Palladium Catalysis in Allylic Rearrangements of Propenyl 2-Pyrimidinyl Carbonates to 1-Propenyl-2(1*H*)-pyrimidinones

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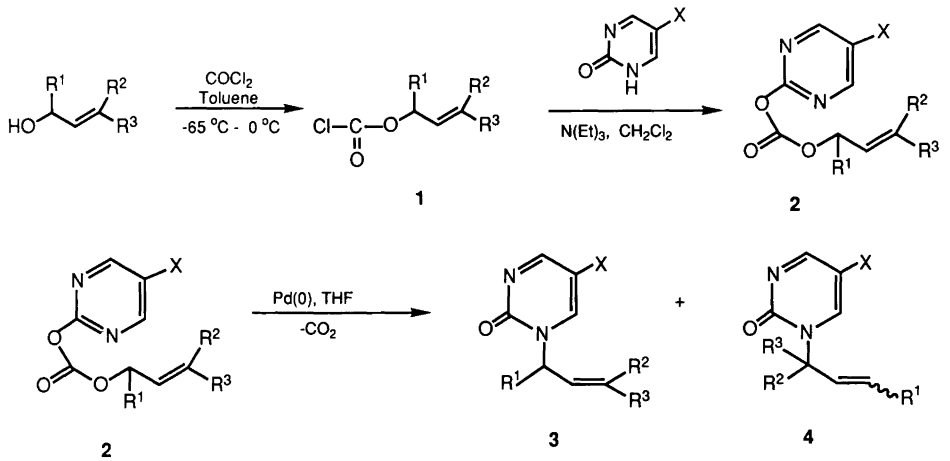
Propenyl 2-pyrimidinyl carbonates have been prepared and found to undergo allylic rearrangement with Pd(0) catalysis under mild conditions to form *N*-propenyl-2-pyrimidinones. Carbon dioxide is expelled in the reaction. The nature of the phosphine ligands and the substitution pattern in the allylic system affect the isomer product ratios.

Palladium-catalyzed rearrangements in allylic systems are useful reactions in organic syntheses.[1] We have reported that propenyloxypyrimidines can be rearranged to *N*-propenyl-2-pyrimidinones by Pd(0) or Pd(II) catalysis.[2] [3,3] Regioselectivity is observed with Pd(II) catalysis, whereas Pd(0) catalysis generally gives both [1,3] and [3,3] rearrangements products. Since the Pd(0) catalysis probably involves a π-allylpalladium complex, the substitution pattern in the allylic system is important for the regiochemistry of the rearrangement, and for the rate of the rearrangement. If the rate of the rearrangement is determined by the rate of formation of the π-allylic system, allylic pyrimidinyl carbonates would be expected to enhance the rate relative to allylic pyrimidinyl ethers, by analogy with the higher reactivity of allylic carbonates over corresponding acetates in allylation reactions of carbon nucleophiles.[3] Furthermore, alkyl allyl ethers are formed by palladium-catalyzed reactions of alkyl allyl carbonates,[4] which would suggest that a Pd-catalyzed reaction of allylic carbonates of the phenolic 2-hydroxypyrimidine would lead to allylation of either the O or the N in the pyrimidine. We herein report rearrangement studies on propenyl 2-pyrimidinyl carbonates **2** (Scheme 1). The propenyl 2-pyrimidinyl carbonates **2** were prepared from 2-pyrimidinones and propenyl chloroformates, which were available from the allylic alcohol and phosgene. The carbonyl chloride is a hard electrophile,[5] and as such reacts selectively on the oxygen to form the carbonate **2**.

The allylic rearrangement of propenyl 2-pyrimidinyl carbonates **2** to *N*-propenyl-2-pyrimidinones with expulsion of carbon dioxide proceeded well with Pd(0) catalysis



	R^1	R^2	R^3	X
a	H	H	H	Cl
b	Pent	H	H	Cl
c	Pent	H	H	Br
d	H	H	Me	Cl

	R^1	R^2	R^3	X
e	H	H	Me	Br
f	H	H	Ph	Cl
g	H	H	Ph	Br
h	H	Me	Me	Br

Scheme 1.

Table 1. Pd(0)-catalyzed rearrangement of propenyl 2-pyrimidinyl carbonates **2**.

