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Regioselective Amidation of Allylic α-Hydroxyphosphonates with Nitriles: A Convenient Route to (3-Acetylamino-1-alkenyl)-phosphonates

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Summary. Reaction of dialkyl (1-hydroxy-2-alkenyl)- and (1-hydroxy-2-cycloalkenyl)phosphonates (**1**) with acetonitrile in the presence of trifluoromethane sulfonic acid (*Tf*OH) affords regiospecifically and with high (*E*)-stereoselectivity the 1,3-transposed acetamides **3** in modest to good yields.

Keywords. Dialkyl (1-hydroxy-2-alkenyl)phosphonates; Dialkyl (3-acetylamino-1-alkenyl)phosphonates; 1,3-Transposition; *Ritter* reaction.

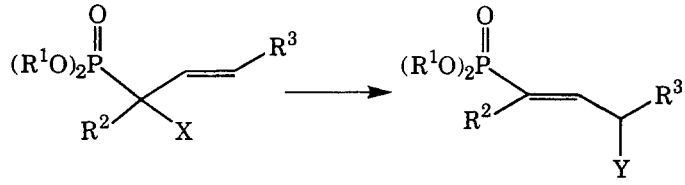
Regioselektive Amidierung von allylischen α-Hydroxyphosphonaten mit Nitrilen: Ein einfacher Weg zu (3-Acetylamino-1-alkenyl)phosphonaten

Zusammenfassung. (1-Hydroxy-2-alkenyl)- und (1-Hydroxy-2-cycloalkenyl)phosphonate (**1**) reagieren mit Acetonitril in Gegenwart von Trifluormethansulfonsäure (*Tf*OH) in mäßigen bis guten Ausbeuten regiospezifisch und mit hoher (*E*)-Stereoselektivität zu den 1,3-umgelagerten Acetamiden **3**.

Introduction

The synthetic utility of allylic α-hydroxyphosphonates and their ester derivatives for the 1,3-interchange of functionalities has been demonstrated in a series of recent publications [1–11] (Scheme 1). 3-Amino- and (3-hydroxyamino-alkyl)phosphonic acids have attracted considerable interest because of their unique biological properties which include herbicidal, antibiotic, and very promising neuroactivities [12]. Starting from allylic α-hydroxyphosphonates, the regioselective introduction of nitrogen to carbon-3 has previously been effected by direct substitution with hydrazoic acid under *Mitsunobu* conditions [8], as well as by *Overman* rearrangement of the corresponding trichloroacetimidic esters [9], and by the palladium(0) catalyzed amination [10] and N-hydroxyamination [11] of their acetates [10] and carbonates [11], respectively.

These pathways are all restricted to the use of secondary α-hydroxyphosphonates with $R^2 = H$ (aldehyde derivatives), tertiary 3-amination products being



X	Y	Ref.
OH	OAc	[1]
OH	=O	[2]
OH	F	[3]
OH	Cl	[4]
OSAr	S(O)Ar	[5]
OAc	$P(O)R^1R^2$	[6]
OAc	CHE^1E^2	[7]
O-C(OEt)=CHR	$CH(R)CO_2Et$	[5]
OH	N_3	[8]
$OC(=NH)CCl_3$	$NHCOCCl_3$	[9]
OAc	NR^1R^2	[10]
OCO_2Me	NR^1OR^2	[11]
OH	$NHCOCH_3$	

Scheme 1

available only *via* prior preparation of the corresponding α-substituted allylic 3-hydroxyphosphonates [8, 11].

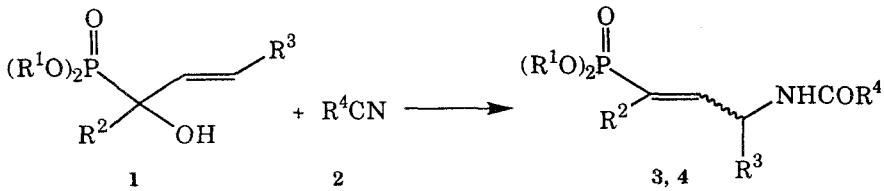
In this paper we present a convenient one step 1,3-conversion of allylic α-hydroxyphosphonates *via* a modified *Ritter* reaction, yielding precisely those rearranged 3-acetylamino-substituted phosphonates that are less accessible by previous methods.

