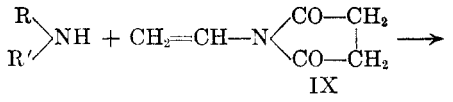
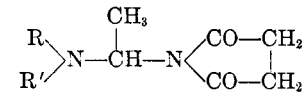
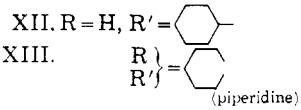
succinimide (XI), *N*-(1-cyclohexylaminoethyl)succinimide (XII) and *N*-(1-piperidinoethyl)succinimide (XIII) by the addition reaction. The results are shown in Table I.



IX



X. R = H, R′ = C_2H_5—
XI. R = H, R′ = OH—$(CH_2)_2$—



N-vinylsulfobenzimide (XIV) was allowed to react with cyclohexylamine and aniline to yield products with empirical formula which correspond to *N*-cyclohexyl-*o*-sulfamoylbenzamide (XV) and *o*-sulfamoylbenzanilide (XVI), respectively. That is to say, neither addition reaction nor imido interchange reaction occurs in the case of *N*-vinylsulfobenzimide.

EXPERIMENTAL

Materials. *N*-vinylphthalimide,[6] *N*-vinylsuccinimide,[7] and *N*-vinylsulfobenzimide[8] were prepared by the pyrolysis of *N*-2-acetoxyethylphthalimide, *N*-2-acetoxyethylsuccinimide, and *N*-2-acetoxyethylsulfobenzimide, respectively.

Addition reactions of N-vinylphthalimide. A mixture of 5 g. of V.P.I. (*N*-vinylphthalimide) and 10 g. of ethylamine (33% water solution), a mixture of 5 g. of V.P.I. and 5.72 g. of cyclohexylamine, and a mixture of 4 g. of V.P.I. and 2.2 g. of piperidine in 15 ml. of benzene, were individually kept at room temperature for 3 hr. The precipitate was filtered and recrystallized. The results are summarized in Table I.

Imido interchange reactions of N-vinylphthalimide. A mixture of 5 g. of V.P.I., 3 g. of aniline, and 1 g. of acetic acid; a mixture of 5 g. of V.P.I., 3.56 g. of *p*-anisidine, and 1 g. of acetic acid; a mixture of 6 g. of V.P.I., 4.8 g. of *p*-aminobenzoic acid, and 1 g. of acetic acid; and a mixture of 4 g. of V.P.I., 1.6 g. of ethanolamine and 1 g. of acetic acid were individually heated at 100° for 4.5, 4, 5, and 10 hr., respectively. The resinous reaction products were rinsed with methanol and recrystallized from benzene and then *N*-phenylphthalimide, *N*-(4-methoxyphenyl)phthalimide, *N*-(4-carboxyphenyl)phthalimide, and *N*-(2-hydroxyethyl)-phthalimide were obtained.

N-phenylphthalimide: yield 85%, m.p. 209–210°.

Anal. Calcd. for $C_{14}H_9NO_2$: C, 75.32; H, 4.06; N, 6.28. Found: C, 75.61; H, 4.06; N, 6.54.

N-(4-methoxyphenyl)-phthalimide: yield 71%, m.p. 160–161°.

Anal. Calcd. for $C_{15}H_{11}NO_3$: C, 71.14; H, 4.37; N, 5.53. Found: C, 71.39; H, 4.55; N, 5.59.

N-(4-carboxyphenyl)-phthalimide: yield 80%, m.p. 261–262° (uncorr.).

Anal. Calcd. for $C_{15}H_9NO_4$: C, 67.41; H, 3.39; N, 5.24. Found: C, 67.18; H, 3.72; N, 5.13.

N-(2-hydroxyethyl)phthalimide: yield 70%, m.p. 128–129°.

Anal. Calcd. for $C_{10}H_9NO_3$: C, 62.82; H, 4.75; N, 7.33. Found: C, 63.06; H, 4.91; N, 7.12.