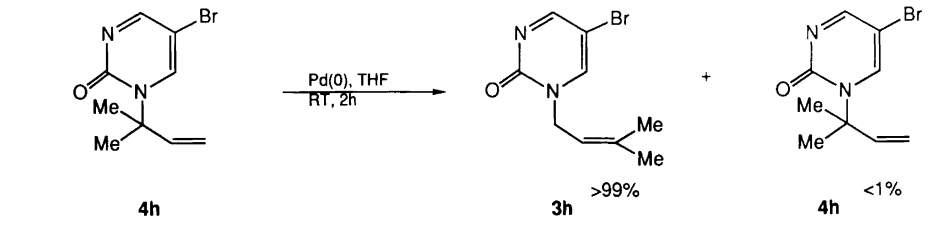
Entry	X	R^1	R^2	R^3	Pd(0)	Yield (%)	Product isomer ratio **3**:**4**	*t*/h (*T*/°C)
1	Cl	H	H	H	*a*	74	—	2 (20)
2	Cl	Pent	H	H	*a*	91	0:100 (2% *cis*)	2 (20)
3	Cl	Pent	H	H	*b*	93	37:63 (7% *cis*)	2 (20)
4	Cl	Pent	H	H	*c*	0		24 (80)
5	Cl	Pent	H	H	*d*	58	46:54 (9% *cis*)	21 (20) then 3.5 (80)
6	Br	Pent	H	H	*a*	93	2:98 (10% *cis*)	2 (20)
7	Br	Pent	H	H	*b*	78	35:65 (7% *cis*)	2 (20)
8	Br	Pent	H	H	*e*	83	23:77 (8% *cis*)	25.5 (20)
9	Cl	H	H	Me	*a*	89	84 (17% *cis*):16	2 (20)
10	Cl	H	H	Me	*b*	94	18 (2% *cis*):82	2 (20)
11	Br	H	H	Me	*a*	68	87 (14% *cis*):13	2 (20)
12	Br	H	H	Me	*f*	73	34 (8% *cis*):66	22 (20) then 3.5 (80)
13	Br	H	H	Me	*d*	51	16 (2% *cis*):84	18 (20) then 8.5 (80)
14	Cl	H	H	Ph	*a*	86	100:0	2 (20)
15	Cl	H	H	Ph	*d*	16	100:0	23 (20) then 6.5 (80)
16	Br	H	H	Ph	*b*	87	100:0	2 (20)
17	Br	H	Me	Me	*a*	83	99:1	1 (20)
18	Br	H	Me	Me	*b*	73	99:1	2 (20)
19	Br	H	Me	Me	*d*	19	99:1	21 (20) then 6 (80)

[a] $Pd[(OiPr)_3P]_4$. [b] $Pd(Ph_3P)_4$. [c] $(dba)_3Pd_2CHCl_3$. [d] $Pd[(o\text{-tolyl})_3P]_4$. [e] $Pd[(MeO)_3P]_4$. [f] $Pd[(nBu)_3P]_4$.

at ambient temperature. In the absence of the catalyst there was no rearrangement. For the corresponding rearrangement from propenyloxypyrimidines, reflux in THF was necessary.[2] Pd(II) salts had no catalytic effect on the rearrangements of the allylic 2-pyrimidinyl carbonates, presumably because such a rearrangement would imply an unfavorable eight-membered cyclic intermediate. In the previously described Pd(II)-catalyzed Claisen rearrangement of propenyloxypyrimidines to *N*-propenylpyrimidinones, however, a more favorable six-membered cyclic intermediate is postulated.[6] The product distribution from both types of rearrangement was similar, which suggests that both reactions go through the same π-allylpalladium complex.

The pure *trans*-propenyl 2-pyrimidinyl carbonates (**2d**, **2e**, **2f** and **2g**) give a mixture of the *cis* and *trans* rearranged products (^{1}H NMR), with the latter as the main product (Table 1). This indicates that the initially formed π-allylpalladium complex is sufficiently long lived for a *syn–anti* isomerization to take place before it is attacked by the nucleophile.[6]

In studies of ligand effects on the regiochemistry in



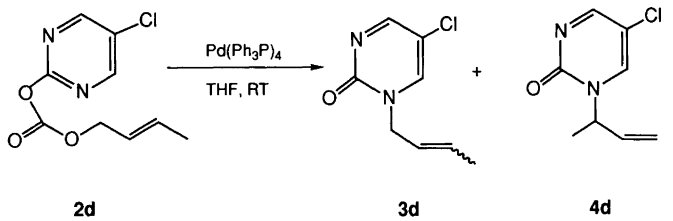


Table 2: Product composition (**3**/**4**) at different time intervals.