Results and Discussion

Recently, we have shown that tertiary allylic α-hydroxyphosphonates (**1**, $R^2 \neq H$) are rearranged on acid catalyzed acetylation to yield regioselectively the thermodynamically more stable 1,3-transposed 3-acetyloxy derivatives [1]. During our studies on the synthetic potential of compounds **1** for the regioselective interchange of O- and N-functionalities [8, 9, 11], we observed that under the conditions of the *Ritter* reaction [13] appropriately substituted α-hydroxyphosphonates undergo smooth conversion to the corresponding rearranged acetamides **3** [14] (Scheme 2).

The starting α-hydroxyphosphonates **1** are easily available by regioselective 1,2-addition of dialkyl phosphites to α,β-unsaturated aldehydes, catalyzed by potassium fluoride [15] (compounds **1** with $R^2 = H$), or to α-enones, catalyzed by sodium methoxide at −35 °C [1] (compounds **1** with $R^2 \neq H$).

Acetamidation with acetonitrile in the presence of trifluoromethane sulfonic acid has been investigated with a variety of substituted allylic α-hydroxyphosphonates (Table 1). The crotonaldehyde derived phosphonates **1a** and **1b** yielded only low amounts of the corresponding allylically rearranged acetamides **3**, even under forced reaction conditions (entries 1 and 2). More satisfactory results were obtained using the 3-phenyl-substituted analogues **1c** and **1d** which provided the 3-acetylamino derivatives (*E*)-**3c** and (*E*)-**3d** stereoselectively with yields of 56 and 68%, respectively



1, 3, 4	a	b	c	d	e	f	g	h
R^1	Me	Et	Me	Et	Me	Me	Me	Me
R^2	H	H	H	H	Me	$(CH_2)_2$	$(CH_2)_3$	$(CH_2)_4$
R^3	Me	Me	Ph	Ph	Ph			

	2a,3	2b,4
R^4	Me	Ph

Scheme 2

(entries 3, 4). The tertiary α-hydroxyphosphonate **1e**, available from benzylidene acetone and dimethyl phosphite [1], afforded a 3:1 ratio of the transposed acetamides (*E*)-**3e** and (*Z*)-**3e** with about 70% total yield (entry 5). Application of the method to the cycloalkenone derivatives **1f–h** (entries 6–8) gave good yields of the corresponding (3-acetylamino-1-cycloalkenyl)phosphonates **3f–h**. Finally, the behaviour of the hydroxyphosphonates **1d** and **1g** was investigated in a similar reaction with benzonitrile (CH_2Cl_2, PhCN (5 equiv.), *Tf*OH (1 equiv.), 0 °C -r.t.), but yields of rearranged benzamides **4** were low in both cases (Table 1, entries 9 and 10).

Starting from secondary allylic α-hydroxyphosphonates, (3-acetylamino-1-alkenyl)phosphonates **3** have previously been synthesized by us *via* the corresponding 3-azido compounds [8]. This pathway precludes the use of 3-phenyl-substituted structures which form mixtures of 1,3-regioisomeric azides under thermodynamic control and, moreover, give vinylazides upon base catalysis [8]. α-Substituted derivatives (as, for example, the cyclohexenyl derivative **3g**) have been prepared by a modification of this pathway *via* prior conversion of **1** to the corresponding regioisomeric 3-hydroxyphosphonate. The sigmatropic rearrangement of trichloroacetimidic esters of compounds **1** to the corresponding 3-trichloroacetamides is also not applicable to tertiary allylic hydroxyphosphonates [9]. In conclusion, the new and simple acetamidation procedure, which works well with cyclic and with 3-aryl-substituted hydroxyphosphonates **1**, provides a ready access to (3-aminoalkyl)phosphonic acids less accessible *via* previous methods.

