

# Synthesis of 2-(R-benzal)-5-pyrrolidones

M. LÁCOVÁ and J. ŠRAGA

Department of Organic Chemistry, Faculty of Natural Sciences,  
Komenský University, 816 31 Bratislava

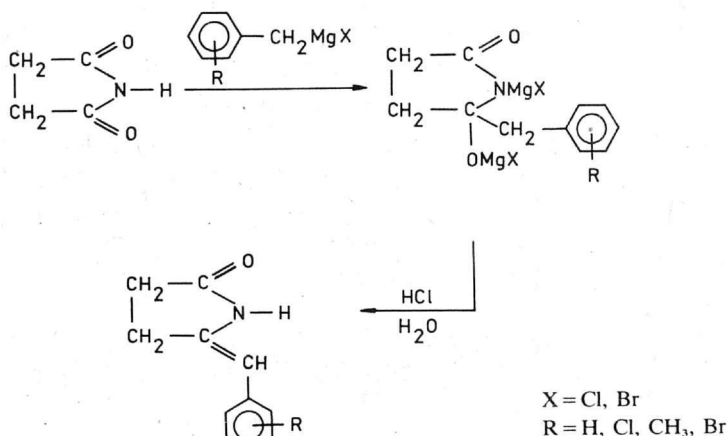
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2-(R-Benzal)-5-pyrrolidones were synthesized from succinimide, benzyl type of Grignard reagents, and subsequent decomposition of the reaction product with dilute hydrochloric acid.

Был осуществлен синтез 2-(R-бензаль)-5-пиролидонов при помощи реакции сукцинимиды с реактивами Гриньяра бензильного типа и последующим разложением продукта разбавленной хлористоводородной кислотой.

The reaction of Grignard reagents with succinimide or its salts has not been described as yet; the salts are, however, less convenient for this purpose for their high hygroscopicity. Under the conditions given later on in more detail, succinimide reacts with two equivalents of Grignard reagent to afford the corresponding pyrrolidones. A like reaction of *N*-alkyl derivatives of succinimide with



Scheme 1

Grignard reagents was already described; it has been found that an exocyclic double bond in the final product is formed providing that the conjugation in the system becomes extended [1—3].

This paper deals with preparation of 2-(R-benzal)-5-pyrrolidones having a more extended delocalized system due to an exocyclic double bond (Scheme 1). The reaction was carried out in ether, tetrahydrofuran, benzene or toluene, the latter being the most advantageous from the standpoint of optimum yields; in the other solvents the yields were by 10% lower. The obtained compounds are relatively unstable. From the decomposition products we were able to isolate the corresponding benzaldehyde.

2-(4-Nitrobenzal)-5-pyrrolidone were prepared by nitration of the appropriate benzalpyrrolidone with nitric acid in acetic anhydride, since the afore-mentioned procedure does not lead to the desired product. Nitration in sulfuric or acetic acids was unsuccessful.

### Experimental

Infrared spectra of compounds I—VII (Table 1) were recorded with a UR-20 (Zeiss, Jena) spectrophotometer in chloroform and of compound VIII in nujol.

#### *Addition of Grignard reagents to succinimide (compounds I—VII)*

An ethereal solution of the respective Grignard reagent (0.12 mole) was dropwise added to a suspension of freshly fused and powdered succinimide (5 g; 0.05 mole) in toluene (100 ml) under vigorous stirring at room temperature. Stirring was continued for 3 h with *p*-derivatives and 4 h with *m*-derivatives. The reaction mixture was then poured onto crushed ice (50 g) acidified with hydrochloric acid 50 ml and allowed to crystallize in a refrigerator. The separated crystals were filtered off and the aqueous layer extracted 3 times with ether. The residue obtained after removal of ether was washed with water, combined with the main crop, and crystallized from ethanol.

#### *2-(4-Nitrobenzal)-5-pyrrolidone (VIII)*

2-Benzal-5-pyrrolidone (1 g) was added in small portions and under stirring to a nitration mixture (acetic anhydride (60 ml)—conc. nitric acid (30 ml)) at 25—30°C and then kept at 25°C for 30 min. The reaction mixture was worked up as above, the yellow precipitate was filtered off, washed with water, and crystallized from ethanol.

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Table 1

Characteristic data of 2-(R-benzal)-5-pyrrolidones

No.	R	Formula	M	Calculated/found				Yield %	M.p. °C	$\bar{\nu}(\text{C}=\text{C})$ $\text{cm}^{-1}$	$\bar{\nu}(\text{C}=\text{O})$ $\text{cm}^{-1}$	$\bar{\nu}(\text{NH})$ $\text{cm}^{-1}$
				% C	% H	% N	% Hal.					
I	H	$\text{C}_{11}\text{H}_{11}\text{NO}$	173.1	76.30	6.35	8.03	—	52	104—106	1670	1712	3435
				76.25	6.35	8.16	—				1753	
II	4-Cl	$\text{C}_{11}\text{H}_{10}\text{ClNO}$	207.6	63.57	4.86	6.74	17.07	51	151—153	1667	1713	3430
				63.68	4.87	6.73	16.79				1733	
III	3-Cl	$\text{C}_{11}\text{H}_{10}\text{ClNO}$	207.6	63.57	4.86	6.74	17.07	56	153—156	1663	1716	3430
				63.49	4.85	6.74	17.00				1734 <sup>a</sup>	
IV	4-Br	$\text{C}_{11}\text{H}_{10}\text{BrNO}$	252.1	52.38	4.00	5.56	31.70	44	152—154	1667	1711	3430
				51.98	3.97	5.57	32.01				1733	
V	4-CH <sub>3</sub>	$\text{C}_{12}\text{H}_{13}\text{NO}$	187.2	76.96	7.00	7.47	—	50	146—148	1669	1709	3435
				77.02	7.03	7.47	—				1732	
VI	3-CH <sub>3</sub>	$\text{C}_{12}\text{H}_{13}\text{NO}$	187.2	76.96	7.00	7.47	—	50	175—176	1665	1711	3435
				76.97	6.95	7.47	—				1733	
VII	3-CH <sub>3</sub> O	$\text{C}_{12}\text{H}_{13}\text{NO}_2$	203.2	70.90	6.45	6.89	—	20	174—175	1661	1711	3440
				71.02	6.44	6.50	—				1720 <sup>a</sup>	
VIII	4-NO <sub>2</sub>	$\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}_3$	218.2	60.59	4.62	12.84	—	45	178—179	1650 <sup>a</sup>	1742	3380
				60.29	4.41	12.46	—				1770	

a) Poorly pronounced peaks.

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