of sodium carbonate and 60 ml. of water, was added simultaneously and gradually 18.7 g. (0.08 mole) of *p*-acetaminobenzenesulfonyl chloride and enough 30% sodium hydroxide to maintain a *p*H of about 10. Stirring was continued for thirty minutes after the additions were complete and then 8 g. of solid sodium hydroxide was added and the mixture refluxed for ninety minutes. Exact neutralization gave 10 g. (47%) of the anilide which after crystallization from dilute methanol melted at 163–164°. Considerable unchanged *p*-fluoroaniline was recovered.

Anal. (Kjeldahl) Calcd. for $C_{12}H_{11}O_2SN_2F$: N, 10.53. Found: N, 10.8.

TABLE I

Compound, -benzenesulfonic acid	Yield,[a] %	Decompn. temp., °C.	Neutral eq.[b] Calcd.	Found
2-Sulfanilamido-5-fluoro-	14[c]	285	346	343
5-Sulfanilamido-2-fluoro-	42	260	346[d]	345
2-Sulfanilamido-5-chloro-	20[c]	300	380.5[e]	383
5-Sulfanilamido-2-chloro-	57	310	362.5	360

[a] These percentages do not take into account unreacted starting materials which were recovered. [b] The indicator used was methyl red–methylene blue. [c] The low yields were partly due to the low solubilities of the original sodium sulfonates. [d] This compound crystallized as the monohydrate. *Anal.* Calcd. for $C_{12}H_{11}O_5N_2S_2F\cdot H_2O$: neut. equiv., 364. Found: neut. equiv., 361.2. [e] This compound crystallized as the monohydrate. *Anal.* Calcd. for $C_{12}H_{11}O_5N_2S_2Cl\cdot H_2O$: H_2O, 4.72. Found: H_2O, 4.89. The dry material absorbed water rapidly when exposed to the air.

Sulfanilamidofluoro- and -chlorobenzenesulfonic Acids.—*p*-Acetaminobenzenesulfonyl chloride was condensed with the aminosulfonic acids essentially according to the procedure of Crossley, Northey and Hultquist,[2] and the sulfanilamido compounds were isolated as the free acids. The sodium salts of these were very soluble in water. The yields and analyses are summarized in Table I.

Summary

The isomeric 4-fluoroaniline- and 4-chloroaniline-sulfonic acids have been prepared. The sulfanilamidohalobenzenesulfonic acids prepared from these did not exhibit therapeutic action against Streptococcus and Type I Pneumococcus infections in white mice. N-Sulfanilyl-4-fluoroaniline showed a slight effect against Streptococcus infections.

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[CONTRIBUTION FROM THE CHEMISTRY DEPARTMENT OF THE UNIVERSITY OF COLORADO]

A Series of 2-Methyl-5-alkyl-4,6-dihydroxypyrimidines

BY LIVINGSTON P. FERRIS II* AND ANTHONY R. RONZIO

E. L. Pinner[1] prepared 2-phenyl-4,6-dihydroxypyrimidine by the condensation of benzamidine with malonic ester. Dox and Yoder[2] continued this investigation obtaining more members of the same series. At this time they pointed out that the condensation using aliphatic amidines had not been tried. Remfry[3] discovered a reaction in which malonamide condensed with substituted malonic esters to give pyrimidines, identical in structure to those which theoretically should form if aliphatic amidines successfully condensed with substituted malonic esters.

We have investigated this reaction using acetamidine and substituted malonic esters in the presence of sodium alcoholate and have found that the expected pyrimidines are formed. The first six members of the series have been prepared for the purpose of a pharmacological study. Absorption spectra data were taken for the first member of the series.

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(1) E. L. Pinner, *Ber.*, **41**, 3517 (1908).

(2) A. W. Dox and L. Yoder, THIS JOURNAL, **44**, 361 (1922).

(3) F. G. P. Remfry, *J. Chem. Soc.*, **99**, 610 (1911).

Experimental Part

Acetamidine hydrochloride, one-eighth mole, and either the redistilled malonic ester or the redistilled monoalkyl substituted malonic ester, one-twentieth mole, were added in the order given, to about 150 cc. of absolute alcohol in which had been dissolved 3.3 g. of sodium (slight excess over one-eighth mole). After standing for two to three days, the solution was exactly neutralized with concentrated hydrochloric acid. Enough water to dissolve the precipitated sodium chloride was then added. The un-

