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Sulfoxonium Ylide Chemistry. VI.[1] A Convenient Method for Synthesizing 1-Substituted 4-Phenyl-3-pyrrolin-2-ones

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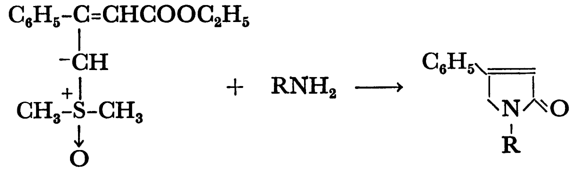
Synopsis. Synthesis of 1-substituted 4-phenyl-3-pyrrolin-2-ones (**2**) from dimethylsulfoxonium 3-ethoxycarbonyl-2-phenylallylide (**1**) and various primary amines is described.

In the continuation of our studies on sulfoxonium ylide chemistry, we have found a convenient method for synthesizing 1-substituted 4-phenyl-3-pyrrolin-2-ones from dimethylsulfoxonium 3-ethoxycarbonyl-2-phenylallylide (**1**)[2] and primary amines in fairly good yield. There have been only a few reports on the synthesis of substituted 3-pyrrolin-2-ones.[3]

Treatment of ylide **1** with aniline in tetrahydrofuran containing a small amount of 10% HCl under reflux for 3.5 h afforded 1,4-diphenyl-3-pyrrolin-2-one (**2a**) in 75% yield. The structure of **2a** was confirmed with the spectral and elemental analysis data. Its IR spectrum showed bands at 1686, 1629, and 1608 cm^{-1} due to the α,β-unsaturated lactam. In the NMR spectrum of **2a** in $CDCl_3$, methylene protons appeared at δ 4.72 (doublet, J=2 Hz), a vinyl proton at 6.48 (triplet, J=2 Hz) and aromatic protons at 7.5 ppm (multiplet).



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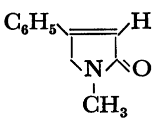
Speckamp *et al.*[3c] reported 1-methyl-4-phenyl-3-pyrrolin-2-one (**3**) and 1-methyl-3-phenyl-3-pyrrolin-2-one (**4**) in which vinyl protons appeared at δ 6.45 and 7.17 ppm, respectively. These data also could support the structure of **2**.



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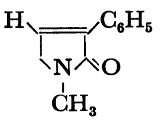

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The 1-substituted 4-phenyl-3-pyrrolin-2-ones (**2**) prepared by this method using various primary amines are listed in Table 1.

Experimental

All the melting points are uncorrected. The NMR spectra were recorded on a Varian A-60 instrument, using TMS as the internal standard. The IR spectra were obtained on a Hitachi EPI-G3 spectrophotometer.

*General Procedure for Synthesis of 1-Substituted 4-Phenyl-3-pyrrolin-2-one (**2**).* A solution of **1** (5 mmol) and an amine (15 mmol) in 10% HCl (1.6 ml) and THF (20 ml) was heated under reflux for 2—4 h. The reaction mixture was diluted with ether and washed successively with 2% HCl solution, H_2O and sat. NaCl solution, and dried over Na_2SO_4. Evaporation of the solvent *in vacuo* gave a crude product, which was purified by column chromatography on silica gel or recrystallization from ethanol.

*1-n-Butyl-4-phenyl-3-pyrrolin-2-one (**2h**).* A solution of **1** (1.33 g) and butylamine (1.09 g) in 10% HCl (1.6 ml) and THF (20 ml) was heated under reflux for 3.5 h. The reaction mixture was diluted with ether (100 ml) and successively washed with 2% HCl solution, H_2O and sat. NaCl solution, and dried over Na_2SO_4. Evaporation of the solvent gave 1.19 g of an oily residue, which was chromatographed on silica gel (10 g) to afford 380 mg of **2h** (35% yield) by eluting with benzene.

*1-Allyl-4-phenyl-3-pyrrolin-2-one (**2j**).* A solution of **1** (1.33 g) and allylamine (855 mg) in 10% HCl (1.6 ml) and THF (20 ml) was heated under reflux for 3.5 h. The reaction mixture was treated as described above to give 1.02 g of an

TABLE 1. 1-SUBSTITUTED 4-PHENYL-3-PYRROLIN-2-ONE (2)

R	NMR (δ)[a]		IR[b] (cm^{-1})	Mp (°C)	Yield[c] (%)	Obsd (%)			Calcd (%)		
	3H	4H				C	H	N	C	H	N
2a; C_6H_5	6.48	4.72	1684	168—169	85	81.45	5.50	5.98	81.68	5.57	5.95
2b; p-$CH_3C_6H_4$	6.48	4.64	1667	156—157	90	82.11	6.31	5.47	81.90	6.06	5.62
2c; p-$CH_3OC_6H_4$	6.51	4.72	1670	153—154	75	77.01	5.80	5.29	76.96	5.70	5.28
2d; p-ClC_6H_4	6.51	4.73	1672	191—192	67	71.18	4.45	5.23	71.24	4.49	5.19
2e; p-BrC_6H_4	6.50	4.47	1672	202—203	70	61.19	3.71	4.51	61.16	3.85	4.46
2f; m-$CF_3C_6H_4$	6.52	4.78	1698	152—153	73	67.51	4.01	4.59	67.32	3.99	4.62
2g; $C_6H_5CH_2$	6.46	4.22	1670	158—159	58	81.83	6.02	5.81	81.90	6.06	5.62
2h; n-C_4H_9	6.43	4.34	1680	oil	35	78.21	7.90	6.60	78.10	7.96	6.51
2i; Cyclohexyl	6.42	4.30	1664	95— 96	50	79.48	7.89	5.98	79.63	7.94	5.80
2j; CH_2=$CHCH_2$	6.45	4.32	1680	oil	39	78.51	6.49	7.00	78.36	6.58	7.03

a) In deuteriochloroform. b) In Nujol for crystals and in liquid film for oils. c) Not optimized.

oily residue, which was chromatographed on silica gel (10 g) to afford 390 mg of **2j** (39% yield).

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