NOTES

Synthesis of N-Vinylsuccinamides

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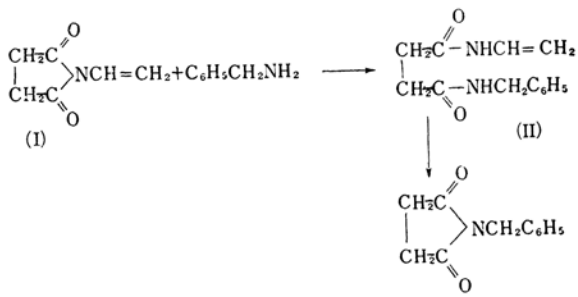
In 1959 Yanagi[1] reported a reaction of *N*-vinylsuccinimide with amines at room temperature. Under his experimental conditions, a white precipitate was given as an addition product, and he presumed the addition of amines to occur on the vinyl double bond. However, the reaction of *N*-vinylsuccinimide with amines may take place in two ways, to yield either an addition product to the double bond or *N*-vinylamide by the opening reaction of the imide ring.

Therefore, the present author has tried to prepare *N*-vinylamide derivatives through the latter reaction. When *N*-vinylsuccinimide (I) was treated with benzylamine at room temperature, a reaction occurred immediately and gave a solid flake (II), m. p. 172~173°C (decomp.). II was converted into *N*-benzylsuccinimide by the thermal decomposition. The infrared spectrum of II (Fig. 1) showed the absorption bands[2] at 3.02, 6.10 and 6.50 μ which suggested the existence of an amide radical. The existence of a double bond in the product was recognized by the Baeyer test and by bromination. Thus, II was concluded to be *N*-vinyl-*N'*-benzylsuccinamide.

Similarly, *N*-vinylsuccinimide reacted with various primary and secondary aliphatic, aromatic and heterocyclic amines and gave the respective vinyl compounds (see Table I).

N-Vinylsuccinamide copolymerized readily with such a monomer as methyl methacrylate or acrylonitrile.



Experimental

Only a few typical procedure are described in detail.

The physical properties and analyses of products are collected in Table I.

Preparation of *N*-Vinyl-*N'*-benzylsuccinamide.— A mixture of 1.1 g. of *N*-vinylsuccinimide and 1.25 g. of benzylamide was kept at room temperature for thirty minutes. The purified sample melted at 172~173°C (decomp.) and weighed 2.21 g. (95.2%).

TABLE I

N-Vinylsuccinamides $ROCCH_2CH_2CONHCH{=}CH_2$

R	M. p. (decomp.) °C	Yield %	Crystn.[a] solvent	Formula	Analyses C, % Calcd.	C, % Found	H, % Calcd.	H, % Found	N, % Calcd.	N, % Found
CH_3NH	153.5~154.5	94.8	E	$C_7H_{12}O_2N_2$	53.83	53.88	7.75	7.91	17.94	18.12
C_2H_5NH	180 ~181	87.0	E	$C_8H_{14}O_2N_2$	56.45	56.30	8.30	8.34	16.46	16.46
C_6H_5NH	158.5~159.5	93.0	M	$C_9H_{14}O_2N_2$	59.32	59.51	7.75	7.68	15.37	15.33
n-C_3H_7NH	168 ~168.5	94.2	E	$C_9H_{16}O_2N_2$	58.67	58.94	8.75	9.12	15.21	15.20
i-C_3H_7NH	164.5~165.5	90.2	E	$C_9H_{16}O_2N_2$	58.67	58.91	8.75	8.85	15.21	15.02
C_7H_7NH	172 ~173	95.2	E	$C_{13}H_{16}O_2N_2$	67.18	67.23	6.94	7.21	12.06	12.20
C_2H_5ONH	134.5~135.5	94.8	BE	$C_8H_{14}O_3N_2$	51.60	51.62	7.59	7.64	15.05	14.87
C_2H_6N	132.5~133	62.9	E	$C_8H_{14}O_2N_2$	56.45	56.58	8.30	8.42	16.46	16.45
$C_5H_{10}N$	86 ~ 87	91.9	AE	$C_{11}H_{18}O_2N_2$	62.82	63.01	8.64	8.72	13.32	13.33

a) AE, Ethyl acetate; BE, Benzene-ethanol; E, Ethanol; M, Methanol

1) K. Yanagi, *J. Org. Chem.*, **24**, 1121 (1959).
2) L. J. Bellamy, "The Infrared Spectra of Complex Molecules", John Wiley and Sons, Inc., New York (1954), p. 176.

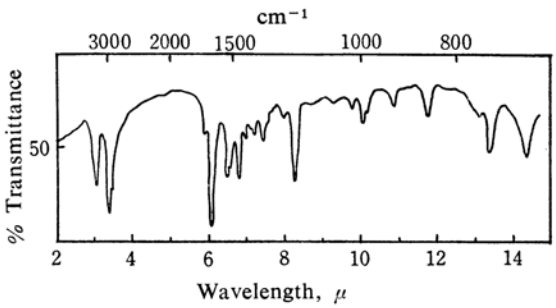


Fig. 1. Infrared spectrum of II (Nujol).

Thermal Decomposition of *N*-Vinyl-*N'*-benzylsuccinamide.—When II was heated at about 180°C for a few minutes, a red liquid was obtained. After recrystallization of the product from methanol, crystals were obtained; m. p. 98~99°C, undepressed by admixture with an authentic compound (Found: N, 7.28%).

Synthesis of *N*-Vinyl-*N'*-2-hydroxyethylsuccinamide.—A mixture of 1.25 g. of *N*-vinylsuccinimide and 0.6 g. of monoethanolamine in 2 ml. of benzene was kept at room temperature for few minutes. The precipitate was then filtered and dried. The product was recrystallized from benzene-ethanol. The purified sample melted at 134.5~135.5°C (decomp.); the yield was 1.75 g. (94.8%).

Reaction of *N*-Vinylsuccinimide with Morphorine.—*N*-Vinylamide was expected to be produced in this run, too, but it was not obtained in an analytically pure state. In the infrared spectrum of the crude material, absorption bands were observed at 3.02, 6.58 and 10.06 μ; these were assigned to the NH stretching vibration of a secondary amide and to CH out-of-plane deformation.

Copolymerization of *N*-Vinyl-*N'*-*n*-propylsuccinamide with Methyl Methacrylate.—A solution of 0.2 g. of *N*-vinyl-*N'*-*n*-propylsuccinamide, 1.8 g. of methyl methacrylate and 0.02 g. of benzoyl peroxide in 7 ml. of dioxane was heated at 65~66°C in a water bath for 6 hr. The polymer solution was poured into 70 ml. of methanol. After filtration and drying, the polymer weighed 0.41 g. (N, 1.69%). It was soluble in acetone, tetrahydrofurane, dioxane, benzene, toluene, chloroform, tetrachloroethane and ethyl acetate.

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