Time (h)	Ratio **3d**/**4d**
0.25	15/85[a]
0.5	15/85[a]
1	17/83[a]
2	18/82
3	24/76
4	31/69
6	42/58
27	55/45
40	78/22
73	100/0

[a] Starting material left

Scheme 2.

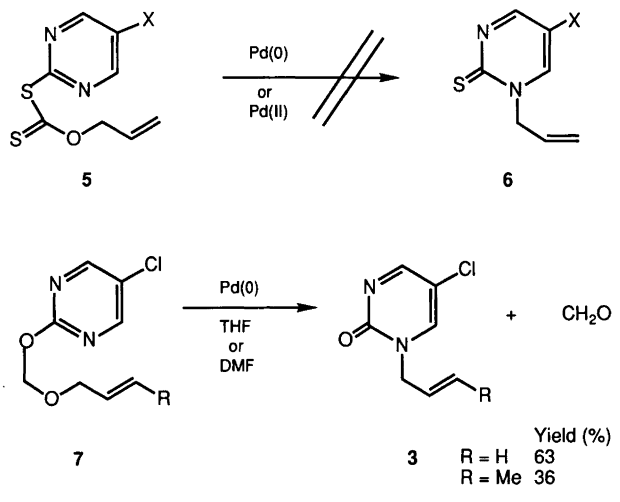
π-allylpalladium complexes it has been found that π-acceptor ligands[7] and bulky phosphine ligands both direct the nucleophile to the more substituted allylic carbon.[8] In the pyrimidinone series, relatively bulky phosphine ligands such as tri(*o*-tolyl)phosphine also direct the nucleophile to the more substituted end of the π-allylpalladium system, more so than the less bulky ligand triphenylphosphine (Table 1, entries 5 and 3, respectively). In some cases no steric effects from ligands were observed (Table 1, entries 14–19). This may be due to rapid isomerization of the initially formed products, or that the steric effects from the substituent(s) in the allylic system are more important than steric effects from the ligands. In the case of compound **4h** rapid isomerization to the thermodynamically more stable product **3h** has been shown (Scheme 2).

With trimethyl phosphite steric and electronic factors are operating in opposite direction. The phosphite is a good π-acceptor ligand[9] and should direct the nucleophile to the most substituted end of a π-allyl system, but the steric bulk of the ligand is low (cone angle 107°),[10] which should favor substitution at the less substituted end of the π-allyl system. In the pyrimidine rearrangements substitution on both ends of the π-allylpalladium complex is observed (Table 1, entry 8). With triisopropyl phosphite as ligand steric factors seem to be more important, and hence the nucleophile is directed towards the less substituted end (Table 1, entries 2, 6, 9, 11).

The primary *N*-allylic products may isomerize. When the isomerization is rapid the product isomer ratio in Table 1 may not be that of the primary products. In the case of **3d** and **3e** isomerization is slow relative to product formation (Scheme 2, Table 2 and Ref. 2). The product distributions seen in Table 1, entries 2–13, are therefore ascribed to steric and electronic effects from the ligands.

In a search for rearrangeable substrates different from the carbonates (**2**), the simple allyl dithio analog **5** was prepared by reaction between potassium *O*-allyl dithiocarbonate and 5-bromo-2-chloropyrimidine. The dithiocarbonate **5**, however, failed to undergo rearrangement under the standard conditions with Pd(II) or Pd(0) catalysis. The search also led to the acetals **7**.[11]

The acetal **7** from allyl alcohol gave a moderate yield (63%) of rearranged product with concurrent loss of formaldehyde. With a methyl-substituted allyl group, however, no rearrangement took place under the standard conditions in THF, but after heating in DMF at 80°C the rearranged product **3** was isolated in 36% yield.



Scheme 3.

Experimental

The ^1H NMR spectra were recorded at 300 MHz with a Varian XL-300 (manual) or at 200 MHz with a Varian Gemini 200 instrument. The ^{13}C NMR spectra were recorded at 75 or 50 MHz on the same instruments. The mass spectra under electron impact conditions were recorded at 70 eV ionizing potential and ammonia, isobutane or methane was used for chemical ionization (CI); the spectra are presented as *m/z* (% rel. int.).

