layer, upon acidification with sulphuric acid, deposited 2.8 gm. of 2,4-dimethoxyphenoxypropionic acid, m.p. 101.5-102.5°C., out of water.
Calc. for $C_{11}H_{14}O_5$: CH_3O, 27.4%. Found: CH_3O, 27.3%.

6,8-Dimethoxy-4-chromanone was prepared by heating for three hours under reflux with continuous stirring 2.1 gm. of 2,4-dimethoxyphenoxypropionic acid with 10 gm. of phosphoric anhydride in 75 ml. of dry benzene. The benzene was decanted, the residue treated with ice and water, then extracted with benzene. The combined benzene solutions were washed with dilute alkali and water, and dried with sodium sulphate. Removal of the benzene with a stream of nitrogen left a sticky yellow solid. Two crystallizations out of cyclohexane yielded 0.30 gm. of colorless 6,8-dimethoxychromanone, m.p. 101-102°C. Calc. for $C_{11}H_{12}O_4$: C, 63.5; H, 5.77%. Found: C, 63.2; H, 6.00%. The oxime, white needles from 50% ethanol, melted at 169-170°C.

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Urea Derivatives[1]

A series of urea derivatives were prepared for the purpose of examining their physical properties. The new ones are recorded in Table I along with their melting points. In addition the cyclic ureas (2-imidazolidones) 1-β-hydroxyethyl-2-imidazolidone and 1,2-*bis*-1-(2-imidazolidonyl)ethane were prepared. These last two compounds on nitration in acetic anhydride – nitric acid medium gave 1-β-nitroxyethyl-3-nitro-2-imidazolidone and 1,2-*bis*-1-(3-nitro-2-imidazolidonyl)ethane which proved to be identical with the hydrolysis products (2) from 1-β-nitroxyethyl-2-nitrimino-3-nitroimidazolidine and 1,2-*bis*-1-(2-nitrimino-3-nitroimidazolidinyl)ethane respectively. This, together with the ultraviolet studies (3), confirms the structures assigned to the 1-substituted-2-nitrimino-3-nitroimidazolidines (2).

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EXPERIMENTAL[2,3]

Isocyanates

p-Tolylisocyanate, *p*-anisylisocyanate, and *p*-phenetylisocyanate were obtained from Eastman Kodak, Rochester, N.Y.

Benzylisocyanate

Benzylisocyanate (b.p.$_{17}$ 103°C.) was prepared in 46% yield as previously described (1).

Ethylisocyanate

Ethylisocyanate (b.p.$_{760}$ 59.5-60°C.) was prepared in 87% yield by the method of Slotta and Lorenz (4).

Urea Derivatives

The isocyanates (0.027 mole) were added to an amine (0.025 mole) dissolved in 30-35 cc. of benzene with cooling when necessary. The corresponding ureas crystallized from the benzene solution almost immediately. Occasionally it was necessary to add petroleum ether (b.p. 30-60°C.) to the benzene solution to complete precipitation of the urea derivative. The urea derivatives were obtained in practically quantitative yields and they melted 1-5° below the purified melting point. They were purified readily by crystallization from 95% ethanol or glacial acetic acid. The hitherto unreported urea derivatives are listed in Table I with their melting points.

1-β-Hydroxyethyl-2-imidazolidone

1-β-Hydroxyethyl-2-imidazolidone (m.p. 58-59°C.) was obtained in 50% yield from urea and 1-hydroxy-5-amino-3-azapentane employing the method of Wilson (5). Wilson reported the melting point as 50-51°C.

Calc. for $C_5H_{10}N_2O_2$: C, 46.15; H, 7.69; N, 21.54%. Found: C, 45.91; H, 7.71; N, 21.30%.

1,2-bis-1-(2-Imidazolidonyl)ethane

Urea (10 gm., 0.165 mole) and 1,8-diamino-3,6-diazaoctane (17.9 gm., 0.124 mole) were heated together at 150-190°C. until ammonia evolution ceased. The cooled reaction mixture was washed with methanol (200 cc.) to give 12.97 gm. (75.1%) of 1,2-*bis*-1-(2-imidazolidonyl)ethane melting at 248-252.5°C. One crystallization from water (29 cc.) raised the melting point to 253.5-254.5°C. The melting point previously reported (5) is 240-245°C. Calc. for $C_8H_{14}N_4O_2$: C, 48.49; H, 7.07; N, 28.28%. Found: C, 48.18; H, 7.40; N, 28.00%.

1-β-Nitroxyethyl-3-nitro-2-imidazolidone

Two grams (0.0154 mole) of 1-β-hydroxyethyl-2-imidazolidone were added to a solution of 6.3 cc. (0.15 mole) of absolute nitric acid in 14.45 cc. (0.15 mole) of acetic anhydride at 0°C. The temperature was allowed to rise to room temperature during the course of 40 min. Then the solution was poured onto ice with stirring. After the solution had stood in an open beaker for three days, a crop of crystals (m.p. 102-103°C.) was obtained, yield 1.66 gm. (49%). This material on admixture with a sample of 1-β-nitroxyethyl-3-nitro-2-imidazolidone (m.p. 102-103°C.) from the hydrolysis of 1-β-nitroxyethyl-2-nitrimino-3-nitro-imidazolidine (2) did not depress the melting point.