Addition reactions of N-vinylsuccinimide. A mixture of 6 g. of V.S.I. (*N*-vinylsuccinimide) and 12 g. of ethylamine (33% water solution), a mixture of 4 g. of V.S.I. and 2.2 g. of ethanolamine in 15 ml. of benzene, a mixture of 4 g. of V.S.I. and 7.5 g. of cyclohexylamine, and a mixture of 4 g. of V.S.I. and 3 g. of piperidine were individually kept at room temperature for 2, 0.5, 18 and 18 hr., respectively. The precipitates were filtered and recrystallized. The results are summarized in Table I.

N-cyclohexyl-o-sulfamoylbenzamide. A mixture of 4.5 g. of *N*-vinylsulfobenzimide and 6 g. of cyclohexylamine was stirred at room temperature. After 17 hr., the precipitate was rinsed with benzene to remove the resinous matter from it. Recrystallization from benzene gave *N*-cyclohexyl-*o*-sulfamoylbenzamide, yield 60%, m.p. 201–202.5°.

Anal. Calcd. for $C_{13}H_{18}N_2O_3S$: C, 55.31; H, 6.53; N, 9.93. Found: C, 55.23; H, 6.51; N, 9.81.

o-Sulfamoylbenzanilide. A mixture of 5.2 g. of *N*-vinylsulfobenzimide, 6 g. of aniline, and 2 ml. of acetic acid was heated at 100° for 3 hr. The reaction product was rinsed with benzene to remove the resinous matter. Recrystallization from ethanol gave 4.92 g. of *o*-sulfamoylbenzanilide, m.p. 189–190°.

Anal. Calcd. for $C_{13}H_{12}N_2O_3S$: C, 56.52; H, 4.83; N, 10.14. Found: C, 56.38; H, 4.51; N, 10.17.

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(8) This is a new compound, m.p. 131–132° (from ethanol). *Anal.* Calcd. for $C_9H_7NO_3S$: C, 51.67; H, 3.37; N, 6.70. Found: C, 51.61; H, 3.36; N, 6.64.

Hydroxyethylation of Imides

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Several methods of preparing *N*-2-hydroxyethylimides are known.[1–3] The present paper describes a new method of hydroxyethylation of imides by ethylene carbonate, which is used not only as solvent but also as reagent.[4–7]

Though monocarboxylic amides showed no reaction, dicarboxylic imides and saccharin were easily hydroxyethylated in good yield by heating the mixture of imide and ethylene carbonate. Aromatic dicarboxylic imides reacted easily with-

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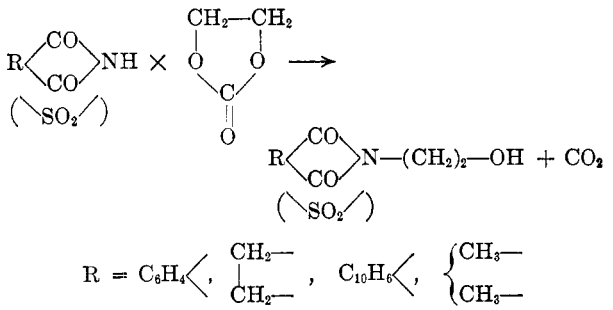
TABLE I

HYDROXYETHYLATION OF IMIDES

Imide	E.C.[a] (mol.) / Imide (mol.)	Alkali	Temp.	Time, Hr.	Yield, %
Phthalimide[b]	1.1	...	200	1.0	95
K-phthalimide[c]	2–3	...	140–160	0.5	60
Naphthalimide[d]	2.0	...	210	1.0	96
Succinimide[e]	1.0	Na_2CO_3 (0.5%)	190	1.0	86
Diacetamide[f]	1.0	Na_2CO_3 (0.5%)	195	1.0	70
Saccharin[g]	1.0	NaOH (0.5%)	210–220	1.0	93