TABLE I

2-METHYL-5-ALKYL-4,6-DIHYDROXYPYRIMIDINES

5 Occupied by	Yield, %	Empirical formula	% C Calcd.	% C Found	% H Calcd.	% H Found	% N Calcd.	% N Found
H	43	$C_5H_6N_2O_2$	47.62	47.61 47.64	4.79	5.07 4.99	22.22	22.22 22.14
Methyl	18	$C_6H_8N_2O_2$	51.42	51.45 51.40	5.75	6.08 6.03	19.99	20.00 19.90
Ethyl	15	$C_7H_{10}N_2O_2$	54.53	54.52 54.49	6.54	6.62 6.72	18.17	18.09 18.28
n-Propyl[a]	22	$C_8H_{12}N_2O_2$	57.13	57.17 57.21	7.19	7.29 7.33	16.66	16.79 16.73
n-Butyl	33	$C_9H_{14}N_2O_2$	59.32	59.52 59.51	7.74	7.80 7.86	15.38	15.32 15.49
n-Amyl	26	$C_{10}H_{16}N_2O_2$	61.20	61.28 61.30	8.22	8.40 8.32	14.28	14.32 14.33

[a] First prepared by Remfry, ref. 3.

dissolved pyrimidine was filtered, decolorized in boiling glacial acetic acid, and reprecipitated by the addition of ether. After several reprecipitations in this manner, the pyrimidines left no residue when ignited and, after drying over phosphorus pentoxide *in vacuo* at 100°, gave correct analyses.

The pyrimidines prepared, as well as experimental and analytical data, are shown in Table I.

The pyrimidines are colorless compounds, slightly soluble in water, the solubility decreasing with increase in molecular weight. Definite melting points were not shown by any member of the series but sublimation took place between 260–350°. In a sealed tube they decomposed without melting.

The pyrimidines required a special Dumas method for correct nitrogen analyses.

The absorption spectra curves for 2-methyl-4,6-dihydroxypyrimidine in neutral, acid and basic solution are shown in Fig. 1. The curves show a maxima at about 2600 Å. The absorption spectrum curve for this pyrimidine in neutral solution shows a slight break at 2700 Å.

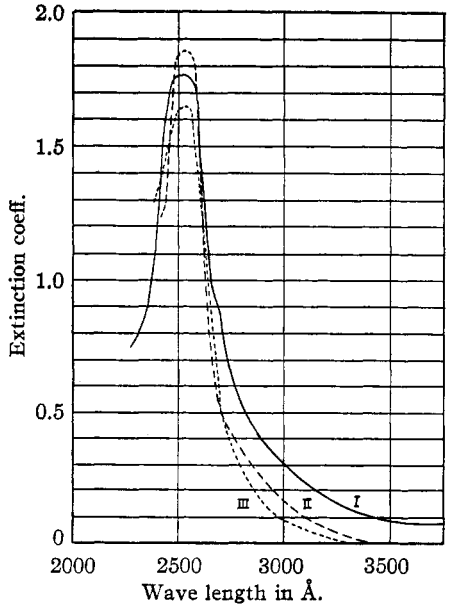

Fig. 1.—2-Methyl-4,6-dihydroxypyrimidine: I, 1.06 mg./50 cc. H_2O; II, 1.01 mg./50 cc. H_2O, 1 cc. concd. HCl; III, 2.06 mg./50 cc. H_2O, 1 cc. 50% KOH. 1-Cm. cell used throughout.

For purposes of comparison the absorption spectrum of 2-phenyl-5-*n*-butyl-4,6-dihydroxypyrimidine in alcoholic solution was taken (Fig. 2). A slight break in the curve at 3350 Å. not reported by Dox and Yoder[2] was observed.

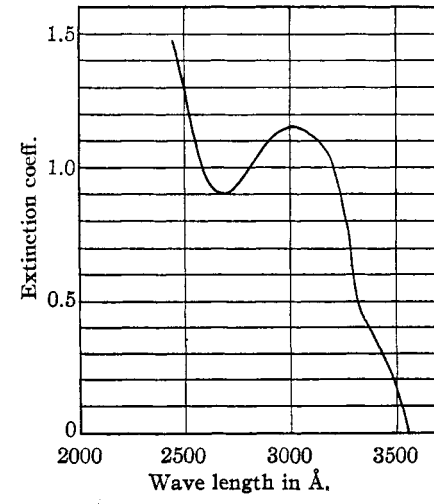

Fig. 2.—2-Phenyl-5-*n*-butyl-4,6-dihydroxypyrimidine: 1.61 mg./50 cc. ethyl alcohol, 1-cm. cell.

These data were obtained using a Hilger E3 spectrograph, Hilger sector photometer and Eastman Panchromatic plates. An under water spark served as light source.

Summary

A series of 2-methyl-5-alkyl-4,6-dihydroxypyrimidines has been prepared.

Absorption spectra curves of 2-methyl-4,6-dihydroxypyrimidine in neutral, acid and basic solution are shown.

The absorption spectrum curve of 2-phenyl-5-*n*-butyl-4,6-dihydroxypyrimidine in neutral solution has been retaken.

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