1,2-bis-1-(3-Nitro-2-imidazolidonyl)ethane

1,2-*bis*-1-(2-Imidazolidonyl)ethane 2 gm. (0.01 mole) was added to a solution

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[2] *All melting points were determined on a Kofler block.*

[3] *Microanalyses by Mr. C. W. Beazley, Skokie, Ill.*

TABLE I
RNHCONHR′

R	R′	M.p., °C.	N		Halogen	
			Calcd.	Found	Calcd.	Found
Benzyl	*p*-Phenetyl	181.5-182	10.35	10.13		
Benzyl	*p*-Chlorophenyl	207.5-208.5			13.61	13.93
Benzyl	*p*-Bromophenyl	222.5-223			26.40	25.95
Benzyl	*o*-Bromophenyl	168-168.5	9.15	9.03		
Ethyl	*m*-Tolyl	99.5-100.5	15.73	15.72		
Ethyl	*o*-Bromophenyl	148-148.5			32.90	32.45
Ethyl	*o*-Anisyl	141.5-142	14.44	14.31		
Ethyl	*β*-Phenylethyl	77-78	14.58	14.36		
p-Tolyl	*p*-Phenetyl	215.5-216.5	10.37	10.60		
p-Tolyl	*m*-Phenetyl	179-180	10.37	10.43		
p-Tolyl	*o*-Phenetyl	175.5-176.3	10.37	10.57		
p-Tolyl	*p*-Anisyl	240-240.5	10.94	11.00		
p-Tolyl	*m*-Anisyl	189-189.5	10.94	11.08		
p-Tolyl	*o*-Anisyl	157.5-158	10.94	10.96		
p-Tolyl	*m*-Tolyl	209-209.2	11.66	11.96		
p-Tolyl	*o*-Chlorophenyl	211.5-212	10.74	11.00		
p-Tolyl	*o*-Bromophenyl	223.5-224			26.23	26.30
p-Anisyl	*p*-Phenetyl	215.5-216	9.79	9.80		
p-Anisyl	*m*-Phenetyl	165-165.5	9.79	9.78		
p-Anisyl	*o*-Phenetyl	137.2-137.8	9.79	10.06		
p-Anisyl	*m*-Anisyl	158-158.2	10.29	10.27		
p-Anisyl	*o*-Anisyl	145.5-145.8	10.29	10.35		
p-Anisyl	*m*-Tolyl	189-189.4	11.14	11.24		
p-Anisyl	*o*-Tolyl	203.5-204.5	11.14	11.05		
p-Anisyl	*p*-Chlorophenyl	271-271.5	10.13	10.42		
p-Anisyl	*m*-Chlorophenyl	202-202.2	10.13	10.13		
p-Anisyl	*o*-Chlorophenyl	193-193.2	10.13	10.20		
p-Anisyl	*m*-Bromophenyl	204.5-205			24.92	24.30
p-Anisyl	*o*-Bromophenyl	199-199.5			24.92	25.30
p-Phenetyl	*m*-Phenetyl	160-160.3	9.33	9.55		
p-Phenetyl	*o*-Phenetyl	147.5-148	9.33	9.76		
p-Phenetyl	*o*-Anisyl	162-162.5	9.79	10.06		
p-Phenetyl	*m*-Tolyl	169.5-170	10.37	10.68		
p-Phenetyl	*o*-Tolyl	210	10.37	10.70		
p-Phenetyl	*p*-Chlorophenyl	246.7			12.22	12.60
p-Phenetyl	*m*-Chlorophenyl	199.2-199.5			12.22	12.46
p-Phenetyl	*o*-Chlorophenyl	182.2-182.8			12.22	12.46
p-Phenetyl	*m*-Bromophenyl	197-197.5			23.88	23.83
p-Phenetyl	*o*-Bromophenyl	189-189.2			23.88	24.46

of 4.19 cc. (0.10 mole) of absolute nitric acid in 9.7 cc. (0.10 mole) of acetic anhydride at 0°C. The temperature was raised to 22°C. and maintained at this level for 30 min. Then the reaction mixture was poured onto ice and the precipitate recovered by filtration. The product melted at 241-243°C. with decomposition, yield 2.35 gm. (78.5%). It did not depress the melting point of 1,2-*bis*-1-(3-nitro-2-imidazolidonyl)ethane (m.p. 242-243°C. with decomposition) obtained from the hydrolysis of 1,2-*bis*-1-(2-nitrimino-3-nitroimidazolidinyl)ethane (2).

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