Experimental

Melting points: Kofler apparatus, uncorrected; ^1H and *J*-modulated ^{13}C NMR spectra: Bruker AM 400 WB; mass spectra: Finnigan MAT spectrometer 311A connected with a Vector 2/Teknivent data system; TLC: Merck silica gel 60 F_{254} plates (visualizaion of alkenyl derivatives by $KMnO_4$/acetone spray, visualization of satd. compounds by iodine vapour); flash chromatography: glass columns

Table 1. Regioselective 3-amidation of allylic α-hydroxyphosphonates **1**

Entry	1	2	Time (h)[a]	Product(s) (yield, %)	R_f[b]	M.p.[c] (°C)	Molecular Formula	Elemental analysis calc. (found) %C	%H	%N	MS (70 eV) *m/z* (%)
1	**1a**	**2a**	24	(*E*)-**3a** (30)	0.38	oil[d]	$C_8H_{16}NO_4P$[d] (221.2)				
2	**1b**	**2a**	24	(*E*)-**3b** (28)	0.25	oil	$C_{10}H_{20}NO_4P$ (249.3)	48.17 (47.89	8.10 7.95	5.62 5.48)	249 (1.5), 206 (33) 112 (33), 70 (100)
3	**1c**	**2a**	26	(*E*)-**3c** (67)	0.16	86–88	$C_{13}H_{18}NO_4P$ (283.3)	55.11 (55.16	6.42 6.07	4.95 4.77)	283 (7), 241 (53), 240 (89), 130 (100)
4	**1d**	**2a**	23	(*E*)-**3d** (56)	0.33	81–83	$C_{15}H_{22}NO_4P$ (311.4)	57.86 (57.58	7.14 6.95	4.50 4.35)	
5	**1e**	**2a**	24	(*E*/*Z*)-**3e**[e] (68)	0.3	–[e]	$C_{14}H_{20}NO_4P$ (297.3)	56.55 (56.30	6.79 6.48	4.71 4.56)	
6	**1f**	**2a**	18	**3f** (84)	0.19	oil	$C_9H_{16}NO_4P$ (233.2)	51.50 (51.21	6.93 6.71	6.01 5.88)	233 (54), 190 (42), 176 (100)
7	**1g**	**2a**	31	**3g** (73)	0.20	oil[d]	$C_{10}H_{18}NO_4P$[d] (247.3)				
8	**1h**	**2a**	96	**3h**[f] (49)	0.22	oil	$C_{11}H_{20}NO_4P$ (261.3)	50.56 (50.23	7.73 7.60	5.36 5.24)	261 (87), 218 (55), 152 (92), 110 (100)
9	**1d**	**2b**	24	(*E*)-**4d** (32)	0.42	135–37	$C_{18}H_{20}NO_4P$ (345.4)	62.59 (62.17	5.85 5.63	4.06 3.91)	
10	**1g**	**2b**	48	**4g**[g] (36)	0.21	122	$C_{15}H_{20}NO_4P$ (309.3)	58.24 (58.01	6.53 6.30	4.53 4.28)	309 (80), 204 (53), 105 (100)

[a] Temp. 50 °C (entry 1), 80 °C (entry 2), 0 °C -r.t. (entry 3), 0–5 °C (entries 4–10); [b] Eluents: EtOAc/MeOH = 5:1 for (*E*)-**3a**, EtOAc/MeOH = 9:1 for (*E*)-**3b**, **3f**, **g**, **h**, CH_2Cl_2/acetone = 7:3 for (*E*)-**3c**, **d**, CH_2Cl_2/acetone = 3:2 for (*E*/*Z*)-**3e**, (*E*)-**4d**, CH_2Cl_2/acetone = 4:1 for **4g**; [c] Recrystallization from EtOAc/Et_2O ((*E*)-**3c**, **e**, (*Z*)-**3e**, (*E*)-**4d**, **4g**) or Et_2O ((*E*)-**3d**); [d] Identical with the acetamides prepared from the corresponding (3-azido-1-alkenyl)phosphonates [8]; [e] The separation of the isomers was effected by flash chromatography with $CHCl_3$/MeOH (30:1) as eluent ((*E*)-**3e**:52%, $R_f = 0.28$, m.p. = 104–105 °C; (*Z*)-**3e**: 16%, $R_f = 0.24$, m.p. = 146–147 °C); [f] Dimethyl (1,3-cycloheptadienyl)phosphonate (11%) and dimethyl (3-hydroxy-1-cycloheptenyl)phosphonate (20%) have been isolated as by-products; [g] Dimethyl (1,3-cyclohexadienyl)phosphonate ($R_f = 0.40$, 21%) has been obtained as by-product

packed with Merck silica gel 60 (230–400 mesh). Oily dialkyl phosphonates were dried by repeated coevaporation with toluene prior to use. The secondary α-hydroxyphosphonates **1a–1d** have been prepared by the procedure of *Texier-Boullet* [15], the α-substituted and/or cyclic analogues **1e–h** have been obtained according to Ref. [1].

