals readily recognized as arising from three Me groups (δ 1.99, 3.68, and 3.95), a CH_2 group of (2.63 ($J = 2.5$, 16.9 Hz) and 3.11 ($J = 9.7$, 16.9 Hz)), an olefinic CH moiety (δ 5.21), and a CH group (δ 5.37, $J = 9.9$ Hz). The isoquinoline moieties gave rise to characteristic signals in the aromatic region of the spectrum. No exchange was observed in the ^{1}H-NMR spectrum of **2a** in D_2O solution. The ^{1}H-decoupled ^{13}C-NMR spectrum of **2a** showed 18 distinct resonances in agreement with the structure of dimethyl 2-[1-(2-oxopropyl)isoquinolin-2(1*H*)-yl]but-2-enedioate. The ^{1}H- and ^{13}C-NMR spectra of compounds **2b** – **f** were similar to those of **2a**, except for the ester moieties, which exhibited characteristic signals with appropriate chemical shifts for the specific substitution patterns.

When dibenzoylacetylene (=1,4-diphenylbut-2-yne-1,4-dione) and isoquinoline in dry CH_2Cl_2 were allowed to react with H_2O in the presence of diketene at room temperature, a yellow crystalline compound **7** was formed in nearly quantitative yield. This product was identified as (2*E*)-2-{1-acetyl-1,10b-dihydro-2-hydroxy-2-phenylpyrrolo[2,1-*a*]isoquinolin-3(2*H*)-ylidene}-1-phenylethanone (**7**).



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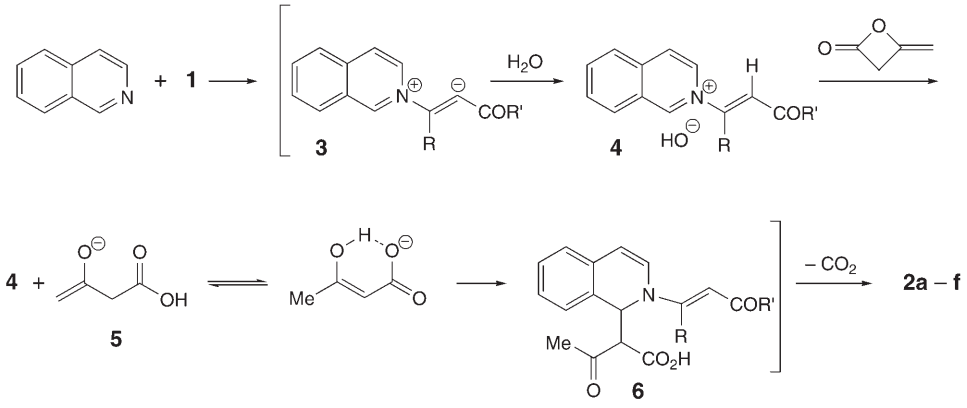
Although the mechanism of the reaction between isoquinoline, electron-deficient acetylene compounds **1**, and diketene in the presence of H_2O has not yet been established in an experimental manner, a putative mechanism is proposed in *Scheme 2*.

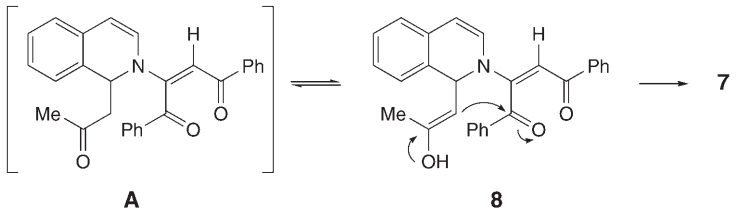
Based on the well-established chemistry of N-containing heterocycles [12–21], it is reasonable to assume that products **2a–f** result from initial addition of isoquinoline to the electron-deficient acetylene compound and subsequent protonation of the 1:1 adduct **3** by H_2O. On the other hand (in a concerted process), an OH^- ion attacks the diketene, and after a proton exchange [22–28], **5** is produced. Next, the positively charged ion of **4** is attacked by the conjugated base **5** of acetoacetic acid to form intermediate **6** that looses a molecule of CO_2 and, therefore, leads to compounds **2a–f**.

Scheme 2



Compound **7** apparently results from the enolization and cyclization of intermediate **A**, when dibenzoylacetylene was used as an electron-deficient acetylene compound (*Scheme 3*) [29].

Scheme 3



In conclusion, we have developed a convenient one-pot method for preparing isoquinoline derivatives. The present method has the following advantage: not only the reaction is performed under neutral and aqueous conditions, but also the substrates can be used in the reaction without any prior activation or modification. The simplicity of the present procedure makes it an interesting alternative to complex multistep approaches.