Compounds available by literature methods: 5-chloro-1-(2-propenyl)-2-(1*H*)-pyrimidinone (**3a**);[2] (*E*)-5-bromo-1-(2-butenyl)-2-(1*H*)-pyrimidinone (**3e**);[2] (*E*)-5-bromo-1-(3-phenyl-2-propenyl)-2-(1*H*)-pyrimidinone (**3g**);[2] 5-bromo-1-(3-methyl-2-butenyl)-2-(1*H*)-pyrimidinone (**3h**);[2] (*E*)-5-chloro-1-(2-octenyl)-2-(1*H*)-pyrimidinone (**4b**);[2] (*E*)-5-bromo-1-(2-octenyl)-2-(1*H*)-pyrimidinone (**4c**);[2] (*E*)-5-bromo-1-(1-methyl-2-propenyl)-2-(1*H*)-pyrimidinone (**4e**).[2]

Preparation of allylic 5-halo-2-pyrimidinyl carbonates (**2**). The allylic alchohol (78.9 mmol) was added dropwise to a solution of phosgene in toluene (52 ml, 1.93 M) at −65°C. The mixture was stirred for 30 min at −65°C and for 1 h at 0°C. Most of the solvent was removed by reduced pressure distillation (25 mmHg) and the residual solution of the chloroformate was used in the subsequent reaction with the 5-halo-2(1*H*)-pyrimidinone.

Triethylamine (0.78 g, 7.72 mmol) in dry dichloromethane (10 ml) was added to a suspension of the 5-halo-2(1*H*)-pyrimidinone (3.86 mmol) in dry dichloromethane (10 ml). The mixture was stirred under N_2 at ambient temperature for 15 min before the solution was cooled to 0°C and a solution of the chloroformate (7.72 mmol) in dry dichloromethane (5 ml) added dropwise. The mixture was stirred for 15 min at 0°C, for 1 h at ambient temperature and then washed with water (2×). The dried ($MgSO_4$) solution was evaporated and the product was purified by flash chromathography on silica gel using hexane–EtOAc 6:1 (v/v) and m.p.s were recorded.

5-Chloro-2-pyrimidinyl 2-propenyl carbonate (**2a**). Oily material, yield 41%. ^1H NMR ($CDCl_3$): δ 4.78–4.80 (OCH_2, m), 5.32–5.48 (=CH_2, m), 5.93–6.06 (CH=, m), 8.67 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 69.7 (OCH_2), 119.7 (CH_2=), 129.2 (C-5), 130.4 (CH=), 151.2 (CO), 158.1

(C-4,6) 158.4 (C-2). MS(CI-NH_3): 217/215 (10/30, $M+1$), 173 (32), 171 (100), 131 (7), 58 (13), 41 (6), 35 (35), 30 (18).

5-Chloro-2-pyrimidinyl 3-oct-1-enyl carbonate (**2b**). Oily material, yield 58%. Anal. $C_{13}H_{17}ClN_2O_3$: C, H. ^{1}H NMR ($CDCl_3$): δ 0.8–0.9/1.2–1.4/1.55–1.75 (C_5H_{11}), 5.1–5.2 (OCH, m), 5.2–5.3/5.3–5.4 (=CH_2, 2×m), 5.75–5.90 (CH=, m), 8.65 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 13.8/22.4/24.4/31.4/33.9 (C_5H_{11}), 81.3 (CHO), 118.3 (CH_2), 129.1 (C-5), 135.1 (CH=), 150.9 (CO), 158.2 (C-2), 158.9 (C-4,6). MS(CI-NH_3): 287/285 (1/3, $M+1$), 243 (32), 242 (15), 241 (100), 169 (12), 133 (11), 131 (40), 69 (12).

5-Bromo-2-pyrimidinyl 3-oct-1-enyl carbonate (**2c**). Oily material, yield 77%. Anal. $C_{13}H_{17}BrN_2O_3$: C, H. ^{1}H NMR ($CDCl_3$): δ 0.8–0.9/1.2–1.4/1.55–1.80 (C_5H_{11}), 5.1–5.4 (OCH and =CH_2, m), 5.7–5.9 (CH=, m), 8.70 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 14.1/22.6/24.7/31.6/34.2 (C_5H_{11}), 81.7 (CHO), 117.9 (C-5), 118.9 (=CH_2), 135.5 (CH=), 151.5 (CO), 159.8 (C-2), 161.1 (C-4,6). MS(CI-NH_3): 331/329 (91/93, $M+1$), 287 (90), 285 (93), 215 (17), 177 (95), 175 (100), 111 (29), 69 (50).