*Synthesis of the Dialkyl (3-Acetylamino-1-alkenyl)phosphonates (**3**; General Procedure)*

To a cold (0–5 °C) solution of *Tf*OH (1.50 g, 10.0 mmol) in dry MeCN (20 ml), a solution of **1** (10.0 mmol) in MeCN (15 ml) is added and stirring is continued under the conditions given in Table 1. After consumption of **1** (TLC control), satd. aqueous $NaHCO_3$ solution (15 ml) is added with stirring, and the mixture is evaporated at reduced pressure. The residue is then dissolved in CH_2Cl_2 (100 ml), washed with a minimum amount of water (5–10 ml), dried (Na_2SO_4), evaporated, and the product(s) are purified by flash chromatography using the eluents given in Table 1.

Table 2. ^1H NMR data of compounds **3** and **4**[a]

Product	^1H NMR ($CDCl_3$/*TMS*), δ (ppm), *J*(Hz)
(*E*)-**3b**	1.28 (d, $J = 7.4$, 3 H, CH_3CH), 1.32 (2d, $J = 7.4$, each 3 H, $POCH_2CH_3$), 2.01 (s, 3 H, $COCH_3$), 4.02–4.12 (m_c, 4 H, $POCH_2$), 4.71 (m_c, 1 H, CHN), 5.76 (ddd, $J_{1,P} = 19.2$, $J_{1,2} = 17.2$, $J_{1,3} = 2$, 1 H, 1-H), 6.68 (ddd, $J_{2,P} = 21.7$, $J_{2,1} = 17.2$, $J_{2,3} = 4.4$, 1 H, 2-H), 6.75 (d, $J = 8.4$, 1H, NH)
(*E*)-**3c**	2.00 (s, 3 H, $COCH_3$), 3.65, 3.68 (2d, $J = 10.8$, 10.3, each 3 H, $POCH_3$), 5.78 (m_c, 1 H, CHN), 5.82 (ddd, $J_{1,P} = 19.7$, $J_{1,2} = 17.2$, $J_{1,3} = 2$, 1 H, 1-H), 6.88 (ddd, $J_{2,P} = 21.7$, $J_{2,1} = 17.2$, $J_{2,3} = 4.9$, 1 H, 2-H), 7.24–7.34 (m, 6 H, H_{arom} + NH)
(*E*)-**3d**	1.25, 1.30 (2t, $J = 7.4$, each 3 H, $POCH_2CH_3$), 2.00 (s, 3 H, $COCH_3$), 3.94–4.09 (m, 4 H, $POCH_2$), 5.78 (m_c, 1 H, CHN), 5.85 (ddd, $J_{1,P} = 19.2$, $J_{1,2} = 17.2$, $J_{1,3} = 1.5$, 1 H, 1-H), 6.85 (ddd, $J_{2,P} = 22.1$, $J_{2,1} = 17.2$, $J_{2,3} = 4.9$, 1 H, 2-H), 7.24–7.34 (m, 5 H_{arom}), 7.38 (d, $J = 8.4$, 1 H, NH)