Experimental Part

General. Isoquinoline, diketene, and electron-deficient acetylene compounds were obtained from *Merck* (Germany) and *Fluka* (Switzerland) and were used without further purification. Dibenzoylacetylene (=1,4-diphenylbut-2-yne-1,4-dione) was prepared according to a published procedure [30][31]. Melting points: *Electrothermal-9100* apparatus. IR Spectra: *Shimadzu-IR-460* spectrometer. ^{1}H- and ^{13}C-NMR Spectra: $CDCl_3$ solns.; *Bruker-DRX-500-Avance* spectrometer at 500.1 and 125.7 MHz, resp. MS: *Finnigan-MAT-8430* mass spectrometer operating at an ionization potential of 20 eV. Elemental analyses: *Heraeus-CHN-O-Rapid* analyzer.

Compounds **2a**–**f**: *General Procedure* (exemplified for **2a**). To a magnetically stirred soln. of isoquinoline (0.13 g, 1 mmol) and H_2O (0.02 g, 1 mmol) in CH_2Cl_2 (2 ml) was added dropwise a soln. of diketene (0.08 g, 1 mmol) and dimethyl but-2-ynedioate (DMAD; 0.14 g, 1 mmol) at 0°. The mixture was stirred for 10 h at r.t. The solvent was evaporated, and the residue was separated by column chromatography (silica gel (230–240 mesh; *Merck*) hexane/AcOEt).

*Dimethyl (2Z)-2-[1-(2-Oxopropyl)isoquinolin-2(1*H*)-yl]but-2-enedioate* (**2a**): Yield 310 mg (95%). Pale yellow powder. M.p. 123–125°. IR (KBr): 1728 (COMe), 1696 (CO_2Me), 1581 (C=C), 1556 and 1508 (arom.), 1215 and 1150 (C–O, ester). ^{1}H-NMR ($CDCl_3$): 1.99 (*s*, 3 H); 2.63 (*dd*, *J* = 16.9, 2.5, 1 H); 3.11 (*dd*, *J* = 16.9, 9.7, 1 H); 3.68 (*s*, 3 H); 3.95 (*s*, 3 H); 5.21 (*s*, 1 H); 5.37 (*d*, *J* = 9.9, 1 H); 5.95 (*d*, *J* = 7.6, 1 H); 6.27 (*d*, *J* = 7.6, 1 H); 7.07 (*d*, *J* = 7.4, 1 H); 7.14–7.16 (*m*, 2 H); 7.21–7.23 (*m*, 1 H). ^{13}C-NMR ($CDCl_3$): 31.1; 47.3; 51.3; 53.3; 53.9; 90.7; 110.2; 125.0; 125.3; 126.4; 127.5; 128.3; 129.1; 131.3; 149.2; 165.1; 166.7, 205.3. EI-MS: 329 (25, M^+), 273 (63), 272 (100), 270 (30), 240 (19), 167 (19), 154 (24), 128 (38), 127 (38), 115 (19), 102 (11), 77 (13), 69 (9), 59 (12), 43 (38). Anal. calc. for $C_{18}H_{19}NO_5$ (329.35): C 65.64, H 5.81, N 4.25; found: C 65.51, H 5.91, N 4.46.

*Diethyl (2Z)-2-[1-(2-Oxopropyl)isoquinolin-2(1*H*)-yl]but-2-enedioate* (**2b**): Yield 330 mg (93%). Pale yellow powder. M.p. 105–107°. IR (KBr): 1718 (COMe), 1699 (CO_2Et), 1578 (C=C), 1556 and 1499 (arom.), 1200 and 1150 (C–O, ester). ^{1}H-NMR ($CDCl_3$): 1.26 (*t*, *J* = 7.1, 3 H); 1.39 (*t*, *J* = 7.1, 3 H); 1.99 (*s*, 3 H); 2.74 (*dd*, *J* = 16.9, 2.4, 1 H); 3.13 (*dd*, *J* = 16.9, 9.9, 1 H); 4.10–4.17 (*m*, 2 H); 4.37–4.47 (*m*, 2 H); 5.19 (*s*, 1 H); 5.37 (*d*, *J* = 9.5, 1 H); 5.95 (*d*, *J* = 7.6, 1 H); 6.30 (*d*, *J* = 7.6, 1 H); 7.07 (*d*, *J* = 7.4, 1 H); 7.13–7.15 (*m*, 2 H); 7.19–7.23 (*m*, 1 H). ^{13}C-NMR ($CDCl_3$): 13.9; 14.4; 31.1; 47.2; 53.8; 59.9; 62.6; 91.1; 110.0; 124.9; 125.4; 126.5; 127.4; 128.2; 129.2; 131.3; 149.1; 164.6; 166.7; 205.3. EI-MS: 357 (9, M^+), 312 (5), 300 (100), 284 (16), 272 (16), 200 (30), 154 (16), 128 (14), 43 (14). Anal. calc. for $C_{20}H_{23}NO_5$ (357.40): C 67.21, H 6.49, N 3.92; found: C 67.44, H 6.31, N 3.87.