[a] "E.C." represents ethylene carbonate. Product of American Cyanamid Co. [b] *Org. Syntheses*, **Coll. Vol. I**, 457 (1948). [c] *Org. Syntheses*, **Coll. Vol. I**, 119 (1948). [d] T. Maki and H. Hashimoto, *J. Chem. Soc. Japan, (Ind. Chem. Sec.)*, **54**, 480 (1951). [e] *Org. Syntheses*, **Coll. Vol. II**, 562 (1943). [f] L. Vanino, *Handbuch der Preparativen Chemie (2 Auflage)*, Ferdinand Enke, Stuttgart, II Bd., p. 207. [g] This was precipitated from the aqueous solution of its sodium salt by dilute hydrochloric acid, m.p. 225–227°.

out alkali carbonate or hydroxide. Aliphatic dicarboxylic imides reacted in the presence of alkali carbonate. Saccharin reacted only in the presence with alkali hydroxide. While potassium phthalimide was hydroxyethylated, sodium saccharin was not. The results are summarized in Table I.



R = C_6H_4<, CH_2—, CH_2—> , $C_{10}H_6$<, {CH_3—, CH_3—}

The dissociation constants of phthalimide,[8] succinimide,[9] and saccharin,[10] which were hydroxyethylated in good yield, are 1.09×10^{-7}, 3.0×10^{-11} and 2.5×10^{-2} respectively (25°) and those of acetamide,[11] benzamide,[12] and acetanilide[13] which were not hydroxyethylated are 8.3×10^{-15}, *ca.* 1×10^{-14} and *ca.* 1×10^{-13}, respectively. Imides whose dissociation constants are larger than 10^{-11}–10^{-12}, seem to be hydroxyethylated by ethylene carbonate.

EXPERIMENTAL

The mixture of imide and ethylene carbonate was heated under the conditions shown in Table I. The reaction products were treated as follows:

The reaction product of phthalimide and ethylene carbonate was recrystallized from water, m.p. 127°. The mixed melting point of this with *N*-2-hydroxyethylphthalimide was 126–127°. The infrared spectra of the two coincided.

The reaction product of potassium phthalimide with ethylene carbonate was washed with water and the precipitate was filtered. After it was treated with dilute hydrochloric acid, recrystallization from water gave N-2-hydroxyethylphthalimide. The mixed melting point of this with the authentic sample was 126–127°, and the infrared spectra of the two coincided.

The reaction product of naphthalimide and ethylene carbonate was recrystallized from water and N-2-hydroxyethylnaphthalimide was obtained, m.p. 172–173°.

Anal. Calcd. for $C_{14}H_{11}NO_3$: N, 5.80. Found: N, 5.99.

Distillation of the reaction product of succinimide with ethylene carbonate under vacuum gave N-2-hydroxyethylsuccinimide, b.p. 161–162° (3 mm. Hg), m.p. 62.5–63.5° (from acetone–carbon tetrachloride).

Anal. Calcd. for $C_6H_9NO_3$: C, 50.34; H, 6.34 N, 9.79. Found: C, 49.73; H, 6.53; N, 10.05.

Distillation of the reaction product of diacetamide with ethylene carbonate gave *N*-2-hydroxyethyldiacetamide, b.p. 129° (2.5 mm. Hg), 124° (1.5 mm.).

Anal. Calcd. for $C_6H_{11}NO_3$: N, 9.65. Found: N, 9.69.

The reaction product of saccharin with ethylene carbonate was recrystallized from water, and *N*-2-hydroxyethylsulfobenzimide was obtained, m.p. 105.5–106.5°.

Anal. Calcd. for $C_9H_9NO_4S$: C, 47.58; H, 3.99; N, 6.17. Found: C, 47.66; H, 3.54; N, 6.21.

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Preparation of Halo-5-nitro-2-furanacrylanilides

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Several halo-5-nitro-2-furanacrylanilides have been reported as being effective anthelmintics and bacteriostats.[1–3] These compounds had been prepared by the reaction of purified 5-nitro-2-furanacryloyl chloride with a halogenated aniline under

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