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Catalytic Addition–Cyclization of Alkyl Ketones with 4-Methyl-1-phenyl-4-penten-1-one.

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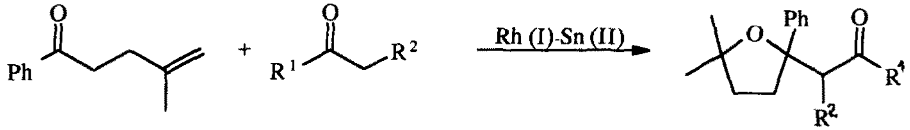
Abstract. Substituted 2-(β-oxoalkyl)tetrahydrofurans were obtained in one-step aldol-type reaction from alkyl ketones and 4-methyl-1-phenyl-4-penten-1-one in the presence of a catalytic system $[RhCl(C_2H_4)_2]_2$—$SnCl_2$

Complexes of Rh(I) catalyze dimerization and codimerization reactions of unsaturated compounds[1] Recently we have described a reaction between α,β- and γ,δ-unsaturated ketones leading to linear[2] and bicyclic[3] codimers *Aldol-type reactions involving carbonyl compounds*[4] *or their derivatives*[5] *catalyzed by rhodium* complexes are also known But neither of these aldol reactions was accompanied by cyclization

Now we have found that reaction of γ,δ-unsaturated ketone, namely, 4-methyl-1-phenyl-4-penten-1-one **1**, with alkyl ketones **2** in the presence of catalytic amounts of Rh(I) and Sn(II) results in formation of a cyclic product **3**



1 **2** **3**

The mechanism proposed is similar to one that proposed in[3] It involves the formation of $R^1COCH[Rh\text{-}H]R^2$ followed by addition of the complex at C=O group of **1** Subsequent intramolecular cyclization of the resulting intermediate leads to **3**

Much attention[6] has been drawn to the synthesis of compounds bearing 2-tetrahydrofuryl substituent and carbonyl group such as **3** owing to presence of similar structures in a wide variety of natural products[7]

Typical procedure A solution containing 4-methyl-1-phenyl-4-penten-1-one (2 77 mmol), $[RhCl(C_2H_4)_2]_2$ (0 014 mmol), and $SnCl_2$ (0 056 mmol) in degassed acetone (14 6 mmol) was heated in an argon atmosphere at 80°C in a sealed tube for 10 h Silica gel column chromatography using hexane/ether (2/1) gave 1-(5,5-dimethyl-2-phenyltetrahydro-2-furyl)propan-2-one **3a** in 43% yield The results of the reaction **1** with alkyl ketones are summarized in Table 1

Table 1 Synthesis of Substituted Tetrahydrofurans Catalyzed by Rh(I) - Sn(II) system[a]

R^1	R^2	Conversion of **1**, %	Yield **3**[b] to converted **2**, %	mol/g-at Rh
Me	H	83	43 **(3a)**	35
Ph	H	68	25 **(3b)**	17
cyclopropyl	H	64	53 **(3c)**	34
Et	Me	86	33 **(3d)**	28
$R^1+R^2= (CH_2)_3$		84	46 **(3e)**	39

[a] 4-methyl-1-phenyl-4-penten-1-one (2 27 mmol), alkyl ketone (14 6 mmol), $[RhCl(C_2H_4)_2]_2$ (0 014 mmol), $SnCl_2$ $2H_2O$ (0 056 mmol), 80 C, 10 h [b] other products are 4-methyl-1-phenyl-3-penten-1-one and *(E)*-4-methyl-1-phenyl-2-penten-1-one

The structures of **3a-e** have been proved by ^1H, ^{13}C, and ^{17}O NMR, IR, mass spectrometry and elemental analysis

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References:

1 Dickson, R S *Homogeneous Catalysis with Compounds of Rhodium and Iridium.* D Reidel Publ Co Dordrecht, Neth **1985.**

2 Nikishin, G I , Klimova, T E , Ignatenko, A V , Kovalev, I P *Tetrahedron Lett.* **1991,** *32*, 1077-1080

3 Kovalev, I P , Ipatkin, V V , Strelenko, Y A , Ignatenko, A V , Nikishin, G I *Tetrahedron Lett.* **1992,** *33*, 1791-1794

4 (a) Sato, S , Matsuda, I , Izumi, Y *Chem Lett.* **1985,** 1875 (b) Aoyama, Y , Tanaka, Y , Yoshida, T , Toi, H , Ogoshi, H *J. Organomet. Chem.* **1987,** *329*, 251-66

5 (a) Sato, S , Matsuda, I , Izumi, Y *Tetrahedron Lett.* **1986,** *27*, 5517-20 (b) Reetz, M T , Vougioukas, A E *Tetrahedron Lett* **1987,** *28*, 793-6 (c) Mukaiyama, T , Soga, T , Takenoshita, H *Chem. Lett.* **1989,** 1273-6

6 (a) Ireland, R E , Anderson, R C , Badoud, R , Fitzsimmons, B J , McGarvey, G J , Thaisrivongs, S , Wilcox, C S *J Amer. Chem Soc* **1983,** *105*, 1988-2006 (b) Kurth, M J , Rodrigues, M J *J. Amer. Chem Soc.* **1987,** *109*, 7577-8 (c) Murata, S , Suzuki, M , Noyori, R , Tetrahedron **1988,** *44*, 4259-75

7 Westley, J W *Polyether Antibiotics. Naturally Occuring Acid Ionophores,* Vol I and II, Marcel Dekker, New York, **1982.**

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