*Di(*tert*-butyl) (2Z)-2-[1-(2-Oxopropyl)isoquinolin-2(1*H*)-yl]but-2-enedioate* (**2c**): Yield 370 mg (90%). Pale yellow powder. M.p. 120–122°. IR (KBr): 1719 (COMe), 1690 (CO_2*t*Bu), 1577 (C=C), 1556 and 1509 (arom.), 1223 and 1151 (C–O, ester). ^{1}H-NMR ($CDCl_3$): 1.46 (*s*, 9 H); 1.60 (*s*, 9 H); 1.97 (*s*, 3 H); 2.73 (*dd*, *J* = 16.8, 2.7, 1 H); 3.15 (*dd*, *J* = 16.8, 10.2, 1 H); 5.08 (*s*, 1 H); 5.32 (*d*, *J* = 9.7, 1 H); 5.92 (*d*, *J* = 7.6, 1 H); 6.35 (*d*, *J* = 7.6, 1 H); 7.05 (*d*, *J* = 7.5, 1 H); 7.12–7.20 (*m*, 3 H). ^{13}C-NMR ($CDCl_3$): 27.9; 28.3; 31.1; 46.9; 53.9; 79.5; 83.9; 93.2; 109.2; 124.7; 125.8; 126.7; 127.0; 128.1; 129.6; 131.3; 149.0; 163.5; 165.9; 205.6. EI-MS: 413 (4, M^+), 356 (12), 300 (19), 244 (51), 200 (25), 154 (13), 57 (100), 43 (32). Anal. calc. for $C_{24}H_{31}NO_5$ (413.51): C 69.71, H 7.56, N 3.39; found: C 69.53, H 7.32, N 3.46.

*Methyl (2Z)-3-[1-(2-Oxopropyl)isoquinolin-2(1*H*)-yl]prop-2-enoate* (**2d**): Yield 240 mg (88%). Pale yellow powder. M.p. 105–107°. IR (KBr): 1720 (COMe), 1690 (CO_2Me), 1602 (C=C), 1555 and 1520 (arom.), 1205 and 1157 (C–O, ester). ^{1}H-NMR ($CDCl_3$): 2.04 (*s*, 3 H); 2.74–2.76 (*m*, 1 H); 2.98–3.07 (*m*, 1 H); 3.71 (*s*, 3 H); 5.16 (*d*, *J* = 13.5, 1 H); 5.41 (br., 1 H); 5.84 (*d*, *J* = 7.3, 1 H); 6.33 (*d*, *J* = 7.3, 1 H); 7.05 (*d*, *J* = 7.5, 1 H); 7.13–7.15 (*m*, 2 H); 7.21–7.23 (*m*, 1 H); 7.46 (*d*, *J* = 13.5, 1 H). ^{13}C-NMR ($CDCl_3$): 31.1; 47.3; 51.0; 52.1; 90.8; 108.5; 124.84; 125.8; 126.2; 127.2; 128.2; 129.8; 130.8; 146.8; 168.9; 205.4. EI-MS: 271 (12, M^+), 265 (9), 254 (6), 238 (24), 214 (100), 154 (11), 129 (21), 115 (7), 102 (9), 84 (15), 43 (18). Anal. calc. for $C_{16}H_{17}NO_3$ (271.31): C 70.83, H 6.32, N 5.16; found: C 70.73, H 6.55, N 5.34.

*Ethyl (2Z)-3-[1-(2-Oxopropyl)isoquinolin-2(1*H*)-yl]prop-1-enoate* (**2e**): Yield 240 mg (85%). Pale yellow powder. M.p. 90–92°. IR (KBr): 1716 (COMe), 1695 (CO_2Et), 1620 (C=C), 1541 and 1510 (arom.), 1284 and 1201 (C–O, ester). ^{1}H-NMR ($CDCl_3$): 1.27 (*t*, *J* = 7.1, 3 H); 2.00 (*s*, 3 H); 2.72–2.75 (*m*, 1 H); 2.98–3.00 (*m*, 1 H); 4.12–4.20 (*m*, 2 H); 5.16 (*d*, *J* = 13.6, 1 H); 5.39 (br., 1 H); 5.85 (*d*, *J* = 7.3, 1 H); 6.33 (*d*, *J* = 7.3, 1 H); 7.04 (*d*, *J* = 7.5, 1 H); 7.10–7.14 (*m*, 2 H); 7.20–7.22 (*m*, 1 H); 7.44 (*d*, *J* = 13.6,