(E)-*2-Butenyl 5-chloro-2-pyrimidinyl carbonate* (**2d**). Yield: 78%, m.p. 72°C. Anal. $C_9H_9ClN_2O_3$: C, H. ^{1}H NMR ($CDCl_3$): δ 1.76 (CH_3, dd, J 6.6, 1 Hz), 4.72 (CH_2, dd, J 6.2, 1 Hz), 5.6–5.95 (CH=CH, m), 8.67 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 17.2 (CH_3), 70.1 (CH_2O), 123.8 (CH=), 129.3 (C-5), 133.5 (CH=), 151.5 (CO), 158.3 (C-4,6), 158.9 (C-2). MS(CI-NH_3): 231/229 (2/7, $M+1$), 187 (18), 185 (58), 133 (25), 131 (76), 74 (9), 55 (100), 54 (14).

(E)-*5-Bromo-2-pyrimidinyl 2-butenyl carbonate* (**2e**). Yield 68%, m.p. 71°C. Anal. $C_9H_9BrN_2O_3$: C, H. ^{1}H NMR ($CDCl_3$): δ 1.69 (CH_3, d, J 6.4 Hz), 4.65 (OCH_2, d, J 6.4 Hz), 5.5–6.0 (CH=CH, m), 8.71 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 18.0 (CH_3), 70.5 (CH_2O), 118.0 (C-5), 124.0 (CH=), 134.2 (CH=), 152.0 (CO), 159.7 (C-2), 161.1 (C-4,6). MS(CI-NH_3): 275/273 (1/1, $M+1$), 231 (93), 230 (19), 229 (100), 177 (52), 175 (55), 149 (11), 55 (80).

(E)-*5-Chloro-2-pyrimidinyl 3-phenyl-2-propenyl carbonate* (**2f**). Yield 40%, m.p. 105°C. Anal. $C_{14}H_{11}ClN_2O_3$: C, H. ^{1}H NMR ($CDCl_3$): δ 4.95 (CH_2O, dd, J 6.6, 1 Hz), 6.3–6.4 (CH=, m), 6.76 (CH-Ph, d, J 16 Hz), 7.3–7.45 (Ph, m), 8.66 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 70.0 (CH_2O), 121.4 (CH=), 126.9/128.6/128.7/136.0 (Ph), 129.4 (C-5), 136.1 (CH=), 151.5 (CO), 158.3 (C-4,6), 158.9 (C-2). MS(CI-CH_4): 293/291 (1/3, $M+1$), 249 (6), 247 (17), 246 (3), 131 (3), 118 (11), 117 (100), 29 (13).

(E)-*5-Bromo-2-pyrimidinyl 3-phenyl-2-propenyl carbonate* (**2g**). Yield 49%, m.p. 107°C. Anal. $C_{14}H_{11}BrN_2O_3$: C, H. ^{1}H NMR ($CDCl_3$): δ 4.95 (CH_2O, dd, J 6.6, 1 Hz), 6.3–6.4 (CH=, m), 6.76 (CH-Ph, d, J 16 Hz), 7.25–7.45 (Ph, m), 8.76 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 70.0 (CH_2O), 117.6 (C-5), 121.4 (CH=), 126.9/128.6/128.7/136.0 (Ph), 136.1 (CH=), 151.5 (CO), 159.1 (C-2), 160.6 (C-4,6). MS: 336/334 (3/3, M), 292 (1), 290 (1), 177 (14), 175 (15), 117 (100), 115 (32), 91 (14).

5-Bromo-2-pyrimidinyl 3-methyl-2-butenyl carbonate (**2h**). Yield 54%, m.p. 90°C. Anal. $C_{10}H_{11}BrN_2O_3$: C, H. ^{1}H NMR ($CDCl_3$): δ 1.76/1.80 (2×CH_3, 2×s), 4.80 (OCH_2, d, J 7.5 Hz), 5.4–5.5 (CH=, m), 8.76 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 18.1 (CH_3), 25.8 (CH_3), 66.4 (OCH_2), 117.2 (CH=), 117.5 (C-5), 141.4 (=C), 151.4 (CO), 159.2 (C-2), 160.5 (C-4,6). MS(CI-CH_4): 288/286 (1/1, $M+1$), 243 (1), 177 (22), 175 (23), 69 (100), 68 (9), 59 (6), 41 (20).

Pd(0)-catalyzed rearrangement of substituted allylic 5-halo-2-pyrimidinyl carbonates (**2**). The carbonate **2** (0.5 mmol) and Pd(0) (7%) were dissolved in dry THF (6 ml) under N_2. Reaction conditions are given in Table 1. The solution was evaporated, and the residual material was subjected to flash chromatography on silica gel column which was eluted with EtOAc.

