crystals formed within one day at room temperature leaving a colorless solution. The reaction took place even with calcium oxide present.

The behavior of morpholine with chloroform is similar. Morpholinium chloride was identified as the product by its melting point, by its reaction with aqueous silver nitrate, and by taking a mixed melting point with morpholinium chloride prepared from hydrogen chloride and from carbon tetrachloride.

HAYDEN MEMORIAL LABORATORIES
NORTHEASTERN UNIVERSITY
BOSTON, MASSACHUSETTS RECEIVED JULY 11, 1942

Solubility of the Flavianates of Certain Organic Bases in Water, Ethanol, and *n*-Butanol at 3 and 30°

BY WILSON D. LANGLEY AND THOMAS R. NOONAN

The use of 2,4-dinitronaphthol-7-sulfonic acid (flavianic acid) for the purification and characterization of organic bases, first recommended by Kossel and Edlbacher, and Kossel and Gross,[1] has been extended by Sievers and Mueller[2] to include solubility data, and by Langley and Albrecht[3] to include crystallographic data. It has been our desire further to extend knowledge of the solubilities of the flavianates in certain solvents, so that flavianic acid may be more satisfactorily used for fractional precipitation of organic bases. Accordingly, we have determined the solubilities reported in the accompanying table. The solvents were selected as being suited for fractionation of extracts of tissues, and it was hoped that quantitative separations of bases could be accomplished readily once the components of mixtures were identified under the microscope. This work has been interrupted, and the prospects of it being resumed are remote.

The solvents used were purified by distillation just prior to use, and purity was established by constancy of boiling point, and by measurement of density (pycnometer). The flavianates used had been analyzed and reported upon previously.[3] Equilibrium was attained by frequent shaking of solvent in contact with solid for various lengths of time ranging from several days to several months. The saturated solutions were filtered, and were pipetted immediately by use of pipets which were calibrated at the temperatures used. Fifty-ml. portions (occasionally 20 ml.) of solutions were pipetted into weighed beakers, and the covered solutions were evaporated on a steam-bath. Final drying of the residue was done in an oven at 100°, constant weight being attained in each case.

SOLUBILITY OF FLAVIANATES IN G. PER LITER

Base	Water 3°	Water 30°	Ethanol (95%) 3°	Ethanol (95%) 30°	*n*-Butanol 3°	*n*-Butanol 30°
Acetylcholine	...	...	...	...	0.09	0.40
Ammonium	14.2	...	2.57	6.22	.29	.39
Choline	...	...	2.81	...	.17	.26
Creatinine	2.65	4.54	1.08	1.52	.09	.43
as-Dimethylguanidine	1.85	...	1.30	3.2	.21	.30
Ethanolamine	...	...	2.45	6.8	.14	.28
Guanidine	1.30	3.34	1.64	3.57	.19	.19
Hydroxylamine	16[a]	70[a]	...	26[a]	2.4[a]	5.2[a]
Hypoxanthine	1.3	3.6	0.95	3.36	0.34	0.4
Methylamine	7.6	...	1.95	4.10	.09	.17
Methylguanidine	2.53	5.7	2.6	4.7	.33	.35
Methylurea	...	...	...	36[a]	.66	1.4[a]
Piperidine	4.0	..	3.3[a]	1.3	.13	0.35
Potassium	3.7[a]	11.2[a]	0.12	0.16	.04[a]	.05
Putrescine	0.25	...	0.31	0.46	..	.06
Tetramethyl-ammonium	4.9[a]	12.8[a]	0.61	1.52	.04	.05
Trimethylamine	47[a]	...	4.4[a]	7.27	.12	.41
Tyramine	...	...	4.40	10.3[a]	.34	.8
Urea	15.7[a]	40[a]	12.4	17	.56	.81

[a] Single determinations.

The figures represent averages of values which were obtained after differences of several weeks in contact time, and which, except for the very small values, seldom disagreed by as much as 5%. Uncertain figures are depressed below the line. When the solubilities were great, duplicate determinations were not always made; these single values are marked.

DEPARTMENT OF BIOLOGICAL CHEMISTRY
UNIVERSITY OF BUFFALO MEDICAL SCHOOL
BUFFALO, N. Y. RECEIVED JUNE 27, 1942

1-Carbamyl-5-methylpyrazole-3-carboxylic Acid

BY ALBERT L. LEHNINGER

During the course of some work on the derivatives of acetopyruvic acid,[1] the reaction between semicarbazide and acetopyruvic acid became of interest as a means of identification of the latter compound. Von Auwers and Cauer[2] had reported that they were unable to obtain the expected product, 1-carbamyl-5-methylpyrazole-3-carboxylic acid (I), since the carbamyl group was apparently lost on ring closure, leading instead to 5-methylpyrazole-3-carboxylic acid (II).[3]

(1) A. Kossel and S. Edlbacher, *Z. physiol. Chem.*, **110**, 241 (1920); A. Kossel and R. E. Gross, *ibid.*, **135**, 167 (1924).

(2) H. Sievers and E. Mueller, *Z. Biol.*, **89**, 37 (1929); **92**, 513 (1932).

(3) W. D. Langley and A. J. Albrecht, *J. Biol. Chem.*, **108**, 729 (1935).

(1) Lehninger and Witzemann, THIS JOURNAL, **64**, 874 (1942).

(2) von Auwers and Cauer, *J. prakt. Chem.*, **126**, 146 (1930).

(3) Knorr and MacDonald, *Ann.*, **279**, 217 (1894).