(*Z*)-**3e**	1.93 (dd, $J_{HP} = 13.8$, $J_{HH} = 1.5$, 3 H, PC-CH_3), 1.97 (s, 3 H, $COCH_3$), 3.65, 3.71 (2d, $J = 11.3$, 10.8, each 3 H, $POCH_3$), 6.36 (m_c, 1 H, CHN), 6.39 (qdd, $J_{2,P} = 51.2$, $J_{2,3} = 9.4$, $^4J_{HH} = 1.5$, 1 H, 2-H), 6.78 (br, d, $J = 6.4$, 1 H, NH), 7.22–7.41 (m, 5 H_{arom})
(*E*)-**3e**	1.88 (dd, $J_{HP} = 14.8$, $J_{HH} = 1.5$, 3 H, PC-CH_3), 1.98 (s, 3 H, $COCH_3$), 3.61, 3.66 (2d, $J = 10.8$, each 3 H, $POCH_3$), 5.95 (dt, $J_{3,2} = J_{3,NH} = 8.9$, $^4J_{3,P} = 3.0$ Hz, 1 H, CHN), 6.75 (qdd, $J_{2,P} = 23.1$, $J_{2,3} = 8.9$, $^4J_{HH} = 1.5$, 1 H, 2-H), 7.22–7.33 (m, 5 H_{arom}), 7.39 (br, d, $J \approx 8$, 1 H, NH)
3f	1.70 (m_c, 1 H), 2.43–2.54 (m, 2 H), 2.59–2.70 (m, 1 H) (CH_2), 1.98 (s, 3 H, $COCH_3$), 5.13 (m_c, 1 H, CHN), 6.49 (qd, $J_{2,P} = 10.8$, $J_{HH} = 2.0$, 1 H, 2-H), 6.65 (br, d, $J = 7$, 1 H, NH)
3h	1.25 (m, 1 H), 1.50 (m, 1 H), 1.68–1.88 (m, 3 H), 1.98 (m, 1 H), 2.23 (m, 1 H), 2.43 (m, 1 H) (H-4 to H-7), 1.99 (s, 3 H, $COCH_3$), 3.68, 3.71 (2d, $J = 10.8$, each 3 H, $POCH_3$), 4.76 (m_c, 1 H, CHN), 6.76 (td, $J_{2,P} = 24.6$, $J_{2,H} \approx 3$, 1 H, 2-H), 6.96 (d, $J = 7.9$, 1 H, NH)
(*E*)-**4d**	3.64, 3.67 (2d, $J = 11.3$, each 3 H, $POCH_3$), 5.88 (ddd, $J_{1,P} = 19.2$, $J_{1,2} = 17.2$, $J_{1,3} = 2.0$, 1 H, 1-H), 6.00 (m_c, 1 H, CHN), 7.03 (ddd, $J_{2,P} = 22.2$, $J_{2,1} = 17.2$, $J_{2,3} = 4.9$, 1 H, 2-H), 7.26–7.50 (m, 9 H, 8 H_{arom} + NH), 7.84 (m_c, 2 H_{arom})
4g	1.60 (m_c, 1 H), 1.73 (m_c, 1 H), 1.83 (m_c, 1 H), 2.08 (m_c, 1 H), 2.18 (m_c, 2 H) [CH_2], 3.71 (2d, $J = 10.8$, each 3 H, $POCH_3$), 4.84 (m_c, 1 H, CHN), 6.63 (dd, $J_{2,P} = 22.1$, $J_{2,H} \approx 2$, 1 H, 2-H), 6.82 (d, $J = 8.4$, 1 H, NH), 7.41 (m_c, 2 H_{arom}), 7.48 (m_c, 1 H_{arom}), 7.81 (m_c, 2 H_{arom})