(E)-*5-Chloro-1-(2-butenyl)-2-(1H)-pyrimidinone* (**3d**). Yield 52%, m.p. 125°C. ^{1}H NMR ($CDCl_3$): δ 1.78 (Me, d, J 6.5 Hz), 4.46 (CH_2N, d, J 6.6 Hz), 5.55–5.9 (CH=CH, m), 7.71 (H-6, d, J 3.6 Hz), 8.52 (H-4, d, J 3.6 Hz). ^{13}C NMR ($CDCl_3$): δ 17.8 (CH_3), 52.6 (CH_2N), 111.6 (C-5), 123.1 (CH=), 134.0 (CH=), 144.0 (C-6), 154.5 (C-2), 164.6 (C-4). MS: 186/184 (9/28, M), 183 (18), 131 (10), 130 (20), 114 (9), 102 (13), 55 (100), 54 (61).

(E)-*5-Chloro-1-(3-phenyl-2-propenyl)-2-(1H)-pyrimidinone* (**3f**). Yield: 86%, m.p. 185°C Anal. $C_{13}H_{11}ClN_2O$: C, H. ^{1}H NMR ($CDCl_3$): δ 4.68 (NCH_2, d, J 7 Hz), 6.25–6.35 (CH=, m), 6.72 (CH=, d, J 16 Hz), 7.3–7.4 (Ph, m), 7.78 (H-6, d, J 3.3 Hz), 8.53 (H-4, d, J 3.3 Hz). ^{13}C NMR ($CDCl_3$): δ 53.1 (CH_2N), 111.2 (C-5), 121.2 (CH=), 126.8/128.7/128.8/135.5 (Ph), 136.9 (CH=), 144.3 (C-6), 154.5 (C-2), 165.0 (C-4). MS: 248/246 (7/21, M), 131 (25), 118 (10), 117 (100), 116 (93), 115 (57), 114 (13), 91 (24).

S-*5-Chloro-2-pyrimidinyl* O-*2-propenyl dithiocarbonate* (**5**). 5-Bromo-2-chloropyrimidine (1 g, 5.19 mmol) and potassium *O*-allyl dithiocarbonate[12] (0.892 g, 5.19 mmol) were dissolved in dry DMF (50 ml). The solution was stirred under N_2 for 24 h at 80°C. DMF was removed at reduced pressure and chloroform was added. The solution was washed with water (×4), dried ($MgSO_4$) and evaporated. The crude product was purified by flash chromatography on silica gel eluting with hexane–EtOAc 10:1 (v/v); yield 25%. ^{1}H NMR ($CDCl_3$): δ 3.79 (CH_2O, d, J 7 Hz),

5.14 (one H in $CH_2=$, d, J 10 Hz), 5.32 (one H in $CH_2=$, dd, J 17, 1.4 Hz), 5.90–6.03 (CH=, m), 8.56 (H-4,6, s). ^{13}C NMR ($CDCl_3$): δ 33.9 (CH_2O), 114.9 (C-5), 118.0 ($CH_2=$), 132.7 (CH=), 157.5 (C-4,6), 170.0 (C-2), 206.5 (CS).

Pd(0)-catalyzed rearrangement of substituted allylic 5-chloro-2-pyrimidinyl acetals (**7**): *5-Chloro-1-(2-propenyl)-2(1H)-pyrimidinone* (**3a**).[2] A mixture of 5-chloro-2-propenyloxymethoxypyrimidine (0.173 g, 0.86 mmol), palladium(II) acetate (0.014 g, 0.06 mmol) and triisopropyl phosphite (0.089 g, 0.43 mmol) in dry THF (7 ml) was heated under reflux for 24 h under N_2. The solution was evaporated and the residual material was subjected to flash chromatography on silica gel eluting with EtOAc; yield 63%.

(E)-*5-Chloro-1-(2-butenyl)-2(1H)-pyrimidinone* (**3d**). A mixture of (*E*)-5-chloro-2-(2-butenyloxymethoxy)-pyrimidine (0.3 g, 1.39 mmol), palladium(II) acetate (0.022 g, 0.98 mmol), and triisopropyl phosphite (0.145 g, 0.70 mmol) in dry DMF (10 ml) was heated at 65°C for 24 h, and then for 16 h at 90°C under N_2. The solution was evaporated and the residual material was subjected to flash chromatography on silica gel eluting first with hexane–EtOAc 6:1 (v/v), and then EtOAc; yield 36%.

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