1 H). ^{13}C-NMR ($CDCl_3$): 14.5; 31.1; 47.2; 53.8; 59.6; 91.2; 108.7; 124.8; 125.8; 126.3; 127.2; 128.2; 129.8; 130.7; 146.6; 168.5; 205.5. EI-MS: 285 (20, M^+), 240 (11), 228 (100), 200 (85), 154 (13), 129 (19), 115 (9), 43 (12). Anal. calc. for $C_{17}H_{19}NO_3$ (285.34): C 71.56, H 6.71, N 4.91; found: C 71.61, H 6.62, N 4.76.

*(3*Z*)-4-[1-(2-Oxopropyl)isoquinolin-2(1*H*)-yl]but-3-en-2-one* (**2f**): Yield 220 mg (85%). Pale yellow powder. M.p. 100–102°. IR (KBr): 1718 (COMe), 1697 (CO_2Me), 1593 (C=C), 1561 and 1540 (arom.). ^{1}H-NMR ($CDCl_3$): 2.06 (*s*, 3 H); 2.16 (*s*, 3 H); 2.78 (br., 1 H); 2.96 (br., 1 H); 5.43 (br., 1 H); 5.60 (*d*, *J* = 12.4, 1 H); 5.90 (*d*, *J* = 7.1, 1 H); 6.35 (*d*, *J* = 7.1, 1 H); 7.04 (*d*, *J* = 7.6, 1 H); 7.09–7.16 (*m*, 2 H); 7.21 (*t*, *J* = 7.3, 1 H); 7.41 (*d*, *J* = 12.4, 1 H). ^{13}C-NMR ($CDCl_3$): 29.7; 31.1; 47.6; 51.3; 92.8; 109.8; 125.0; 126.1; 127.4; 128.3; 129.7; 131.0; 146.4; 196.2; 205.5. EI-MS: 255 (9, M^+), 238 (27), 223 (29), 208 (35), 198 (100), 180 (9), 145 (21), 129 (54), 115 (18), 77 (32), 43 (90). Anal. calc. for $C_{16}H_{17}NO_2$ (255.31): C 75.27, H 6.71, N 5.49; found: C 75.33, H 6.54, N 5.28.

*(2*E*)-2-{1-Acetyl-1,10b-dihydro-2-hydroxy-2-phenylpyrrolo[2,1-*a*]isoquinolin-3(2*H*)-ylidene}-1-phenylethanone* (**7**): Yield 320 mg (75%). Pale yellow crystals. M.p. 190° (dec.). IR (KBr): 3490 (OH), 1726 (COMe), 1702 (COPh), 1580 (C=C), 1555 and 1498 (arom.), 1165 (C–OH). ^{1}H-NMR ($CDCl_3$): 2.35 (*s*, 3 H); 4.14 (*d*, *J* = 9.4, 1 H); 5.73 (*d*, *J* = 9.4, 1 H); 6.05 (*s*, 1 H); 6.37 (*d*, *J* = 7.5, 1 H); 6.69 (*d*, *J* = 7.5, 1 H); 6.89 (*d*, *J* = 7.5, 1 H); 7.17 (*d*, *J* = 7.2, 1 H); 7.21–7.28 (*m*, 3 H); 7.32 (*t*, *J* = 7.7, 2 H); 7.36 (*t*, *J* = 7.6, 2 H); 7.47 (*t*, *J* = 7.6, 1 H); 7.52 (*d*, *J* = 6.6, 2 H); 7.79 (*d*, *J* = 7.6, 2 H); 9.01 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 31.2; 59.9; 61.3; 67.8; 84.6; 89.1; 116.5; 122.1; 123.8; 124.8; 125.7; 127.7; 128.1; 128.2; 128.3; 128.7; 130.6; 131.6; 131.9; 138.9; 140.2; 166.8; 188.9; 204.9. EI-MS: 421 (2, M^+), 360 (16), 277 (23), 236 (10), 167 (17), 149 (33), 129 (51), 105 (100), 94 (24), 77 (72), 57 (32), 43 (44). Anal. calc. for $C_{28}H_{23}NO_3$ (421.49): C 79.79, H 5.50, N 3.32; found: C 79.61, H 5.68, N 3.41.

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