[a] The data of compounds (*E*)-**3a** and **3g** are reported in Ref. [8]

Table 3. ^{13}C NMR data of compounds **3** and **4**[a]

Product	^{13}C NMR ($CDCl_3$, *TMS*), δ (ppm), J(Hz)
(*E*)-**3b**	16.18 (J_{PC} = 6.4, $POCH_2\mathbf{C}H_3$), 19.62 ($\mathbf{C}H_3CH$), 22.95 ($CO\mathbf{C}H_3$), 46.60 ($^3J_{PC}$ = 22.4, CHN), 61.77 (J_{PC} = 5.8, $POCH_2$), 115.68 ($^1J_{PC}$ = 188.2, PC), 153.28 ($^2J_{PC}$ = 5.0, C-2), 169.49 (CO)
(*E*)-**3c**	22.86 ($\mathbf{C}H_3CO$), 52.37 (J_{PC} = 5.5, $POCH_3$), 54.97 ($^3J_{PC}$ = 22.6, CHN), 115.69 ($^1J_{PC}$ = 188.3, PC), 127.30, 128.83 (*o*, *m*-CH), 127.98 (*p*-CH), 138.84 ($^4J_{PC}$ = 1.0 Hz, *i*-C), 151.97 ($^2J_{PC}$ = 5.5, C-2), 169.49 (CO)
(*E*)-**3d**	16.18, 16.20 (J_{PC} = 6.2, 6.4, $POCH_2\mathbf{C}H_3$), 22.87 ($CO\mathbf{C}H_3$), 54.91 ($^3J_{PC}$ = 22.4, CHN), 61.86 (J_{PC} = 5.7, $POCH_2$), 117.16 ($^1J_{PC}$ = 187.4, PC), 127.29, 128.78 (*o*, *m*-CH), 127.89 (*p*-CH), 139.09 (*i*-C), 150.99 ($^2J_{PC}$ = 5.5, C-2), 169.48 (CO)
(*Z*)-**3e**	21.78 ($^2J_{PC}$ = 10.8, $PC\mathbf{C}H_3$), 23.22 ($CO\mathbf{C}H_3$), 51.96, 52.35 (J_{PC} = 5.6, 5.3, $POCH_3$), 52.23 ($^3J_{PC}$ = 6.6, CHN), 124.56 ($^1J_{PC}$ = 172.7, PC), 126.94, 128.64 (*o*, *m*-CH), 127.48 (*p*-CH), 140.77 (*i*-C), 146.73 ($^2J_{PC}$ = 11.6, C-2), 169.19 (CO)
(*E*)-**3e**	12.98 ($^2J_{PC}$ = 9.4, $PC\mathbf{C}H_3$), 22.96 ($CO\mathbf{C}H_3$), 50.64 ($^3J_{PC}$ = 21.9, CHN), 52.14, 52.32 (J_{PC} = 5.9, 5.4, $POCH_3$), 125.51 ($^1J_{PC}$ = 177.2, PC), 126.77, 128.75 (*o*, *m*-CH), 127.62 (*p*-CH), 140.09 (*i*-C), 145.68 ($^2J_{PC}$ = 10.3, C-2), 169.37 (CO)
3f	22.96 ($CO\mathbf{C}H_3$), 31.71 (J_{PC} = 3.0), 31.83 (s) (C-4, C-5), 52.39, 52.53 (J_{PC} = 6.0, $POCH_3$), 56.13 ($^3J_{PC}$ = 22.9, CHN), 133.86 ($^1J_{PC}$ = 189.2, PC), 147.04 ($^2J_{PC}$ = 15.1, C-2), 169.72 (CO)
3h	23.02 ($CO\mathbf{C}H_3$), 25.64 (J_{PC} = 6.7), 27.97 (J_{PC} = 9.1), 28.90 (s), 33.33 (J_{PC} = 2.6) (CH_2), 51.21 ($^3J_{PC}$ = 25.3, CHN), 52.21, 52.34 (J_{PC} = 5.9, 5.3, $POCH_3$), 130.48 ($^1J_{PC}$ = 177.7, PC), 152.90 ($^2J_{PC}$ = 10.2, C-2), 169.23 (CO)
(*E*)-**4d**	52.38 (J_{PC} = 5.7, $POCH_3$), 55.53 ($^3J_{PC}$ = 22.8, CHN), 116.34 ($^1J_{PC}$ = 188.3, PC), 127.23, 127.41, 128.39, 128.95 (*o*, *m*-CH), 128.13, 131.62 (*p*-CH), 133.86, 138.87 (*i*-C), 151.72 ($^2J_{PC}$ = 5.8, C-2), 166.65 (CO)
4g	20.11 ($^3J_{PC}$ = 11.5, C-5), 23.97 ($^2J_{PC}$ = 8.5, C-6), 28.41 ($^4J_{PC}$ = 1.4, C-4), 45.63 ($^3J_{PC}$ = 19.8, CHN), 52.25, 52.35 (J_{PC} = 5.6, 6.1, $POCH_3$), 126.94, 128.25 (*o*, *m*-CH), 129.68 ($^1J_{PC}$ = 180.0, PC), 131.30 (*p*-CH), 134.11 (*i*-C), 142.67 ($^2J_{PC}$ = 8.6, C-2), 166.76 (CO)

[a] The data of compounds (*E*)-**3a** and **3g** are reported in Ref. [8]

*Synthesis of the Dialkyl (3-Benzoylamino-1-alkenyl)phosphonates (**4**; General Procedure)*

To a cold (0–5 °C) solution of *Tf*OH (150 mg, 1.0 mmol) and benzonitrile (515 mg, 5.0 mmol) in dry dichloromethane (3 ml), a solution of **1** (1.0 mmol) in CH_2Cl_2 (2 ml) is added dropwise. Stirring is continued for the time given in Table 1. Then, the solution is diluted with CH_2Cl_2 (10 ml), washed with satd. aqueous $NaHCO_3$ solution (1.5 ml) and water (2 ml), dried (Na_2SO_4), and evaporated. The products are isolated by flash chromatography on silica gel using the eluents given in Table 1.

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